

DRAFT

**UNIFORM FEDERAL POLICY-QUALITY ASSURANCE
PROJECT PLAN**

**PER- AND POLYFLUOROALKYL SUBSTANCES
REMEDIAL INVESTIGATION
YAKIMA TRAINING CENTER, WASHINGTON**

Contract No. W9124J-18-D-0004, Delivery Order No. W9124J-22-F-0144

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LIST OF ACRONYMS AND ABBREVIATIONS

| | |
|---------|--|
| ± | plus or minus |
| < | less than |
| ≤ | less than or equal to |
| > | greater than |
| ≥ | greater than or equal to |
| % | percent |
| %R | percent recovery |
| °C | degrees Celsius |
| °F | degrees Fahrenheit |
| µg/kg | micrograms per kilogram |
| A | Analytical |
| AAF | Army Airfield |
| AEL | Advanced Environmental Laboratories, Inc. |
| AFCEC | Air Force Civil Engineering Center |
| AFFF | aqueous film-forming foam |
| amu | atomic mass unit |
| AOI | area of interest |
| AOPI | area of potential interest |
| APP | Accident Prevention Plan |
| Arcadis | Arcadis U.S., Inc. |
| AS | Associates of Science |
| ASTM | American Society for Testing and Materials International |
| bgs | below ground surface |
| BHHRA | Baseline Human Health Risk Assessment |
| BS | Bachelor of Science |
| CA | corrective action |
| CAS | Chemical Abstract Service |
| CCB | continuing calibration blank |
| CCI | Contaminant Control, Inc. |
| CCV | continuing calibration verification |
| CERCLA | Comprehensive Environmental Response, Compensation and Liability Act |
| CFR | Code of Federal Regulation |
| CIH | Certified Industrial Hygienist |
| CO | carbon monoxide |
| CoC | chain-of-custody |
| COR | Contracting Officer's Representative |
| CQM | Construction Quality Management |
| CRB | Columbia River Basalt |
| CSM | conceptual site model |
| CSP | Certified Safety Professional |
| DERP | Defense Environmental Restoration Program |
| DL | detection limit |
| DO | dissolved oxygen |
| DoD | Department of Defense |
| DPT | direct-push technology |
| DQCR | daily quality control report |
| DQI | data quality indicator |
| DQO | data quality objective |
| DUA | data usability assessment |

| | |
|------------------|---|
| DUP | sample duplicate |
| DVR | data validation report |
| EB | equipment blank |
| ECC | Environmental Chemical Corporation |
| EDD | electronic data deliverable |
| EIS | extracted internal standard |
| ELAP | Environmental Laboratory Accreditation Program |
| ELLE | Eurofins Lancaster Laboratories Environment Testing |
| EMI | electromagnetic inductions |
| EQASOP | EPA Quality Assurance Standard Operating Procedure |
| ERI | electrical resistivity imaging |
| ESM | Environmental Support Manager |
| EST | Eastern Standard Time |
| FB | field blank |
| FD | field duplicate |
| FFTA | Former Fire Training Area |
| Fm | Formation |
| FS | Feasibility Study |
| ft | feet |
| FTL | Field Team Leader |
| GIS | geographic information system |
| GPS | global positioning system |
| GW | groundwater |
| H ₂ S | hydrogen sulfide |
| H&S | Health and Safety |
| HAZWOPER | Hazardous Waste Operations and Emergency Response |
| HDPE | high density polyethylene |
| HFPO-DA | hexafluoropropylene oxide dimer acid |
| IAR | ion ratio |
| IB | instrument blank |
| ICAL | initial calibration |
| ICB | initial calibration blank |
| ICV | initial calibration verification |
| IDQTF | Intergovernmental Data Quality Task Force |
| IDW | investigation-derived waste |
| IMCOM | United States Army Installation Command Management |
| IRP | Installation Restoration Program |
| IS | internal standard |
| ISC | instrument sensitivity check |
| JBLM | Joint Base Lewis-McChord |
| LC/MS | liquid chromatography/mass spectrometry |
| LC/MS/MS | liquid chromatography with tandem mass spectrometry |
| LCS | laboratory control sample |
| LCSD | laboratory control sample duplicate |
| LEL | lower explosive limit |
| LLCS | Low-level laboratory control sample |
| LLOPR | low-level ongoing precision and recovery |
| LOD | limit of detection |
| LOQ | limit of quantitation |
| MB | method blank |

| | |
|----------------|---|
| MD | matrix duplicate |
| mg | milligrams |
| mg/kg | milligrams per kilogram |
| mg/L | milligrams per liter |
| mL | milliliter |
| MPC | Measurement Performance Criteria |
| MPH | Master of Public Health |
| MRL | minimum reporting limit |
| mS/cm | milliSiemens per centimeter |
| MS | matrix spike |
| MS | Master of Science |
| MSD | matrix spike duplicate |
| MW | monitoring well |
| N/A | not applicable |
| ND | non-detect |
| ng/g | nanograms per gram |
| ng/L | nanograms per liter |
| ng/mg | nanograms per milligram |
| NAPL | non-aqueous phase liquid |
| NELAP | National Environmental Laboratory Accreditation Program |
| N-Et-FOSA | N-ethylperfluorooctansulfonamid |
| N-Et-FOSE | N-ethylperfluorooctane sulfonamidoethanol |
| NFPA | National Fire Protection Association |
| NIS | Non-extracted Internal Standards |
| N-Me-FOSE | N-methylperfluorooctanesulfonamidoethanol |
| NTU | nephelometric turbidity unit |
| O ₂ | oxygen |
| OPR | ongoing precision and recovery |
| ORP | oxygen-reduction potential |
| OSD | Office of the Secretary of Defense |
| OSHA | Occupational Safety and Health Administration |
| oz | ounce |
| PA | preliminary assessment |
| PAL | project action limits |
| PE | Professional Engineer |
| PFAS | per- and polyfluoroalkyl substances |
| PFBS | perfluorobutanesulfonic acid |
| PFDA | perfluorodecanoic acid |
| PFHxA | perfluorohexanoic acid |
| PFHxS | perfluorohexanesulfonic acid |
| PFNA | perfluoronanoic acid |
| PFOA | perfluorooctanoic acid |
| PFOS | perfluorooctane sulfonate |
| PG | Professional Geologist |
| PM | Project Manager |
| PMP | Project Management Professional |
| POC | point of contact |
| ppm | parts per million |
| PST | Pacific Standard Time |
| PVC | polyvinyl chloride |

| | |
|----------------|--|
| PWE | Public Works – Environmental Division |
| QA | quality assurance |
| QAPP | Quality Assurance Project Plan |
| QC | quality control |
| QP | quality procedure |
| QSM | Quality Systems Manual |
| r ² | correlation coefficient |
| RCRA | Resource Conservation and Recovery Act |
| Rev. | Revision |
| RF | response factor |
| RG | Registered Geologist |
| RI | remedial investigation |
| ROE | right of entry |
| RPD | relative percent difference |
| RSD | relative standard deviation |
| RSL | regional screening level |
| RT | retention time |
| S | Sampling |
| SAIC | Science Applications International Corporation |
| SDG | sample delivery group |
| SEDD | Staged Electronic Data Deliverable |
| SERES | SERES Engineering & Services, LLC |
| Shapiro | Shapiro and Associates, Inc. |
| SI | site inspection |
| SOP | standard operating procedure |
| SSHO | Site Safety and Health Officer |
| SSHP | Site Safety and Health Plan |
| SU | standard pH unit |
| SURR | surrogate |
| SVOC | semivolatile organic compound |
| SW | surface water |
| SWMU | Solid Waste Management Unit |
| TBD | to be determined |
| TCDCA | sodium taurochenodeoxycholate |
| TDCA | taurodeoxycholic acid |
| TGI | technical guidance instructions |
| TIC | total inorganic carbon |
| TOC | total organic carbon |
| TPP | technical project planning |
| Tt | Tetra Tech |
| TUDCA | tauroursodeoxycholic acid |
| UCMR3 | Third Unregulated Contaminant Monitoring Rule |
| UFP | Uniform Federal Policy |
| U.S. | United States |
| USACE | United States Army Corps of Engineers |
| USAEC | United States Army Environmental Command |
| USEPA | United States Environmental Protection Agency |
| UTM | Universal Transverse Mercator |
| UU | Unlimited Use |
| UE | Unrestricted Exposure |

| | |
|---------|--|
| VAP | vertical aquifer profiling |
| VOA | volatile organic analysis |
| VOC | volatile organic compound |
| Ecology | Washington State Department of Ecology |
| WC | wet chemistry |
| WGS | World Geodetic System |
| YTC | Yakima Training Center |

INTRODUCTION

This Uniform Federal Policy (UFP)-Quality Assurance Project Plan (QAPP) addresses the per- and polyfluoroalkyl substances (PFAS) Remedial Investigation (RI) for various areas of interest (AOIs) identified at Yakima Training Center (YTC), in south-central Washington. The AOIs were identified during the Preliminary Assessment (PA) and Site Inspection (SI) phase (Arcadis U.S., Inc. [Arcadis], 2021) of the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) process. Historical releases of PFAS (i.e., perfluorooctane sulfonate [PFOS], and perfluorooctanoic acid [PFOA], and other PFAS) into the environment have resulted from the United States (U.S.) Department of Defense (DoD) activities at YTC. The objectives of this QAPP are to generate project data that are technically defensible and useful for meeting the project goals. These goals are to conduct an RI at YTC in accordance with CERCLA and that characterizes the nature and extent of PFAS in impacted environmental media through prescriptive and adaptive sampling programs, estimate exposure and health risks to exposed human and ecological receptors that have contacted impacted media, and delineate the extent of PFAS that has migrated off-post.

This UFP-QAPP addresses six primary elements:

- Project management
- General conceptual site model (CSM) description based on the preliminary understanding of site conditions
- Site-specific investigation design, health and safety, and data acquisition to determine the nature and extent of PFAS in various environmental media
- Measurement and data acquisition
- Assessment and oversight; and
- Data validation and usability.

Advanced Environmental Laboratories (AEL) is the selected analytical laboratory for this project. AEL will follow the analytical methodologies presented in this UFP-QAPP and has been accredited by the Environmental Laboratory Accreditation Program (ELAP). All analyses will be performed in accordance with the DoD Quality Systems Manual (QSM), Version 5.3 (DoD, 2019). Copies of AEL's DoD ELAP certifications and laboratory standard operating procedures (SOPs) and relevant laboratory information are provided in **Appendix A**.

This RI was developed in accordance with United States Environmental Protection Agency (USEPA) Guidance for Conducting RIs and Feasibility Studies (FSs) under CERCLA (USEPA, 1988) and this UFP-QAPP incorporates quality assurance/quality control (QA/QC) requirements from the following documents:

- *USEPA Requirements for Quality Assurance Project Plans, USEPA QA/R-5*, March (USEPA, 2001).
- *USEPA UFP for QAPPs*, Final Version, March (USEPA, 2005).
- *USEPA Guidance on QAPPs, CIO-2106-G-05*, January (USEPA, 2012a).
- *USEPA Optimized UFP for QAPPs*, Final Version, March (USEPA, 2012b).
- *USEPA Guidance on Systematic Planning Using the Data Quality Objectives (DQO) Process, USEPA QA/G-4, EPA/240/B-06/001*, February (USEPA 2006).
- DoD QSM, Version 5.4, October 2021, and later versions as they become available (DoD 2021,)

In 2010, a subgroup of members from the participating agencies was established to review and optimize

the UFP-QAPP worksheets in close coordination with the USEPA update of QA/G-5 (i.e., CIO-2106-G-05), Intergovernmental Data Quality Task Force [IDQTF], 2012). The optimized worksheet format is used for this QAPP. **Table 1-1** provides a crosswalk between the optimized worksheet numbers and titles and the CIO-2106-G-05 guidance.

Table 1-1: UFP-QAPP Worksheets

| Optimized UFP-QAPP Worksheets | | | |
|-------------------------------|---|---------------------------------|--|
| Worksheet # | Worksheet Title | 21-6-G-05 QAPP Guidance Section | |
| 1 & 2 | Title and Approval Page | 2.2.1 | Title, Version, and Approval/Sign-Off |
| 3 & 5 | Project Organization and Quality Assurance Project Plan Distribution | 2.2.3 | Distribution List |
| | | 2.2.4 | Project Organization and Schedule |
| 4, 7 & 8 | Personnel Qualifications and Sign-off Sheet | 2.2.1 | Title, Version, and Approval/Sign-Off |
| | | 2.2.7 | Special Training Requirements and Certification |
| 6 | Communication Pathways | 2.2.4 | Project Organization and Schedule |
| 9 | Project Planning Session Summary | 2.2.5 | Project Background, Overview, and Intended Use of Data |
| 10 | Conceptual Site Model | 2.2.5 | Project Background, Overview, and Intended Use of Data |
| 11 | Project/Data Quality Objectives | 2.2.6 | Data/Project Quality Objectives and Measurement Performance Criteria |
| 12 | Measurement Performance Criteria | 2.2.6 | Data/Project Quality Objectives and Measurement Performance Criteria |
| 13 | Secondary Data Uses and Limitations | Chapter 3 | QAPP Elements for Evaluating Existing Data |
| 14 & 16 | Project Tasks & Schedule | 2.2.4 | Project Organization and Schedule |
| 15 | Project Action Limits and Laboratory-Specific Detection / Quantitation Limits | 2.2.6 | Data/Project Quality Objectives and Measurement Performance Criteria |
| 17 | Sampling Design and Rationale | 2.3.1 | Sample Collection Procedure, Experimental Design, and Sampling Tasks |
| 18 | Sampling Locations and Methods | 2.3.1 | Sample Collection Procedure, Experimental Design, and Sampling Tasks |
| | | 2.3.2 | Sampling Procedures and Requirements |
| 19 & 30 | Sample Containers, Preservation, and Hold Times | 2.3.2 | Sampling Procedures and Requirements |
| 20 | Field QC | 2.3.5 | QC Requirements |
| 21 | Field SOPs | 2.3.2 | Sampling Procedures and Requirements |
| 22 | Field Equipment Calibration, Maintenance, Testing, and Inspection | 2.3.6 | Instrument/Equipment Testing, Calibration and Maintenance Requirements, Supplies and Consumables |
| 23 | Analytical SOPs | 2.3.4 | Analytical Methods Requirements and Task Description |
| 24 | Analytical Instrument Calibration | 2.3.6 | Instrument/Equipment Testing, Calibration and Maintenance Requirements, Supplies and Consumables |
| 25 | Analytical Instrument and | 2.3.6 | Instrument/Equipment Testing, Calibration |

| Optimized UFP-QAPP Worksheets | | | |
|--------------------------------------|--|--|--|
| Worksheet # | Worksheet Title | 21-6-G-05 QAPP Guidance Section | |
| | Equipment Maintenance, Testing, and Inspection | | and Maintenance Requirements, Supplies and Consumables |
| 26 & 27 | Sample Handling, Custody, and Disposal | 2.3.3 | Sample Handling, Custody Procedures, and Documentation |
| 28 | Analytical QC and CA | 2.3.5 | QC Requirements |
| 29 | Project Documents and Records | 2.2.8 | Documentation and Records Requirements |
| 31, 32 & 33 | Assessments and CA | 2.4 | Assessments and Data Review |
| | | 2.5.5 | Reports to Management |
| 34 | Data Verification and Validation Inputs | 2.5.1 | Data Verification and Validation Targets and Methods |
| 35 | Data Verification Procedures | 2.5.1 | Data Verification and Validation Targets and Methods |
| 36 | Data Validation Procedures | 2.5.1 | Data Verification and Validation Targets and Methods |
| 37 | Data Usability Assessment | 2.5.2 | Quantitative and Qualitative Evaluations of Usability |
| | | 2.5.3 | Potential Limitations on Data Interpretation |
| | | 2.5.4 | Reconciliation with Project Requirements |

Notes:

CA – Corrective Action

QC – Quality Control

SOP – Standard Operating Procedure

WORKSHEETS #1 & 2: TITLE AND APPROVAL PAGE

(UFP-QAPP Manual Section 2.1)
(USEPA 2106-G-05 Section 2.2.1)

1. Project Identifying Information

- a. **Site name/project name:** U.S. Army Environmental Command (USAEC) PFAS RI at Yakima Training Center, Washington
- b. **Site location/number:** Yakima Training Center, Washington, U.S.
- c. **Contract/Work assignment number:** W9124J-18-D-0004 / W9124J-22-F-0144

2. Investigative Organization – Environmental Chemical Corporation (ECC)

- a. Audra Balson, Professional Geologist (PG), Project Manager (PM)

Signature

Date

- b. Grace Carmichael, Assistant PM

Signature

Date

3. Lead Organization: USAEC

- a. Roger Walton, Professional Engineer (PE), Contracting Officer's Representative (COR)

Signature

Date

- b. Michael Brown, Environmental Support Manager (ESM) and Technical Point of Contact (POC)

Signature

Date

4. Support Organizations:

- a. Eric Brouwer, Director, U.S. Army; YTC Directorate of Public Works – Environmental Division (PWE)

Signature

Date

b. Bethany Mills, Acting Chief, Environmental and Natural Resources Division, YTC, PWE

 Signature

 Date

5. List plans and reports from previous investigations relevant to this project

| Title | Date |
|---|----------------|
| USEPA. 2017. <i>Occurrence Data for the Unregulated Contaminant Monitoring Rule: UCMR3 (2013 to 2015) Occurrence Data</i> . January. Available online at: https://www.epa.gov/dwucmr/occurrence-data-unregulated-contaminant-monitoring-rule | 2017 |
| Department of the Army. 2018. <i>MEMORANDUM – Army Guidance for Addressing Releases for Per- and Polyfluorinated Compounds</i> . | September 2018 |
| Arcadis. 2019. <i>Final Preliminary Assessment of Per- and Polyfluoroalkyl Substances, Yakima Training Center, Yakima Washington</i> . October. | October 2019 |
| Arcadis. 2020. <i>Final Uniform Federal Policy-Quality Assurance Project Plan Addendum, USAEC Per- and Polyfluoroalkyl Substances for Preliminary Assessments and Site Inspections, Yakima Training Center, Washington</i> . August. | August 2020 |
| Arcadis. 2021. <i>Final Preliminary Assessment and Site Inspection of Per- and Polyfluoroalkyl Substances, Yakima Training Center, Yakima, Washington</i> . October. | October 2021 |
| Office of the Secretary of Defense. 2022. <i>Memorandum: Investigating Per- and Polyfluoroalkyl Substances within the Department of Defense Cleanup Program</i> . | July 2022 |

Notes:

- Arcadis – Arcadis U.S., Inc.
- UCMR3 – Third unregulated contaminant monitoring rule
- USAEC – United States Army Environmental Command
- USEPA – United States Environmental Protection Agency

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WORKSHEETS #3 & 5: PROJECT ORGANIZATION AND UFP-QAPP DISTRIBUTION (UFP-QAPP Manual Section 2.3 and 2.4) (USEPA 2106-G-05 Section 2.2.3 and 2.2.4)

This worksheet identifies the programmatic key personnel, as well as lines of authority and lines of communication for USAEC, YTC, ECC, and Arcadis personnel (**Figure 3-1**). The government team members, along with their contact information, are presented in **Table 3-1**. The ECC and Arcadis team members are presented in **Table 3-1** along with their contact information.

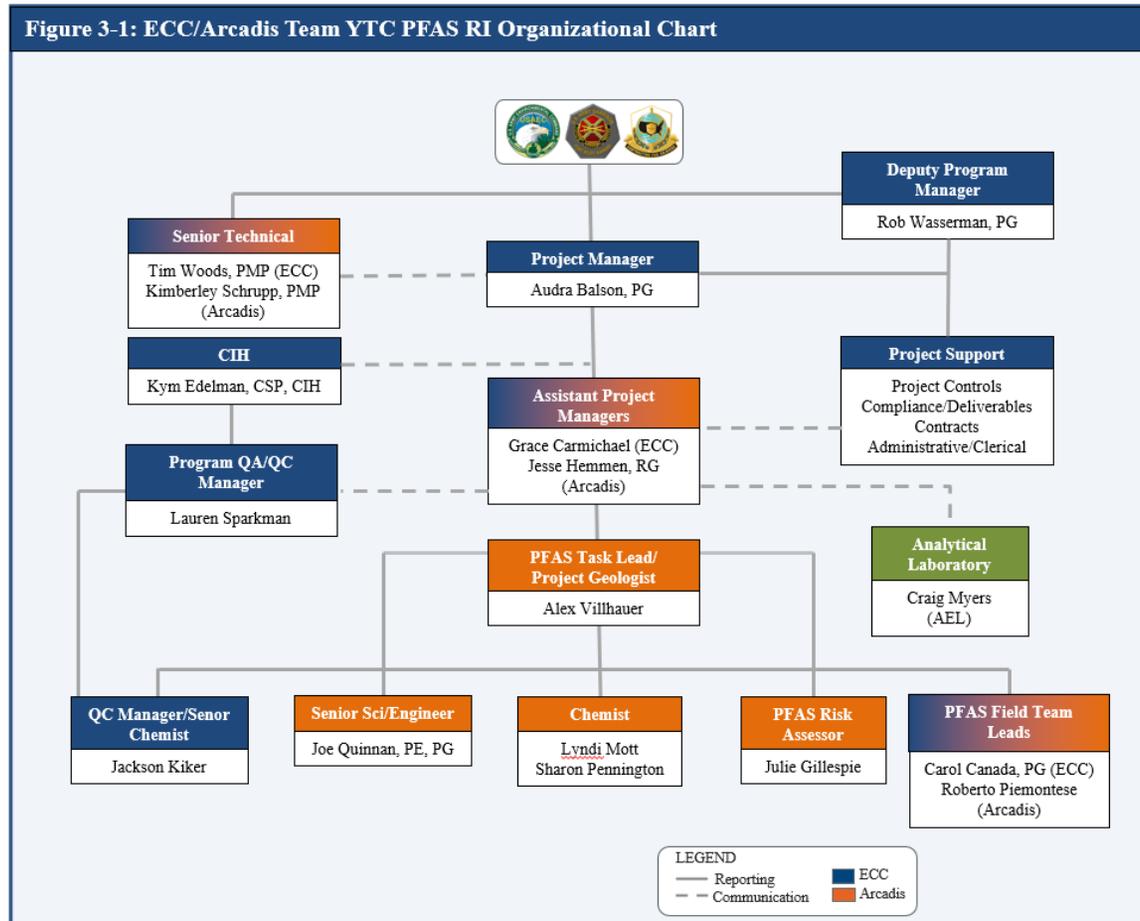


Table 3-1: ECC/Arcadis Project Team Personnel Roles and Responsibilities

| Name | Organization | Roles/Responsibilities | Telephone Number | E-mail address |
|------------------------------|---|---|--|--|
| Audra Balson, PG | ECC | ECC PM, overall communication and contracting, client communication | Cell: (717) 940-8808 | Abalson@ecc.net |
| Tim Woods, PMP | | Regulatory Specialist: regulatory support, deliverable review | Cell: (484) 343-8721 | Twoods@ecc.net |
| Rob Wasserman, PG | | ECC Program Manager: Army and programmatic support | Cell: (703) 785-6436 | Rwasserman@ecc.net |
| Jesse Hemmen, RG | Arcadis | Deputy PM – Task Lead: day to day project coordination, assists the PM | Cell: (503) 449-0778 | Jesse.Hemmen@arcadis.com |
| Rhonda Stone, PMP | | PFAS Technical/Regulatory Specialist: regulatory and programmatic support | Office: (610) 563-6122 | Rhonda.Stone@arcadis.com |
| Kimberley Schrupp, PMP | | PFAS Task Lead: day to day project coordination, deliverable review | Cell: (303) 916-1193 Office: (720) 344-3712 | Kimberley.Schrupp@arcadis.com |
| Alex Villhauer | | PFAS Technical Expert: subject matter expert, deliverable technical review | Cell: (517) 324-5036 | Alex.Villhauer@arcadis.com |
| Joe Quinnan, PE, PG | | Senior Engineer: subject matter expert, deliverable technical review | Cell: (810) 225-1943 | Joseph.Quinnan@arcadis.com |
| Lyndi Mott | | Project Chemist: data management, data validation | Cell: (713) 953-4829 | Lyndi.Mott@arcadis.com |
| Sharon Pennington | | | Cell: (865) 924-6930 | Sharon.Pennington@arcadis.com |
| Julie Gillespie | | Senior Risk Assessor: CSM development | Office: (973) 800-8094 | Julie.Gillespie@arcadis.com |
| Grey Coppi, CIH | | Program H&S Manager: H&S document review and development, safety compliance | Cell: (908) 917-6948 | Grey.Coppi@arcadis.com |
| Courtney Bigelow | | ECC | Task Lead: day to day laboratory/project coordination | Cell: (415) 404-5375 |
| Leili Arjomand, PE, CQM, PMP | Technical QA/QC Manager: companywide QA/QC compliance | | Cell: (650) 208-5482 | Larjomand@ecc.net |
| Lauren Sparkman | Project QA Officer: QA/QC management and compliance | | Cell: (808) 479-0668 | Lsparkman@ecc.net |
| Kym Edelman, CIH, CSP | CIH: H&S document review and development, safety compliance | | Cell: (757) 435-5384 | Kedelman@ecc.net |
| Jackson Kiker | QC Manager/Senior Chemist: data management review, data validation review | | Office: (508) 229-2270 x22124 Cell: (774) 245-0904 | Jkiker@ecc.net |
| Carol Canada, PG | Field Lead | | Cell: (615) 693-9915 | Ccanada@ecc.net |

| Name | Organization | Roles/Responsibilities | Telephone Number | E-mail address |
|-------------------------------|--------------|--|----------------------|--|
| Roberto Piemontese | Arcadis | Field Team Leader: field team and subcontractor management | Cell: (510) 328-5660 | Roberto.Piemontese@arcadis.com |
| Additional Field Team Leaders | TBD | | | |

Notes:

Arcadis – Arcadis, U.S., Inc.
 CIH – Certified Industrial Hygienist
 CQM – Construction Quality Manager
 CSM – Conceptual Site Model
 CSP – Certified Safety Professional
 ECC – Environmental Chemical Corporation
 H&S – Health and Safety
 PE – Professional Engineer

PFAS – per- and polyfluoroalkyl substances
 PG – Professional Geologist
 PM – Project Manager
 PMP – Project Management Professional
 QA – Quality Assurance
 QC – Quality Control
 RG – Registered Geologist
 TBD – to be determined

WORKSHEET #4, 7 & 8: PERSONNEL QUALIFICATIONS AND SIGN-OFF SHEET

**(UFP-QAPP Manual Sections 2.3.2 – 2.3.4)
 (USEPA 2106-G-05 Section 2.2.1 and 2.2.7)**

This worksheet is used to identify key project personnel for each organization performing tasks defined in this UFP-QAPP.

LEAD ORGANIZATION: USAEC

| Name | Agency | Project Title/Role | Signature* |
|------------------|--------|--------------------------------------|--------------------------|
| Roger Walton, PE | USAEC | Contracting Officer's Representative | <input type="checkbox"/> |
| Michael Brown | USAEC | Environmental Support Manager | <input type="checkbox"/> |

Note: * Signature check boxes indicate that personnel have read and agree to implement this UFP-QAPP as written.

PE – Professional Engineer

USAEC – United States Army Environmental Command

LEAD ORGANIZATION: YTC

| Name | Agency | Project Title/Role | Signature* |
|---------------|------------------------|--------------------------------------|--------------------------|
| Eric Brouwer | Yakima Training Center | Director of Public Works | <input type="checkbox"/> |
| Bethany Mills | Yakima Training Center | Interim Environmental Division Chief | <input type="checkbox"/> |

Note: * Signature check boxes indicate that personnel have read and agree to implement this UFP-QAPP as written.

SUPPORTING ORGANIZATION: ECC (Prime Contractor), Arcadis (Subcontractor), and Advanced Environmental Laboratories, Inc. (AEL).

| Name | Project Title/Role | Education/Experience | Specialized Training/Certifications | Signature* |
|--------------------|-----------------------|---|-------------------------------------|--------------------------|
| <i>ECC (Prime)</i> | | | | |
| Audra Balson | PM | B.S. and M.S. Geology 19 years of experience; 11 years of management experience | • PG | <input type="checkbox"/> |
| Rob Wasserman | Program Manager | B.S. – Geology 25 years of experience; 16 years of management experience | • PG | <input type="checkbox"/> |
| Tim Woods | Regulatory Specialist | MPH 24 years of experience; 10 years of management experience | • PMP | <input type="checkbox"/> |
| Courtney Bigelow | Task Lead | B.S. – Biology 5 years of experience | | <input type="checkbox"/> |
| Leili Arjomand | QA/QC Manager | Master of Business Administration 26 years of civil and environmental industry experience; 24 years of expertise as Quality Manager for DoD and USACE programs | • PE • CQM • PMP | <input type="checkbox"/> |

| Name | Project Title/Role | Education/Experience | Specialized Training/ Certifications | Signature* |
|---------------------------------------|-----------------------|--|---|--------------------------|
| Kym Edelman | CIH | B.S. – Environmental Health, Industrial Hygiene Concentration 29 years of environmental and construction, government, and private sector work | <ul style="list-style-type: none"> • CIH • CSP | <input type="checkbox"/> |
| Jackson Kiker | Chemical QA Manager | M.S. – Analytical Chemistry; B.S. – Chemistry 21 years of experience | | <input type="checkbox"/> |
| Carol Canada | Field Lead (SSHO) | B.S. Geology, 16 years of experience | <ul style="list-style-type: none"> • PG | <input type="checkbox"/> |
| Grace Carmichael | Grace Carmichael | B.S. Geology, 8 years of experience | | <input type="checkbox"/> |
| <i>Arcadis (Subcontractor)</i> | | | | |
| Jesse Hemmen | Deputy PM | B.A. and M.S. Geology 19 years of consulting experience | <ul style="list-style-type: none"> • RG • OSHA: Initial 40-Hour HAZWOPER • OSHA: HAZWOPER 8-Hour Refresher 29 CFR 1910.120(e)(8) | <input type="checkbox"/> |
| Kimberley Schrupp | PFAS Task Lead | B.S. – Biochemistry 22 years of management experience for Hazardous, Toxic and Radioactive Waste projects USAEC Environmental Remediation | <ul style="list-style-type: none"> • PMP | <input type="checkbox"/> |
| Julie Gillespie | Senior Risk Assessor | B.S. – Natural Resource Management; M.S. – Environmental Policy Studies 18 years of experience conducting CERCLA human health and ecological risk assessments for federal/ industrial clients | <ul style="list-style-type: none"> • OSHA: Initial 40-Hour HAZWOPER • OSHA: HAZWOPER 8-Hour Refresher 29 CFR 1910.120(e)(8) | <input type="checkbox"/> |
| Alex Villhauer | PFAS Technical Expert | B.S. - Geology 12 years of experience involving CERCLA reporting, site characterization, CSM development, and task managing | <ul style="list-style-type: none"> • OSHA: Initial 40-Hour HAZWOPER • OSHA: HAZWOPER 8-Hour Refresher 29 CFR 1910.120(e)(8) | <input type="checkbox"/> |
| Grey Coppi | Program H&S Manager | M.S. – Industrial Hygiene 30 years of experience with site safety audits/reviews for USACE projects; development of SSHPs and APPs. | <ul style="list-style-type: none"> • CIH • CSP | <input type="checkbox"/> |
| Lyndi Mott | Project Chemist | B.S. – Chemistry 40 years of experience with more than 17 years of project chemistry experience in coordination with AFCEC, USAEC, USACE, and USEPA. | | <input type="checkbox"/> |

| Name | Project Title/Role | Education/Experience | Specialized Training/Certifications | Signature* |
|--------------------------------|------------------------|---|-------------------------------------|--------------------------|
| Sharon Pennington | Project Chemist | B.S. – Chemistry 39 years of experience with more than 25 years of project chemistry experience in coordination with Air Force Civil Engineer Center, USAEC, USACE, and USEPA. | | <input type="checkbox"/> |
| <i>AEI (Laboratory)</i> | | | | |
| Craig Myers | Client Service Manager | A.S. Environmental Sciences – 28 years of total experience | N/A | <input type="checkbox"/> |
| Heather Quilan-lan | QA Manager | B.A. Biology and Psychology (Minor) | N/A | <input type="checkbox"/> |

Notes:

* Signature check boxes indicate that personnel have read and agree to implement this UFP-QAPP as written. Training records will be maintained by the ECC YTC project-specific repository.

AEI – Advanced Environmental Laboratories, Inc.

AFCEC – Air Force Civil Engineer Center

APP – Accident Prevention Plan

Arcadis – Arcadis U.S., Inc.

A.S. – Associates of Science

B.A. – Bachelor of Arts

B.S. – Bachelor of Science

CERCLA – Comprehensive Environmental Response Compensation and Liability Act

CFR – Code of Federal Regulations

CIH – Certified Industrial Hygienist

CQM – Construction Quality Manager

CSM – Conceptual Site Model

CSP – Certified Safety Professional

DoD – Department of Defense

ECC – Environmental Chemical Corporation

HAZWOPER – Hazardous Waste Operations and Emergency Response

H&S – Health and Safety

MPH – Master of Public Health

M.S. – Master of Science

N/A – not applicable

OSHA – Occupational Safety and Health Administration

PE – Professional Engineer

PFAS – per- and polyfluoroalkyl substances

PG – Professional Geologist

PM – Project Manager

PMP – Project Management Professional

RG – Registered Geologist

QA – Quality Assurance

QAPP – Quality Assurance Project Plan

QC – Quality Control

SSHO – Site Safety and Health Officer

SSHP – Site Safety and Health Plan

UFP – Uniform Federal Policy

USACE – U.S. Army Corps of Engineers

USAEC – U.S. Army Environmental Command

USEPA – U.S. Environmental Protection Agency

WORKSHEET #6: COMMUNICATION PATHWAYS

**(UFP-QAPP Manual Section 2.4.2)
 (USEPA 2106-G-05 Section 2.2.4)**

| Communication Driver | Name/Organization | Contact Information | Procedure (e.g., timing, pathway, documentation) |
|--|--|--|--|
| Technical lead decisions and modifications | Roger Walton, PE | (210) 466-1063 (Office) (210) 665-5253 (Cell) | Communicate technical lead decisions and modifications to USAEC, YTC, and/or ECC as necessary. All modifications will be included in the amendments to the UFP-QAPP by ECC and approved within seven working days. |
| Aid/support in technical decisions and modifications | Mike Brown (USAEC) Bethany Mills (YTC) (Worksheets #1 & #2, #3 & 5) | (210) 793-7896 (Cell) (509) 577-3535 (Office) | Aid in technical decisions and modifications and communicate to USAEC, YTC, and/or ECC as necessary. All modifications will be included in the amendments to the UFP-QAPP by ECC and approved within seven working days. |
| Programmatic and project issues | Rob Wasserman (ECC) | (703) 785-6436 | ECC management team will notify USAEC and YTC of any programmatic and/or project issues. |
| Minor field modifications not affecting groundwater, surface water, soil, and sediment data usability or quality | FTL (ECC or Arcadis) | (TBD) | Secure same-day verbal approval from ECC/Arcadis project team. |
| Field modifications affecting groundwater, surface water, soil, and sediment data usability or quality | Courtney Bigelow (ECC) | (415) 404-5375 | Secure same-day verbal approval from ECC/Arcadis project team. These will also include notification and/or approval from USAEC and YTC POCs (as necessary). If/When the USAEC and YTC POCs cannot be reached for approval in a timely matter as to not affect the field schedule, notification may be sufficient via email and/or voicemail. ECC/Arcadis Field Lead or PM will secure approval for modifications to the UFP-QAPP as necessary from USAEC and YTC. All modifications will be included in the amendments to the UFP-QAPP and approved within seven working days. |
| Field progress reports | (ECC) PM | Varies by Task | ECC PM will send field progress reports via email daily to the USAEC and YTC POCs. |
| Stop work due to safety issues | FTL (ECC or Arcadis) | (TBD) | Work may be stopped at any time for any safety concern. Persons other than the responsible entity may also stop work for safety concerns. USAEC and YTC will be notified by the ECC/Arcadis Field Lead or PM within one hour of any significant safety-related work stoppages and will be consulted before re-starting work. |
| UFP-QAPP changes before field work | Audra Balson, PG (ECC) Jesse Hemmen, RG (Arcadis) | (610) 505-6533 (503) 449-0778 | Submit documented amendments within 10 working days for transmittal to USAEC and YTC for approval. |

| Communication Driver | Name/Organization | Contact Information | Procedure (e.g., timing, pathway, documentation) |
|---|---|--|---|
| UFP-QAPP addendum changes during project execution | FTL (ECC or Arcadis) Audra Balson, PG (ECC) Jesse Hemmen, RG (Arcadis) | TBD (717) 940-8808 (503) 449-0778 | Secure same-day approval from ECC/Arcadis Field Team Lead. Field Team Lead or PM will secure approval for modifications to the UFP-QAPP as necessary from USAEC and YTC. When the USAEC and YTC POCs cannot be reached for approval in a timely matter as to not affect the field schedule, notification may be sufficient via email and/or voicemail. All modifications will be included in the amendments to the UFP-QAPP and approved within seven working days. |
| Field Corrective Actions | FTL (ECC or Arcadis) Audra Balson, PG (ECC) Kimmie Schrupp, PMP (Arcadis) | TBD (717) 940-8808 (303) 916-1193 | The ECC/Arcadis Field Team communicates stop work immediately to the ECC PM by phone followed by inclusion in daily field progress report. Resolution of the corrective action will be determined by the ECC/Arcadis Field Lead/PM in consultation with USAEC and YTC may be documented by email, depending on significance. Work will be allowed to start once all parties have agreed to the resolution. When USAEC and/or YTC cannot be reached for approval in a timely matter as to not affect the field schedule, notification may be sufficient via email and voicemail. |
| Sample receipt variances | Courtney Bigelow (ECC) | (415) 404-5375 | All project field samples variance issues will be reported by the laboratory to the ECC PM or designee within two business days of identification of the technical concern. |
| Laboratory QC variances | Courtney Bigelow (ECC) | (415) 404-5375 | All QA/QC issues with project field samples will be reported by the laboratory to the ECC PM or designee within two business days of identification of the technical concern. |
| Analytical CAs | Jackson Kiker (ECC) Lyndi Mott (Arcadis) Sharon Pennington (Arcadis) | (774) 245-0904 (713) 953-4829 (865) 924-6930 | The need for laboratory CAs will be determined by the Project Chemist and ECC PM (or designee) and/or Laboratory PM, as appropriate, and will be documented in a memorandum to the PM and Technical QC Manager. The PM will notify USAEC and YTC if the changes to the data impact reports/data that have already been submitted. Otherwise, the memorandum will be included with the validated data. |
| Data verification issues (e.g., incomplete records) | Jackson Kiker (ECC) Lyndi Mott (Arcadis) Sharon Pennington (Arcadis) | (774) 245-0904 (713) 953-4829 (865) 924-6930 | All verification issues will be reported by the laboratory to the ECC PM (or designee) and Project Chemist via email within 24 hours of identification of the technical concern. The Technical QC Manager will be notified of the issue by the PM or Chemist and will take appropriate action if necessary. |
| Data validation issues (e.g., non-compliance with procedures) | Lyndi Mott (Arcadis) Sharon Pennington (Arcadis) Jackson Kiker (ECC) | (713) 953-4829 (865) 924-6930 (774) 245-0904 | All validation issues will be reported by the Data Validator to the Senior Chemist and PM via email within 24 hours of identification of the technical concern. The Technical QC Manager will be notified of the issue by the Data Validator and/or Senior Chemist and will take appropriate action if necessary. |

| Communication Driver | Name/Organization | Contact Information | Procedure (e.g., timing, pathway, documentation) |
|----------------------|--|--|--|
| Data review CAs | Jackson Kiker (ECC) Lyndi Mott (Arcadis) Sharon Pennington (Arcadis) | (774) 245-0904 (713) 953-4829 (865) 924-6930 | The need for data review CAs will be determined by the Senior Chemist and PM and/or Laboratory PM, as appropriate, and will be documented via email to the PM. The Technical QC Manager will be notified of the issue by the PM and will take appropriate action if necessary. |

Notes:

Arcadis – Arcadis U.S., Inc.
 CA – Corrective Action
 ECC – Environmental Chemical Corporation
 FTL – Field Team Leader
 PE – Professional Engineer
 PFAS – per- and polyfluoroalkyl substances
 PG – Professional Geologist
 PM – Project Manager
 PMP – Project Management Professional

POC – Point of Contact
 RG – Registered Geologist
 QA – Quality Assurance
 QAPP - Quality Assurance Project Plan
 QC – Quality Control
 TBD – to be determined
 UFP – Uniform Federal Policy
 USAEC – United States Army Environmental Command
 YTC – Yakima Training Center

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WORKSHEET #9: PROJECT PLANNING SESSION SUMMARY

(UFP-QAPP Manual Section 2.5.1)
(USEPA 2106-G-05 Section 2.2.5)

Project planning sessions are summarized throughout this worksheet. Project planning materials, including attendance lists, discussion details, and presentation materials, are provided in **Appendix B**.

Project Kick-off Teleconference Call

Date/Time of Planning Session: 14 October 2022, 10:00 – 11:00 Eastern Standard Time (EST)

Location: Conference call

Purpose: Project planning session with USAEC, YTC, and ECC/Arcadis.

Summary of discussion topics: See **Appendix B**.

Documented Changes since Planning Session: See **Appendix B**.

Project Scoping Call I

Date/Time of Planning Session: 9 January 2023, 09:00 – 11:00 Pacific Standard Time (PST)

Location: YTC, Directorate of Public Works

Purpose: Project planning session with USAEC, YTC, and ECC/Arcadis.

Summary of discussion topics: See **Appendix B**.

Documented Changes since Planning Session: See **Appendix B**.

Project Scoping Call II

Date/Time of Planning Session: 10 January 2023, 09:00 - 12:00 PST

Location: Washington State Department of Ecology (Ecology) – Union Gap, Washington office

Purpose: Project planning session with USAEC, Ecology, YTC, and ECC/Arcadis.

Summary of discussion topics: See **Appendix B**.

Documented Changes since Planning Session: See **Appendix B**.

The UFP-QAPP Acknowledgement Form is provided as **Appendix C**.

WORKSHEET #10: CONCEPTUAL SITE MODEL

(UFP-QAPP Manual Section 2.5.2) (USEPA 2106-G-05 Section 2.2.5)

This worksheet includes an overview of the CSM for YTC and presents the location and relevant current or historical operations for each AOI, physical setting including topography, climate, geology, and hydrogeology, known or suspected sources of PFAS, transport mechanisms, PFAS extents, and potential receptors and exposure pathways.

This RI is being conducted for YTC in Yakima, Washington (**Figure 10-1**) in accordance with the CERCLA under USAEC Contract Number W9124J18D0004, Delivery Order Number W9124J22F0144 by ECC and Arcadis. This RI addresses the characterization and on- and off-post delineation of PFAS constituents PFOS, PFOA, perfluorobutanesulfonic acid (PFBS), perfluorononanoic acid (PFNA), perfluorohexane sulfonate (PFHxS), and hexafluoropropylene oxide dimer acid (HFPO-DA) at YTC associated with the AOIs (**Figure 10-2**) previously identified in the PA/SI (Arcadis, 2021). This CSM further updates the CSM presented in the CSM Technical Memorandum (ECC/Arcadis 2023a) and incorporates data sets, analysis, and interpretations generated during the boundary investigation, which included characterization of subsurface structural geology and identification of geologic features that may potentially act as preferential groundwater and subsequently, PFAS migration pathways along YTC boundaries. The CSM provides a framework useful for identifying data needs for the RI.

Background Information

YTC (originally known as the Yakima Firing Center) is active sub-installation of Joint Base Lewis-McChord (JBLM) (located approximately 179 miles west of YTC), and encompasses 327,231 acres in central Washington, five miles north of the city of Yakima (population 97,000), in Yakima and Kittitas Counties (**Figure 10-1**). The eastern border of the facility is the Columbia River. The YTC population is primarily transient soldiers in training, with a small number of permanent party military members and civilian employees. Fewer than 500 military and civilians are permanently stationed at YTC, including active-duty service members, the Washington National Guard, the Army Reserve, and Marine Reserve members. Few population centers are situated around YTC, the largest being the city of Terrace Heights, with a population of 9,114.

Physical Setting

Topography and Climate

Information in this section is excerpted from the PA (Shapiro and Associates, Inc. [Shapiro], 1991), *Resource Conservation and Recovery Act (RCRA) Facility Assessment Report* (Science Applications International Corporation [SAIC], 1995), and *Periodic Review Report* (USACE, 2012a) for YTC. YTC is located within the Walla Walla Plateau, a sub-province of the Columbia Plateau physiographic province. The area constitutes a transitional zone between the Cascade Mountains to the west and the main part of the Columbia Plateau to the east. The Walla Walla Plateau consists of a series of southeast-trending ridges and intervening valleys; this topography is a result of folding and uplifting of basalts and interbedded sediments of the Columbia River Basalt (CRB) Group (Shapiro, 1991). Landforms in the Columbia Basin are characterized by irregular plains and table lands with moderate to high relief. Elevations on YTC vary from approximately 440 feet above mean sea level along the eastern border with the Columbia River to over 4,000 feet along some of the major east-west trending anticlinal and synclinal ridges (SAIC, 1995). Continued uplift of the plateau has allowed streams and rivers to cut deeply into the basalts, resulting in steep-sided ravines. North-south trending drainages dissect the ridges and flow parallel toward the Columbia River to the east or the Yakima River to the west. In general, the western part of the installation is rolling to hilly, and the topography becomes increasingly rugged to the east in transition down to the

Columbia River (Shapiro 1991).

YTC is located within the semiarid Columbia Basin, which is characterized by sagebrush/wheatgrass steppe and grasslands (SAIC, 1995). Precipitation is generally limited to the winter months in the form of snow and averages 8.8 inches. Winters are cool, and summers are hot and dry. Mean annual temperature is 51 degrees Fahrenheit (°F). The average January temperature is 28 °F and the average July temperature is 72 °F. Prevailing winds are from the west-northwest in both seasons and are controlled by valley trends (Shapiro, 1991). Average annual potential evapotranspiration is estimated to be between 25 and 37 inches which significantly limits local recharge to aquifers at the site from precipitation (USACE, 2012b).

Surface Water Hydrology

Information in this section is excerpted from the PA for the YTC (Shapiro 1991), the *Final RCRA Facility Assessment Report* (SAIC, 1995), and the Fort Lewis Grow the Army Final Environmental Impact Statement: Chapter 5 Affected Environment - YTC (DoD, 2010). The dominant surface water bodies in the region around YTC are the Columbia River to the east and the Yakima River to the west. Both rivers flow from north to south in the vicinity of YTC. The Yakima River flows into the Columbia River approximately 120 miles downstream from YTC. The Columbia River's flow (more than 120,000 cubic feet per second) is regulated by a series of dams. Two major hydroelectric dams (Wanapum and Priest Rapids) are located on the Columbia River near the eastern border of YTC (SAIC 1995). The Columbia River receives runoff from several streams draining from the eastern side of YTC, including Hanson Creek, Alkali Canyon Creek, Corral Canyon Creek, Sourdough Canyon Creek, and Cold Creek. The Yakima River's flow (average of approximately 2,500 cubic feet per second) is regulated by the Roza Dam in the vicinity of YTC. The Yakima River receives runoff from several streams draining from the western side of YTC, including Squaw, Burbank, and Selah Creeks. Selah Creek receives flow from several on-post ephemeral drainages and springs (e.g., Selah Springs). High evapotranspiration and low precipitation limit surface runoff from YTC. Only Alkali, Cold, and Squaw Creeks are perennial; most other creeks and drainages are ephemeral though a few are intermittent following a large storm event (Shapiro 1991). Though some flash runoff events may occur at YTC if rain falls on snow or frozen ground, flooding is not an issue within the YTC boundaries (DoD 2010). Peak surface water runoff occurs during the winter-spring snowmelt period (Shapiro 1991).

Several springs (ranging from seasonal to perennial) and seeps feed some of the local stream systems. Approximately 148 springs have been developed at YTC to provide water for agriculture and livestock. Three surface water impoundments or ponds (Kiddies Pond, Taylor Pond, and Eaton's Pond) are located at YTC, supported by earthen dams to hold water year-round. Taylor Pond has historically been used primarily in support of fire suppression activities, and Kiddies Pond serves as a fishing pond for juvenile use.

In the vicinity of YTC, two irrigation canals divert water from the Yakima River to supplement irrigation water. A seasonally operated (April through November) unlined and partially covered irrigation canal flows through the cantonment from the north, which enters the installation near the intersection of Tipp Road and Latigo Lane and flows offsite near the running track (**Figure 10-2**).

Geology and Hydrogeology

Information in this section is excerpted from the Periodic Review Report for YTC (USACE, 2012a) and the Groundwater Monitoring Report for the Fire Training Pit and Tracked Vehicle Repair/Old Mobilization and Training Equipment Site (Tetra Tech [Tt], 2017). The YTC and surrounding region is underlain by a thick sequence of basalt lava flows of the CRB Group with interbedded, weakly consolidated sediments of the Ellensburg Formation (Fm) (**Exhibit 1**). CRB lava flows underlie much of eastern Washington and have a total thickness of greater than 10,000 feet in parts of the region. Individual flows range from a few feet to more than 100 feet in thickness. Each flow typically consists of a vesicular or rubbly flow top, a relatively thick internal zone that has a hackly texture of random cooling joints, and lower zone that is characterized

by columnar jointing perpendicular to the base of the flow (USACE, 2012a).

The Ellensburg Fm is composed of partially consolidated sand and gravel, with sediments ranging from unconsolidated sand, silt, and clay to weakly indurated sandstone, siltstone, and claystone. These sediments range from a few feet to several hundred feet in thickness and are generally thickest underlying lowland areas. Younger deposits that locally overlie the Ellensburg Fm and the CRB in the YTC area include unconsolidated quaternary alluvial sand and gravel along the stream channels and floodplains, alluvial fan deposits of silty sand and gravel along the flanks of the ridges, and windblown silt (loess) deposits (USACE, 2012a).

YTC is situated in a tectonically active zone known as the Yakima Fold Belt of south-central Washington, also referred to as the Yakima Fold and Thrust Belt. Within the Yakima Fold Belt, the CRB and Ellensburg Fm have been deformed into a series of east-northeast-trending anticlines and synclines. Owing to the relatively young age of this deformation, the anticlines are expressed as ridges and intervening synclines form valleys. The YTC cantonment area is mostly located within the synclinal valley between the anticlinal Yakima Ridge to the south and Umtanum Ridge to the north (Tt, 2017).

Extensive folding of the sedimentary and CRB strata in the area has created a complex groundwater system with highly variable hydraulic properties, depths to groundwater, and flow directions at any given location at YTC. Groundwater in the region occurs largely within the following principal aquifers (not all are present everywhere across YTC): surficial unconsolidated alluvial deposits, sedimentary units (principally the sand and gravels) of the Ellensburg Fm, the Saddle Mountains Basalt, the Wanapum Basalt, and the Grande Ronde Basalt (Tt, 2017).

The alluvial deposits are typically moderately to highly permeable, and groundwater within them generally is unconfined. The water table in these deposits is typically at or near the elevation of the nearby streams. In the Ellensburg Fm, groundwater is found in the gravel layers within the surficial sedimentary formations and can be either unconfined or confined by overlying finer-grained materials, depending on the thickness and composition of the formation. Within the sequences of basalt, groundwater is predominantly found within the weathered, more fractured contact zones and within sedimentary interflow zones (Tt, 2017 and USACE, 2012a). The basalt flows and associated sedimentary interbeds form the most productive aquifer system in the region. Groundwater within this system occurs principally within fracture and rubble zones of the basalt flows and in the sand and gravel layers that occur between some of the flows. The water-

Exhibit 1. Stratigraphy of sedimentary interbeds and Columbia River Basalt Group (Reidel *et al.*, 2020).

| Approximate Age (Ma) | Formation | Members | | |
|------------------------|-----------------------------|--|-------------------------|---------------------|
| 0.07 | Loess (Ql) | | | |
| | Alluvium (Qa) | | | |
| | Landslide (Qld) | | | |
| 2.5 | Colluvium (Qco) | | | |
| | Missoula Flood Deposits | Touchet Beds - (Qhz) Sand-dominated - (Qhs) Gravel-dominated - (Qhg) | | |
| 7.5 | Ringold Formation (Mrg) | | | |
| 8.5 | Columbia River Basalt Group | Walla Walla (Mww) | | |
| | | Ice Harbor (Mih) | | |
| | | Basalt of Goose Island (Mig) | | |
| | | Basalt of Martindale (Mim) | | |
| | | Basalt of Basin City (Mib) | | |
| | | Levey sediments (Mel) | | |
| | | 10 | Elephant Mountain (Mem) | |
| | | 11 | Rattlesnake Ridge (Mrr) | |
| | | Saddle Mountains Basalt | Pomona (Mp) | |
| | | | Selah (Mes) | |
| | | | Esquatzel (Me) | |
| | | | Cold Creek (Mcc) | |
| | | | Umatilla (Mu) | |
| | | 13 | Mabton (Mm) | |
| | | 16 | Wanapum Basalt | Priest Rapids (Mpr) |
| | | | | Quincy |
| Roza (Mr) | | | | |
| Squaw Creek | | | | |
| undifferentiated (Mwu) | Frenchman Springs | (Msf) Basalt of Silver Falls | | |
| | | (Msh) Basalt of Sand Hollow | | |
| | | (Mg) Basalt of Ginkgo | | |
| | | (Mpf) Basalt of Palouse Falls | | |
| | | (Msg) Basalt of Sentinel Gap | | |
| 16.7 | Grande Ronde Basalt | Vantage (Mv) | | |
| | | undifferentiated (Mgr) | | |
| | | Ellensburg Formation | | |

yielding zones within this sequence range from a few feet to over 50 feet in thickness. Their lateral extent ranges from short distances or up to several miles, depending on the stratigraphic continuity of the water-bearing unit (USACE, 2012a).

Reported depths to groundwater range from 20 feet below ground surface (bgs) in stream valleys to more than 200 feet bgs at higher elevations at YTC. Groundwater springs occur where incised stream valleys intercept aquifers (Tt, 2017). In the cantonment area of YTC, the uppermost groundwater occurs in shallow, perched zones (Selah Interbed Aquifer) in the vesiculated fractured basalt near the top or base of the Pomona Flow of the Saddle Mountain Basalt, depending on the area. Depth to groundwater can range from 10 to 100 feet bgs in the cantonment area, and the flow direction of the perched water is generally to the west and southwest, and off-post toward the Yakima River. The Selah Interbed Aquifer is underlain by a thick sequence of basalt flows within the CRB Group (Tt, 2017) and is known to exhibit localized artesian groundwater conditions typically related to structural features which act as vertical conduits and lateral flow barriers (Vaccaro *et al.*, 2009). Tables 10-1 and 10-2 summarize the well construction details for on-post potable water wells and monitoring wells, respectively, which are screened in the Selah Interbed Aquifer and CRB Group.

As part of the initial RI activities, a Boundary Investigation was conducted in accordance with the *Final Boundary Investigation Technical Memorandum Work Plan* (ECC/Arcadis, 2023b), which involved a surface geophysical survey (seismic and resistivity methods) conducted along three transects positioned parallel and adjacent to the installation boundary (**Figure 10-3**), followed by the installation, development, and sampling of eight boundary monitoring wells (**Figure 10-3**). Well Construction Logs for the eight monitoring wells are provided in **Appendix D**, and the Data Validation Reports (DVRs) associated with the Boundary Well groundwater samples are provided in **Appendix E**. Implementation of the Boundary Investigation was described in the CSM Tech Memo (ECC/Arcadis 2023a). Key geologic interpretations from the geophysical profiles show variable elevation of the basalt flow top underlying the Ellensburg Fm (**Figures 10-4 through 10-6**) and suggest significant structural deformation and/or displacement along interpreted faults. Above the basalt, the Ellensburg Fm generally exhibits low resistivity, which suggests units of lower permeability and high clay content. Notable increases in resistivity observed in sedimentary deposits within the Ellensburg Fm may be interpreted as a more permeable alluvial channel deposit (refer to anomaly A5 on **Figure 10-4**). Along Transect C (**Figure 10-6**) resistivity results show the presence of a shallow basalt unit (interpreted to be the Pomona Member) overlying the Ellensburg Fm, and a deeper basalt unit below (interpreted to be a member of the Grande Ronde Fm). High shear wave velocities and resistivity values observed in the basalts along Transect C suggest a lower permeability, competent basalt. The resistivity profile shows significant irregularity in the elevation of the bottom of the Pomona Member and may be a result of basalt flows infilling paleo-surface features, displacement due to faulting, or some combination of the two. Results of X-ray fluorescence fingerprinting of select basalt chip samples collected during well installation confirm the identification of the Pomona Basalt encountered in the shallow subsurface along Transect C and indicate that the basalt unit encountered at depth along Transects A and B likely corresponds to a flow within the Grande Ronde Fm (ECC/Arcadis, 2023a).

During advancement of the monitoring well boreholes, grab groundwater samples were collected from the first water-bearing zone for laboratory analysis of PFAS. First-encountered groundwater elevations ranged from 75 feet bgs (MW-03) to 225 feet bgs (MW-06). Following well construction and development, groundwater samples were collected from the newly installed wells using formal low-flow sampling methods. The lowest PFAS concentrations in the Boundary Investigation wells were reported in MW-08, which is screened in the perched groundwater zone, and in MW-02 and MW-05, where PFAS was reported as non-detect (ND) during the low-flow sampling event. Other notably low PFAS concentrations were detected in groundwater monitoring well samples from MW-01 (**Table 10-3**). The highest concentrations of PFAS identified during the Boundary Investigation were associated with the low-flow samples (**Table**

10-3), as follows:

- PFOS: 1,500 nanograms per Liter (ng/L) at MW-03
- PFHxS: 1,100 ng/L at MW-06
- PFNA: 16 ng/L at MW-03; and
- PFOA: 140 ng/L at MW-03.

Groundwater Use

A highly productive regional basalt aquifer underlies the cantonment area at depth. The groundwater at depth in this area occurs in basalt fractures and interbedded sediments. This flow regime is presumably recharged from a considerably higher area up-slope and is confined under pressure beneath less permeable strata consisting of basalt or fine-grained sediment (USACE, 2012a). Groundwater in the basalt aquifers generally flows westward toward the Yakima River with a more northwesterly flow component closer to the river (Ecology and Environment, Inc. 1993, and SAIC 1995).

The regional basalt aquifer serves as the primary drinking water supply for YTC, as well as the Pomona Artesian Irrigation Company water system, which provides drinking water to approximately 60 homes and businesses near the Installation. These water supply wells are screened at depths greater than 350 feet bgs. The Pomona well (operated by YTC) and the Pomona Artesian Irrigation Company well are located within the YTC cantonment area and operate under artesian conditions. The Pomona well is completed in the Wanapum and/or Grande Ronde Fm, with open borehole completion between depths of approximately 353 and 407 feet bgs. Historical surveys indicate that groundwater enters the Pomona well at approximately 401 feet bgs, along a sedimentary interbed or fracture zone (Tt, 2017). This flow system is presumably recharged from an area that is considerably higher in elevation to the east (up-slope) and under confined pressure beneath less permeable basalt or fine-grained sediment (USACE, 2012a). The upward hydraulic gradients encountered at YTC, in addition to the overlying low permeability materials that contribute to the observed artesian conditions, have thus far prevented the downward migration of contaminants from shallower aquifers.

The drinking water supply for YTC is provided entirely from groundwater sources. Six wells (i.e., Pomona, Jordan, Badger Gap, Bowers, Multi-Purpose Range Complex, and Yakima Research Station wells; **Table 10-1**) provide water for three permitted drinking water distribution systems located in the cantonment area, the Yakima Research Station, and the Multi-Purpose Range Complex (DoD, 2010). Prior to distribution and use, this water is treated, typically at the wellhead, by chlorination. Water for the permitted drinking water distribution system in the cantonment area is stored in two tanks with a combined storage capacity of 1,130,000 gallons. Additional wells designated for potable water supply are located within the range/training areas (**Figure 10-2**) and have a combined storage capacity of 415,300 gallons (DoD, 2010). Water from these remaining wells located throughout the range/training areas is treated as needed but is not part of the primary drinking water system (DoD, 2010).

A groundwater well designated for potable use is positioned southwest of the airstrip and is connected to a water stand for rapid filling of vehicles. This well is screened from approximately 73 to 91 feet bgs, which is assumed to be in the perched aquifer, though little data are available regarding the groundwater conditions in this area (U.S. Army Public Health Command, 2010). The water well and stand were shut down and designated as no-use status following the detections of PFAS constituents observed at the well and water stand in 2019.

While YTC and the Pomona Artesian Irrigation Company source drinking water from the regional basalt aquifer the majority of off-post residents within the Phase 1, 2, and 3 outreach areas shown on **Figure 10-7** rely on private wells drilled to a variety of depths and sourcing water from various discrete water bearing

zones for drinking water and other private uses.

Known or Suspected Chemicals of Concern and Sources

In May 2016, the USEPA issued a lifetime health advisory for PFOS and PFOA of 70 ng/L, individually or combined (USEPA, 2016). Subsequently, in 2016, the Army issued a guidance publication for PFAS assessments (U.S. Army 2016a, 2016b, and 2016c). In response to these actions, the third Unregulated Contaminant Monitoring Rule, and U.S. Army Installation Management Command Operations Order 16-088, Army installations began initial PFAS sampling of water supply wells in 2016.

Six potable water supply wells at YTC (Pomona, Bowers, Jordan, Multi-Purpose Range Complex, Badger Gap, and Yakima Research Station) were sampled in October 2016 and analyzed for six PFAS; results were non-detect at all six wells for all six constituents analyzed, including PFOS, PFOA, and PFBS (Arcadis, 2021).

In August 2019, 11 of the potable wells on-post were sampled for 14 PFAS; all results were ND except at the Mettie Airstrip (formerly Selah Airstrip) well. The Mettie Airstrip well is installed in the perched aquifer to a total depth of 91 feet bgs and has a static water level of approximately 47 feet bgs. In addition, samples were collected from the water stand which is supplied from the Mettie Airstrip well. The water stand detections included PFOS (4.2 ng/L), PFOA (96 ng/L), and PFBS (11 ng/L). Concentrations of PFOA at the Mettie Airstrip were greater than the USEPA lifetime health advisory and the 2022 Office of the Secretary of Defense (OSD) risk screening levels (**Appendix F**). To evaluate PFAS in groundwater at its withdrawal point, follow-up samples were collected at the Mettie Airstrip well house in November 2019; the well house samples yielded similar PFAS concentrations (Arcadis, 2022). Water supplied from the Mettie Airstrip production well (and water stand) has also been piped to buildings at the Airstrip. Currently, the well pump has been turned off and is in no-use status.

In 2019, a PA was conducted to identify Areas of Potential Interest (AOPIs) based on probable use, storage, and/or disposal of PFAS-containing materials. An SI was initiated to investigate the AOPIs in accordance with CERCLA and included multi-media sampling at AOPIs to determine the presence or absence of PFAS constituents. The results of the PA/SI, including the identification and investigation of AOPIs, and recommendations for AOIs that should be further investigated in the RI phase were presented in the *Final PA/SI of PFAS* (Arcadis, 2021). The PA/SI also identified the potential for PFAS to be present in off-post residential drinking water wells (SERES Engineering and Services, LLC [SERES]/Arcadis, 2023). **Figure 10-7** shows the area evaluated for potential PFAS impacts in residential wells during three phases of off-post residential well sampling completed during the PA/SI (SERES/Arcadis, 2023). The results of SI sampling are included in **Tables 10-3, 10-4, 10-5, and 10-6** and shown on **Figures 10-8 through 10-11**.

Nearby residents anecdotally report that brush fires have occurred at and near YTC and they observed aqueous film-forming foam (AFFF) being used to suppress the brush fires. The specific occurrences and locations of these events were not documented in the PA/SI and are unknown.

In 2023, a baseline sampling event was completed in accordance with the *Final Baseline Sampling Work Plan* (ECC/Arcadis, 2023c) to refine the understanding of the presence and extent of PFAS detected in previous sampling efforts prior to developing the UFP-QAPP. The baseline sampling included collocated surface water (if present at the time of sampling) and sediment samples at locations up-, down-, and/or cross gradient of the AOIs and groundwater samples from select monitoring wells downgradient of the Former Fire Training Pit and Bird Bath Wash Rack areas. The results of baseline sampling are included in **Tables 10-3, 10-5, and 10-6** and shown on **Figures 10-8 through 10-11**.

Areas of Interest

Three AOPIs were identified during the PA, and four additional areas were categorized as AOPIs following the PA, based on available PFAS data and at the direction of the Army. A brief history for each of the locations is provided below in **Table 10-7**. These seven AOPIs have been carried forward as AOIs for the RI and are shown on **Figure 10-2**.

| Table 10-7: Locations and Background of AOIs | | |
|---|--------------------------------------|---|
| AOI | Area Description | Relevant Site History |
| AOI 1 | Former Fire Training Pit (YFCR-53) | The YFCR-53 Former Fire Training Pit site is in the northeast portion of the cantonment area and was identified as SWMU 59 in the 1995 RCRA Facility Investigation. The site was used to practice extinguishing fires two or three times per year from an unknown start date until 1987, and a single training event in 1990. Practice events consisted of saturating an open, unlined earthen pit with water, adding and igniting 500 to 1,000 gallons of waste fuel, and then extinguishing the fire. Given the period of operation, it is suspected that AFFF was used during the firefighter training activities. During the 1990s, the site was used for storing stockpiles of waste sand filter material and sediments from the adjacent vehicle wash rack treatment system (i.e., Bird Bath Wash Rack) as well as storing fuel bladders. A RCRA Facility Investigation was conducted in 2001 to determine the extent of petroleum impacts in soil, and based on the results, a removal action of approximately 1,350 tons of petroleum-impacted soil was completed in 2003; much of the area was excavated to bedrock (approximately 8 feet; USACE 2012). The disposal location of the soil is not known; however, it has been documented that some of the excavated material was used as the excavation's own backfill and may therefore still contain PFAS. A 2016 groundwater monitoring report indicated that Teflon® bailers have been used to purge groundwater from the existing monitoring wells at the site (Tt, 2017). |
| AOI 2 | Bird Bath Wash Rack | Retired installation personnel noted historical firefighter training activities (and therefore likely AFFF use) in the area prior to construction of the wash rack facility. Google Earth aerial imagery indicates that the current wash rack structure was built sometime between 1996 and 2003; the imagery of the area from 1996 appears to show a rectangular depression or bermed pit, potentially with a prop in the center where the wash rack pad is located. The area is adjacent to the YFCR-53 Former Fire Training Pit AOPI. |
| AOI 3 | AFFF Storage Area (Building 821) | Building 821 was formerly utilized as a storage area by the YTC fire department. Approximately one pallet of AFFF (consisting of 27 to 36, 5-gallon containers) was historically stored here at the north end of the building near the loading dock. No drains exist in the building. There has been no evidence of a release. |
| AOI 4 | Refractometer Solutions Testing Area | East of the fire station storage facility, the asphalt parking lot was reportedly used for refractometer testing of mixed AFFF solutions from at least 1997 to 2004 (quarterly testing). The AFFF and water were mixed at this location, and some of the solution was discharged to the asphalt ground. Most of the solution reportedly dried up on the asphalt before it could flow to the ditch to the north of the parking lot; however, residual PFAS may have run off during precipitation events to the ditch (which flows to an oil/water separator that eventually leads to an outfall off-post). Based on its proximity to the old Tracked Vehicle Repair/Mobilization and Training Equipment Site where groundwater monitoring wells have been installed, groundwater may be expected to be encountered at 10 to 45 feet bgs near this AOPI. |
| AOI 5 | Fire Station 29 (Building 346) | Fire Station 29 is the primary fire station for the installation and houses two pumper trucks formerly equipped with AFFF. |
| AOI 6 | AFFF Storage Area (Fire | Two racks of firefighting agents including Class A foams and Class B foams remained in storage at this building at the time of the PA site visit. Some empty Class B AFFF |

| Table 10-7: Locations and Background of AOIs | | |
|--|---|--|
| AOI | Area Description | Relevant Site History |
| | Department Bay, Building 321) | containers have been repurposed to store Class A foams that have been drained from other equipment and are scheduled for disposal. The fire department noted that these Class A foams stored in Class B containers were not used elsewhere due to cross-contamination concerns. A drain exists in the building that directs wastewater to the on-post wastewater treatment plant. There has been no evidence of a release. |
| AOI 7 | Mettie Airstrip (formerly Selah Airstrip) | Following completion of the PA, the Mettie Airstrip was identified as an AOPI based on the detections of PFAS at the Mettie Airstrip water stand and well house. The area of AFFF use at the airstrip is unknown. The former crash truck station (former Building 2065) was reportedly used in the 1980s and 1990s (U.S. Army Public Health Command 2010); personnel interviews indicated that an AFFF crash truck was parked outside and stored AFFF in its tank. However, there were no reports of AFFF use, leaks, or spills. The former crash truck station building was demolished in 2016. |

Notes:

AFFF – aqueous film-forming foam
 AOI – Area of Interest
 AOPI – Area of Potential Interest
 bgs – below ground surface
 PA – Preliminary Assessment

PFAS – Per- and Polyfluoroalkyl substances
 RCRA – Resource Conservation and Recovery Act
 SWMU – Solid Waste Management Unit
 YTC – Yakima Training Center

A discussion of PFAS source areas, summary of PFAS concentrations in groundwater (Table 10-3), soil (Table 10-4), surface water (Table 10-5), and sediment (Table 10-6), as well as interpreted relevant migration pathways and the current understanding of the relationship between source area and downgradient groundwater, is included below.

AOI 1 - Former Fire Training Pit and AOI 2 - Bird Bath Wash Rack

The Former Fire Training Pit and the Bird Bath Wash Rack are directly adjacent and located in the northeast portion of the cantonment area (Figure 10-8). Sampling was conducted at the Former Fire Training Pit and the Bird Bath Wash Rack during both the SI and baseline investigation events at the locations shown on Figure 10-8. Groundwater samples collected from shallow bedrock wells in this area during the SI (Table 10-3) contained PFOS, PFOA, PFBS, PFHxS and PFNA at concentrations exceeding the risk screening levels (OSD, 2022). The highest concentrations of each PFAS constituent were identified in groundwater collected from monitoring well YTC-FTP-1 (positioned in the center of the Former Fire Training Pit): PFOS at 45,000 ng/L; PFOA at 5,200 ng/L; PFBS at 5,900 ng/L; PFHxS at 23,000 ng/L; and PFNA at 75 ng/L. Soil was not sampled at the Former Fire Training Pit or the Bird Bath Wash Rack because the ground has been significantly reworked during previous Installation Restoration Program and/or construction activities. Two baseline sediment samples collected in the irrigation canal downgradient of the AOIs (Figure 10-8) were reported as ND for PFAS (Table 10-6), which suggests that PFAS impacted groundwater is likely not discharging to the canal. Groundwater samples from monitoring wells MTS-2, -3, and -4 (considered to be downgradient of the AOIs based on perched groundwater flow directions) exhibited detections of PFAS at concentrations several orders of magnitude below the concentrations identified at the AOIs (Table 10-3). Monitoring well MRC-2, located at the western installation boundary is considered downgradient of the AOIs from a regional groundwater flow perspective, and contained PFOS, PFOA, and PFHxS at concentrations of 1,100 ng/L, 51 ng/L, and 860 ng/L, respectively. Groundwater sampled at the AOI 1 originated within vesiculated basalt at a significantly higher elevation (approximately 1,440 feet above mean sea level) than the screened intervals of downgradient monitoring wells sampled during the baseline event (approximately 1,190 to 1,250 feet above mean sea level). Wells MTS-2, -3, -4, and MRC-2 are each screened in fractured basalt at or near the base of the Pomona Member. The potential for hydraulic

communication between these shallow and deep flow zones is not yet fully understood.

AOI 3 - AFFF Storage Area (Building 821)

Building 821 is located north of Firing Center Road (**Figure 10-2**). Sampling was conducted at Building 821 during both the SI and baseline investigation events at the locations shown on **Figure 10-9**. SI soil samples from Building 821 (**Table 10-4**) contained PFAS at concentrations below the OSD 2022 risk screening levels. However, groundwater in side-gradient/downgradient monitoring wells TVR-5 and 815-2 (**Table 10-3**) exhibited PFOS concentrations of 180 ng/L and 260 ng/L respectively, exceeding the OSD 2022 risk screening levels. In addition, groundwater samples collected from three existing upgradient and side-gradient monitoring wells during the baseline event contained PFAS exceeding the OSD 2022 risk screening level. AOI 1 is located upgradient of AOI 3. As such, baseline groundwater sampling results suggest that PFAS observed in groundwater around Building 821 may be associated with AOI 1 or AOI 3. One baseline surface water sample (SW-04; **Table 10-5**) collected at the drainage ditch along the north side of Firing Center Road (which bounds AOI 3 to the south; **Figure 10-9**) contained PFOS, PFOA, PFBS, PFNA, and PFHxS at concentrations exceeding the OSD 2022 risk screening levels. This location receives surface runoff from both Building 821 and the Refractometer Solutions Test Area, and it is unclear from which AOI, if not both, the PFAS in the drainage ditch originates. Additionally, the surface water sample was collected from standing water in a channel depression, not actively flowing surface water, and as such, may not be representative of surface water discharge. The potential for ongoing discharges through this pathway is being evaluated.

AOI 4 - Refractometer Solutions Test Area, AOI 5 - Fire Station 29 (Building 346), and AOI 6 - AFFF Storage Area (Building 321)

The Refractometer Solutions Test Area, Building 346, and Building 321 are located on the south side of Firing Center Road (**Figure 10-2**). Sampling was conducted at the Refractometer Solutions Test Area, Building 346, and Building 321 during both the SI and baseline investigation events at the locations shown on **Figure 10-10**. Two soil samples were collected from this area during the SI (**Table 10-4**), and two existing monitoring wells (MMP-1 and MMP-2) screened at the lower interface of the Pomona Member were sampled, all of which contained PFOS concentrations exceeding applicable OSD 2022 risk screening levels. In addition, PFOA, PFNA, and PFHxS concentrations exceeded the OSD 2022 risk screening levels in the two groundwater samples. MMP-1 and MMP-2 were resampled during the baseline event and exhibit similar results to the SI samples (**Table 10-3**). Sediment collected from the drainage ditch immediately north of the Refractometer Solutions Test Area during the SI contained PFOS at a concentration of 0.10 milligrams per kilogram (mg/kg), which exceeds the soil OSD 2022 risk screening level. PFOS detected in baseline sediment sample SED-05, collected approximately 250 feet downstream of the SI sample location was below the soil OSD 2022 risk screening level (**Table 10-6**). The detections in sediment indicate that overland transport of PFAS from the AOIs to the drainage ditches has occurred. The potential for ongoing discharges through this pathway is being evaluated.

AOI 7 - Mettie Airstrip (formerly Selah Airstrip)

The Mettie Airstrip AOI is located approximately 6 miles northeast of the main cantonment area (see inset on **Figure 10-2**). Due to the distance between the cantonment AOIs and the Mettie Airstrip, in conjunction with the complex regional geology, the causal correlation of PFAS in the subsurface between these areas is unlikely. Sampling was conducted at the Mettie Airstrip during both the SI and baseline investigation events at the locations shown on **Figure 10-11**. A groundwater sample collected in 2019 from the Mettie Airstrip supply well (screened from approximately 73 to 91 feet bgs) contained PFOA at a concentration of 100 ng/L (**Table 10-4**). Soil samples collected from within the limits of the former crash truck parking area (a suspected AFFF release area) associated with the AOI, contained PFOS concentrations exceeding the OSD

2022 risk screening level at a maximum detection of 0.12 mg/kg (**Table 10-5**). Local geologic and hydrogeologic conditions are poorly understood due to a lack of subsurface data at and near the Mettie Airstrip, and it is unclear if the supply well is positioned downgradient or side-gradient of the former crash truck parking area. PFAS was not detected in the surface water sample collected downgradient of the Selah Spring during the baseline sampling event (**Table 10-5**). This sample was collected to evaluate potential PFAS impacts in groundwater discharging to Selah Creek. Sediment samples collected from a dry creekbed both upgradient and downgradient of Mettie Airstrip contained PFOS below the OSD 2022 risk screening level (**Table 10-6**).

Boundary Well Groundwater Results

The boundary investigation was undertaken to establish a monitoring well network along the installation perimeter between AOIs and off-post receptors. Results of grab groundwater samples collected from first encountered groundwater during borehole advancement and low-flow groundwater sampling of the newly installed wells are presented in **Table 10-3** and shown on **Figure 10-12**.

Concentrations of PFBS and PFNA were below applicable risk screening levels in grab groundwater samples collected during drilling of the boundary wells (MW-01 through MW-08); however, MW-01 through MW-07 contained PFOS, PFOA, and/or PFHxS exceeding the applicable risk screening level. The highest concentration of PFOS (200 ng/L) was reported in MW-06. The highest concentrations of PFOA (20 ng/L and 26 ng/L for the primary and duplicate sample) and PFHxS (200 ng/L) was reported in MW-03. No PFAS constituents were detected in the grab groundwater sample from MW-08.

Concentrations of PFBS reported during the formal low-flow sampling event were below applicable risk screening levels in each of the boundary wells. Groundwater samples collected from MW-01, MW-03, MW-04, MW-06, and MW-07 contained PFOS, PFOA, PFHxS, and/or PFNA exceeding the applicable risk screening level. The highest concentrations of PFOS (1,500 ng/L), PFOA (140 ng/L), and PFNA (16 ng/L) was reported in MW-03, and the highest concentration of PFHxS (1,100 ng/L) was reported in MW-06. PFAS constituents were not detected in MW-02, MW-05, or MW-08 during the low-flow sampling event.

Contaminant Fate and Transport Pathways

PFAS impacts have been identified in soil, sediment, and groundwater associated with the AOIs and the potential source areas described above. The following interpretation of contaminant transport pathways provide a framework for understanding the distribution of PFAS in the environment at YTC. Multi-media sample sets collected to date provide a basis for adaptive characterization activities to be completed as part of the PFAS RI.

Contaminant transport pathways are heavily influenced by the complex geologic environment described above. Potential migration pathways identified in the CSM Tech Memo (ECC/Arcadis, 2023a) at YTC include:

- Leaching to groundwater from PFAS impacted soils at the source areas based on PFAS concentrations identified in soil samples.
- Storm and surface water drainages that receive direct runoff from several of the AOIs, including the Refractometer Solutions Test Area, Fire Station 29 (Building 346), AFFF Storage Area (Building 321), and AFFF Storage Area (Building 821). PFAS detections in surface water and sediment suggest that drainage ditches within the cantonment area have historically received and transported runoff containing AFFF. Additional surface water drainage channels located near Mettie Airstrip contained sediment impacted with PFOS and may receive intermittent seepage of PFAS-impacted groundwater or may have received overland flow containing PFAS from the airstrip.

- Lateral groundwater migration occurs within vesiculated and/or fractured upper and lower contacts of the Pomona Member. Based on the massive nature of the basalt units observed during drilling, vertical migration within the Pomona Member is likely limited to areas of faulting or, to a limited extent, fractures. Groundwater that occurs along the upper surface of basalt flows may be limited to areas of higher elevation.
- The sandstones encountered within the Ellensburg Fm (anomaly A5 on **Figure 10-4** and anomaly B1 on **Figure 10-5**) likely serve as preferential pathways for lateral migration, as well as a vertical pathway to the permeable sediments and vesicular basalt observed at the surface of the underlying basalt. Elevated PFAS concentrations were observed in off-post wells screened in the sandstone units. Additionally, these sandstones produced the highest yields observed during boundary well drilling.
- The contact between the Ellensburg Fm and underlying basalt consistently exhibits a thin volcanic ash lens, followed by a thin layer of coarse material (i.e., basal deposits). These basal deposits likely serve as a generally continuous zone of relatively higher permeability which could facilitate lateral migration of impacted groundwater. This migration pathway may be interrupted where the permeable strata are offset by faults.

Potential Human Receptors and Exposure Pathways

As described above, releases of AFFF may have occurred at YTC during firefighter training, fire station activities, equipment testing, AFFF storage, or possible emergency response efforts (e.g., at the Mettie Airstrip AOI). AFFF use, leaks or spills were not reported for the Mettie Airstrip AOI; however, personnel interviews indicated that an AFFF crash truck was parked outside and stored AFFF in its tank, and PFAS were detected in groundwater at the Mettie Airstrip water stand and well house.

A preliminary understanding of potential human receptors and exposure pathways based on current and/or reasonably anticipated future land uses at the installation's AOIs is presented on **Figure 10-13** (Cantonment Area AOIs) and **Figure 10-14** (Mettie Airstrip AOI). The human health CSMs were prepared in accordance with the USACE Engineer Manual on CSMs, EM 200-1-12 (USACE, 2012b). Based on the suspected or confirmed historical use of AFFF at the AOIs, affected media are likely to consist of soil, groundwater, ephemeral surface water, and/or sediment. Release and transport mechanisms include dissolution/desorption from soil to groundwater, transport via sediment carried in and dissolution to stormwater and surface water, recharge from surface water to groundwater, and adsorption/desorption between surface water and sediment. Once released to the environment, a primary factor that inhibits the movement of PFAS constituents is the presence of organic matter and organic co-constituents in soils and sediments. Generally, PFAS constituents are mobile in the potentially affected media, and they are not known to be fully broken down by natural processes.

Potential human receptors include on-post outdoor site workers (e.g., military base personnel), construction workers, trespassers/recreational users, and residents. Subsistence users are additional potential receptors for the Mettie Airstrip due to the known use of the on-post area around the airstrip by the Yakama Nation for subsistence activities (e.g., root digging, hunting, and fishing). Off-installation receptors may include drinking water users and recreational users accessing downgradient surface water features. Generally, human exposure to PFAS in soil at the AOIs may occur through incidental ingestion, dermal contact, and inhalation of dust (particulates). Volatilization from soil and groundwater and vapor intrusion are not meaningful pathways for PFAS, as they are not volatile.

The AOIs are currently industrial sites used for military training or support (e.g., fire station, airstrip, etc.) and are likely to remain military/industrial in the foreseeable future. However, the Baseline Human Health Risk Assessment (BHHRA) will conservatively evaluate an onsite Unlimited Use/Unrestricted Exposure (UU/UE) scenario to inform future risk-management decisions and the FS, if applicable. This scenario will

include hypothetical future onsite residential exposure to soil and the evaluation of onsite groundwater as a source of potable water. A remedial response will not necessarily be taken based on the results of the future onsite UU/UE scenario, given future residential development of the AOIs is not a reasonably anticipated future use, per the DoD Defense Environmental Restoration Program (DERP) Management Manual (DoD 2012), which states “*The DoD Component shall consider current and reasonably anticipated future land uses in risk assessments. The DoD Component does not have to assume that the reasonably anticipated future land use is residential.*”

Drinking water for YTC is supplied by on-post groundwater wells. The cantonment AOIs are upgradient or cross-gradient of the on-post drinking water wells (i.e., Pomona and Jordan wells). These wells are screened greater than 350 feet bgs, below a confining layer and are artesian. The deeper aquifer used for these drinking water wells is not hydraulically connected to the shallow aquifer beneath YTC. Testing for PFAS from these drinking water wells had shown that this aquifer is not impacted. The Mettie Airstrip water well and stand were turned off and put in no-use status following the detections of PFAS observed at the well and water stand in 2019.

For all YTC AOIs, the groundwater exposure pathway (via drinking water ingestion and dermal contact) for on-installation site workers is currently incomplete. However, the groundwater exposure pathways (via drinking water ingestion and dermal contact) for hypothetical future site workers and residents are potentially complete to account for a hypothetical future scenario in which the downgradient on-post groundwater that is screened above the confining unit is used as a potable water source, a scenario where vertical gradients reverse and/or the confining unit is compromised and the artesian aquifer were to become susceptible to PFAS impacts, and to consider the potential for the Mettie Airstrip well to be turned back on. Similarly, the groundwater exposure pathway for off-installation drinking water receptors is potentially complete because groundwater originating at the AOIs has the potential to flow off-post and the groundwater contained in the shallow aquifer above the confining layer may be consumed by off-post receptors.

PFAS may have traveled laterally in soil, sediment, or surface water through erosion or stormwater runoff to nearby drainage features and surface water bodies, particularly at the AOIs where the distance from the source to surface water channel and the site-specific topography are conducive to this migration pathway. Downgradient of the AOIs, on-post surface water bodies are intermittent (i.e., only flowing after heavy precipitation events) and are not used for drinking water or recreation. Despite YTC’s arid climate and the intermittent nature of surface water features on-post, constituents could migrate from soil and shallow groundwater to off-post surface water bodies. Generally, human exposure to PFAS in surface water and sediment may occur through incidental ingestion and dermal contact. There is the potential for exposure associated with the two irrigation canals that divert water from the Yakima River in the vicinity of YTC. The receptors and potential for human exposure associated with this pathway will be evaluated during the RI. Ingestion of aquatic biota (e.g., fish) that may bioaccumulate PFAS is a potentially complete human exposure pathway; however, PFAS impacts to aquatic biota will not be evaluated during the RI. Due to the inherent uncertainties associated with modeling potential uptake and bioaccumulation in animals, human exposure through consumption of aquatic biota will be discussed in the uncertainty section of the BHHRA.

Additionally, the potential exposure of hunters through consumption of game animals that may have bioaccumulated PFAS will not be evaluated in the BHHRA. Recreational hunting at YTC occurs in designated areas outside the cantonment. Wild game habitat at the cantonment AOIs is limited due to the presence of buildings, pavement/concrete, maintained/mowed areas, and human activity. Therefore, game animals are not expected to forage at the cantonment AOIs, and the game ingestion exposure pathway is considered to be incomplete. The likelihood of this exposure scenario occurring at the Mettie Airstrip AOI will be evaluated and discussed in the uncertainty section of the BHHRA.

Data Gaps

PFAS has been detected in on-post groundwater, soil, surface water, and/or sediment associated with each AOI. The following data gaps were identified while developing the CSM and will be addressed as part of the ongoing PFAS RI at YTC:

- The lateral and vertical extents of PFAS in soil and groundwater at each AOI are currently undefined and relative PFAS contributions from individual AOIs to the entire PFAS plume are unknown. Contributions attributable to an individual source can be influenced by source mass, connections to preferential pathways, the degree of surface infiltration, and the age of each source. Further source characterization and evaluation of preferential contaminant migration pathways will be undertaken in the RI.
- Perched groundwater at the Former Fire Training Pit and Bird Bath Wash Rack contains elevated concentrations of PFAS; however, the degree of hydraulic communication between water-bearing zone(s) within the up-slope basalts and the downgradient deeper water-bearing zone identified at the Installation boundary has not been determined. Migration pathways at and downgradient of the Mettie Airstrip are unknown due to a lack of subsurface data and distance from the other AOIs.
- The mechanism for vertical migration of perched groundwater to the underlying sand-rich units of the Ellensburg Fm is not completely understood. Some vertical groundwater transport may occur through fractured zones within the Pomona Member. However, the more massive sections of the Pomona Member likely enhance lateral migration along the surface of the basalt until it encounters the edge of the Pomona Member and infiltrates into the more permeable Ellensburg Fm. If lateral groundwater migration occurs along the surface of the Pomona Member, a refined understanding of the extent of the Pomona Member will be critical to defining the migration pathway.
- Current groundwater data sets are insufficient to adequately delineate the lateral and vertical extent of PFAS exceeding the risk screening levels. PFAS extent in the downgradient direction is currently based on analytical results generated from private water supply wells. However, the absence of well construction details for most of off-post private wells inhibits accurate correlation between wells.
- Soil quality has not been delineated at the Refractometer Solutions Test Area, Building 321, or Mettie Airstrip. Additionally, despite the significant soil reworking that occurred at the Former Fire Training Pit and Bird Bath Wash Rack, soil sampling will be required to confirm that PFAS is not present in soils above applicable risk screening levels.
- The irrigation canal was dry at the time of baseline sampling; however, sediment samples collected at locations SED-01 and SED-02 were reported as non-detect for PFAS (**Figure 10-10**). The irrigation canal is unlined and is interpreted to be a losing stream when flowing (Margaret Taaffe, pers. com. 2022). Due to the unlined construction of the canal potential for PFAS transport from off-post sources in the irrigation canal should be evaluated.

Worksheet #11 establishes the DQOs that will be used to guide the investigation methods for the RI. **Worksheet #17** provides the rationale and framework and investigation approach for the RI. **Worksheets #18 and #20** will be iteratively populated and memorialized in Field Change Forms to list the proposed sample identifications and required QC samples for each medium as the RI progresses. The documentation of iterative phases of RI scope in Field Change Forms is a collectively preferred approach that was established to meet both the project needs and the needs of Regulators and stakeholders to collaborate, review, and document the locations and rationale for sampling and investigation performed for the RI.

WORKSHEET #11: PROJECT/DATA QUALITY OBJECTIVES

(UFP-QAPP Manual Section 2.6.1) (USEPA 2106-G-05 Section 2.2.6)

Worksheet #11 describes the DQOs using USEPA's seven-step DQO process: *Guidance on Systematic Planning Using the Data Quality Objectives Process, USEPA QA/G-4, EPA/240/B-06/001*, February 2006. The selected investigation design is presented on **Worksheet #17**. This worksheet will state the problem and identify the goal of the study, the information inputs, and performance or acceptance criteria.

Step 1: State the Problem

PFAS (including PFOS, PFOA, PFBS, PFNA, PFHxS, and HFPO-DA) are a group of synthetic fluorinated compounds used to make everyday products more resistant to heat, stains, grease, and water; they are also used as components in firefighting foams. These chemicals were used by the military in AFFF to extinguish fires or for training purposes, as well as in some waterproofing and laundry activities. The chemical structures of PFAS make them very resistant to breakdown in the environment. Because of their persistence, bioaccumulation potential, mobility, and toxicity, PFAS could have a potential impact on human health and the environment (USEPA, 2022a).

Historical use of PFAS has resulted in their release to environmental media at YTC. PFAS-impacted groundwater has migrated to downgradient drinking water supplies. PFAS-impacted soils may act as ongoing sources to groundwater and may pose a risk to on-post human receptors. The AOIs identified at YTC include locations in which PFAS-containing materials are known to have been used, and existing groundwater, surface water, and soil analytical results confirm that PFAS are present. A total of seven PFAS AOIs will be characterized as part of the RI. The vertical and lateral extent of previously identified PFOA and PFOS contamination in groundwater and other environmental media originating from the seven AOIs and risk to potentially exposed receptors are unknown at this time.

Step 2: Identify the Goal of the Study

The primary goal of the study is to complete a CERCLA and DERP compliant RI that will include sampling to compile sufficient analytical data to determine the nature and extent (both lateral and vertical) of PFAS in media associated with the AOIs, refine the preliminary CSMs, and determine the risk to potentially exposed receptors. Specifically:

- Perform environmental sampling to compile sufficient analytical data to determine the nature and extent (both lateral and vertical) of PFAS in soil, groundwater, and surface water associated with the AOIs
- Determine if soil concentrations of PFAS may be contributing to ongoing groundwater contamination
- Refine the preliminary CSMs to:
 - determine groundwater flow and subsequent off-post migration of PFAS
 - determine the risk to potentially exposed human receptors
 - identify exposure pathways and sampling requirements in subsequent investigations (if needed).

The sampling will be conducted in conformance with the OSD Memorandum (OSD, 2022) and the *DoD Emerging Chemicals of Environmental Concern Instruction* (DoD, 2019). PFAS results for soil, groundwater, surface water, and sediment will be evaluated based on the available OSD risk screening levels for residential exposure using a hazard quotient of 0.1 for the purposes of this RI. A summary of the OSD residential screening levels that will be referenced is included as **Worksheet #15**.

Table 11-1: Summary of 2022 OSD Risk Screening Levels

| Chemical of Concern | Matrix | Units | OSD Risk Screening Level (Residential ¹) (Hazard Quotient equals 0.1) |
|---------------------|---------------|-------|---|
| PFOS | Soil/sediment | mg/kg | 0.013 |
| PFOA | Soil/sediment | mg/kg | 0.019 |
| PFBS | Soil/sediment | mg/kg | 1.9 |
| PFNA | Soil/sediment | mg/kg | 0.019 |
| PFHxS | Soil/sediment | mg/kg | 0.13 |
| HFPO-DA | Soil/sediment | mg/kg | 0.023 |
| PFOS | Water | ng/L | 4 |
| PFOA | Water | ng/L | 6 |
| PFBS | Water | ng/L | 601 |
| PFNA | Water | ng/L | 6 |
| PFHxS | Water | ng/L | 39 |
| HFPO-DA | Water | ng/L | 6 |

Notes:

¹Risk screening levels for tap water and soil/sediment provided by the OSD 2022. Memorandum: Investigation Per- and Polyfluoroalkyl Substances within the DoD Cleanup Program. July 6. (OSD 2022). These standards are not applicable for sediment or surface water but can be used as comparison values for reference for these environmental media. There are currently no human health screening criteria for PFOS, PFOA, PFBS, PFNA, PFHxS, or HFPO-DA in surface water or sediment; therefore, for reference, surface water and sediment results will be compared to groundwater and soil criteria (respectively).

HFPO-DA - hexafluoropropylene oxide dimer acid

mg/kg – milligrams per kilogram

ng/L – nanograms per Liter

OSD – Office of the Secretary of Defense

PFBS – perfluorobutanesulfonic acid

PFHxS – perfluorohexanesulfonic acid

PFNA – perfluoronanoic acid

PFOA – perfluorooctanoic acid

PFOS – perfluorooctane sulfonate

The data needed to accomplish the goals of the sampling and analysis activities for this RI are:

- Historical data sets from previous PFAS sampling programs, including the U.S. Army Installation Management Command Operations Order 16-088 sampling of water supply wells in 2016, and the PA/SI (SERES/Arcadis, 2023). These data will be used in conjunction with information inputs summarized below to meet the goals in Step 2. The *Final Site Inspection Report* (Arcadis, 2021) summarizes concentrations of PFOS, PFOA, and PFBS in soil, sediment, groundwater, and surface water from each of the AOIs included in this UFP-QAPP.
- Analytical data associated with groundwater, surface water, and sediment samples collected during the baseline sampling event in February 2023. Baseline sampling results are summarized in **Worksheet #10** as well as in the 2023 CSM Technical Memorandum (ECC/Arcadis 2023a).
- Hydrogeologic data, interpreted geophysical findings, and analytical results from the Boundary Investigation are summarized in **Worksheet #10**.
- Geographic information system (GIS) data from existing well locations and AFFF use area boundaries (**Figures 10-8 through 10-11**).
- Hydrogeologic information to evaluate groundwater flow and transport pathways beneath and downgradient from source areas.

- Aerial imagery of select areas at YTC to evaluate downgradient sampling locations based on geomorphologic interpretation.
- Well construction details and geologic data associated with existing on-post water supply wells and monitoring wells.
- Surface geophysical data sets generated during the Boundary Investigation.
- Future RI surface geophysical data sets as described in **Worksheet #17**.
- Future RI groundwater, surface water, sediment, and soil samples described in subsequent worksheets. Laboratory analytical methods and PFAS constituents for RI samples are summarized in **Worksheet #17**.
- Hydraulic testing data as described in **Worksheet #17**.
- Groundwater and surface water elevation data, including historically available data sets, in addition to elevation data collected during the scope outlined in **Worksheet #17**. Groundwater elevation monitoring will include gauging data for the groundwater and surface water interaction described in **Worksheet #17**.
- Parameters and analytical methods for proposed environmental samples are identified in **Worksheets #19 & #30**. Field sample collection methods are summarized in **Worksheets #17 & #21**.

Step 4: Define the Boundaries of the Sampling

The RI approach includes characterization of PFAS, specifically PFOS, PFOA, PFBS, PFNA, PFHxS, and HFPO-DA, in groundwater, soil, surface water, and sediment within, upgradient, and downgradient from the seven AOIs. Detailed scope elements are described in **Worksheet #17**.

Analytical sample collection will be within, upgradient, and downgradient from the seven AOIs identified as potential source areas (**Worksheet #10**). Proposed sample nomenclature for each medium and proposed sample locations are listed in **Worksheet #18** of this UFP-QAPP. In addition, proposed sample locations are shown on **Figures 17-1** through **17-5**.

The RI approach includes the systematic planning and sequencing of work that uses a combination of prescriptive locations (pre-determined based on initial CSM) and adaptive locations, which includes “step outs” to complete delineation of lateral and vertical impacts and/or “step ins” to zoom in on source hotspots or the core of the groundwater impacts. Prescriptive soil borings will be advanced at PFAS source areas, along with on- and off-post groundwater, surface water, soil, and sediment sampling (Figures 17-1 through 17-5). Samples will be submitted for laboratory analysis as described in Step 5, below. As laboratory analytical data become available, the CSM will be updated, and additional step-out locations will be identified. The adaptive scope will be determined based on review of data, with locations selected through coordination with regulatory stakeholders. Adaptive investigation locations will be positioned on-post and may include areas outside the Installation boundary as needed to adequately delineate the extent of PFAS in environmental media.

Step 5: Develop the Analytic Approach

Environmental samples will be submitted for analysis to AEL, a DoD and Washington State-certified, National ELAP (NELAP)-certified laboratory located in Jacksonville Florida, for analysis of PFAS in accordance with requirements in the DoD/Department of Energy Consolidated QSM for Environmental Laboratories Version 5.4 Table B-24 PFAS Analysis by liquid chromatography with tandem mass spectrometry (LC/MS/MS) by USEPA Draft Method 1633 (Method 1633). Eurofins Lancaster Laboratories

Environment Testing, LLC (ELLE) is included as a secondary laboratory, and may be used in the event of capacity issues and/or equipment breakdowns, etc. Laboratory-specific Worksheets for ELLE are included in this UFP-QAPP as **Appendix G**. PFAS samples will be analyzed in accordance with DoD QSM version 5.4, which includes 40 PFAS compounds (including PFOS, PFOA, PFBS, PFNA, PFHxS, and HFPO-DA) as listed in **Worksheet #15**. **Worksheets #15A** and **#15B** identify specific PFAS analytes and the laboratory limits of detection (LODs), limits of quantitation (LOQs), and detection limits (DLs) for each PFAS analyte at AEL and ELLE, respectively. Unless otherwise noted, further references to Worksheet #15 refer to the primary laboratory, AEL. Quality Assurance/Quality Control (QA/QC) samples will be collected, as detailed in **Worksheet #20**.

Select soil samples will be collected and analyzed for total organic carbon (TOC) and grain size to further support analysis of PFAS transport in the unsaturated zone. During monitoring well installation activities, soil samples will be collected from selected soil borings within the screen interval and evaluated for PFAS and grain-size (including hydrometer analysis as needed) to assist the evaluation of fate and transport.

Sediment and surface water samples will be analyzed for PFAS by Method 1633 QSM version 5.4 only per the methods listed in **Worksheet #15**.

If collected, drinking water samples will be analyzed for PFAS by USEPA Method 537.1. Water quality field parameters (i.e., temperature, pH, conductivity, dissolved oxygen [DO], turbidity, and oxidation-reduction potential [ORP]) will be recorded during sample collection, as applicable.

Worksheets #17 and **#18** further identify project investigation approach, sampling methods, and analysis rationale.

Investigation-derived waste (IDW) consisting of solids (e.g., soil cuttings) and liquids (e.g., purge water and decontamination water) will be generated during the RI field activities. IDW will be contained, disposed, and/or stored pending analysis as appropriate for each waste stream. The final waste characterization and disposal plan for IDW will be implemented in accordance with U.S. Army guidance and state/local regulations. Disposal of IDW will be discussed in **Worksheet #17**.

Step 6: Specify Performance or Acceptance Criteria

Measurement Performance Criteria (MPC) for precision and accuracy are provided in **Worksheets #12** and **#28**. Field monitoring and detection equipment will be routinely calibrated, as detailed on **Worksheet #22**, which confirms that equipment used is of the proper type, range, accuracy, and precision to provide data compatible with the specified requirements and desired results. The Data Usability Assessment (DUA) process is described in **Worksheet #37**.

Step 7: Develop the Plan for Obtaining Data

The sampling plans and rationale to achieve the DQOs established for this RI are presented in **Worksheet #17**. Sampling plans may be revised based on field conditions or site planning meetings, with appropriate notification and concurrence of USAEC, YTC, and USACE. In addition, the Army will consult with Regulators as needed during the revision process and/or to discuss any deviations from the sampling plans described herein. Deviations from this UFP-QAPP will be documented in the RI Report. All work will be performed in accordance with the Accident Prevention Plan/Site Safety and Health Plan, provided as **Appendix H**.

WORKSHEET #12: MEASUREMENT PERFORMANCE CRITERIA

WORKSHEET #12-1: MEASUREMENT PERFORMANCE CRITERIA - PFAS IN SOIL AND SEDIMENT

(UFP-QAPP Manual Section 2.6.2) / (USEPA 2106-G-05 Section 2.2.6)

| Matrix | Soil/Sediment | | |
|--|---|--|--|
| Analytical Group/Method/SOP | PFAS per USEPA 1633 Draft and QSM 5.4 Table B-24 / AEL SVOC-043 | | |
| Concentration Level - Low | | | |
| DQI | QC Sample or Measurement Performance Activity | MPC | QC Sample Assesses Error for S, A, or both S&A |
| Precision | FDs | RPD ≤ 50% | S&A |
| Accuracy/Bias | EIS (Isotope Dilution Analogues) | %R must be within in-house limits. Preliminary inhouse acceptance criteria of 20-150% must be used until in-house limits are generated in accordance USEPA 1633. The lower limit of inhouse acceptance criteria cannot be < 20%. | A |
| Accuracy/Bias | LCS | % Rec. See table below | A |
| Analytical Accuracy/Bias (matrix interference) | MS/MSD | %R same as LCS | S&A |
| Precision | LCSD and MSD | RPD ≤ 30% | S&A |
| Accuracy/Bias (contamination) | MB, FB, or EBs as appropriate | No analytes detected > ½ LOQ or > 1/10th the amount measured in any associated sample or 1/10th the regulatory limit, whichever is greater | A |
| Completeness | Useable data (not rejected) | > 90% | S&A |
| Sensitivity | Instrument Sensitivity Check | All analyte concentrations must be within ± 30% of their true values. | A |

Notes: The table above complies with the requirements of USEPA Draft method 1633 and DoD QSM 5.4 Table B-24.

- | | | |
|------------------------------|---|---|
| % - percent | EIS – Extracted Internal Standard | PFAS – Per- and Polyfluoroalkyl substances |
| ± plus or minus | FB – Field Blank | QC – Quality Control |
| %R – percent recovery | FD – Field Duplicate | QSM – Quality Systems Manual |
| < less than | LCS – laboratory control spike | RPD – relative percent difference |
| ≤ less than or equal to | LCSD – laboratory control spike duplicate | RPD – relative percent difference |
| > greater than | LOQ – limit of quantitation | S – Sampling |
| A – Analytical | MB – Method Blank | SOP – Standard Operating Procedure |
| DoD – Department of Defense | MPC – measurement performance criteria | USEPA – United States Environmental Protection Agency |
| DQI – data quality indicator | MS – matrix spike | |
| EB – Equipment Blank | MSD – matrix spike duplicate | |

PFAS Accuracy Limits – USEPA Draft Method 1633 compliant with DoD QSM 5.4 Table B-24

| Analyte | Acronym | Chemical Abstracts Service (CAS) Number | Accuracy Limits (Percent Recovery [%R]) |
|---|----------|---|---|
| PFAS – Solids | | | |
| 1H,1H,2H,2H-Perfluorohexanesulfonic acid | 4:2FTS | 757124-72-4 | 40-150 |
| 1H,1H,2H,2H-Perfluorooctanesulfonic acid | 6:2FTS | 27619-97-2 | 40-150 |
| 1H,1H,2H,2H-Perfluorodecanesulfonic acid | 8:2FTS | 39108-34-4 | 40-150 |
| 4,8-Dioxa-3H-perfluorononanoic acid | ADONA | 919005-14-4 | 40-150 |
| N-Ethyl-perfluorooctane sulfonamidoacetic acid | NEtFOSAA | 2991-50-6 | 40-150 |
| N-Methyl-perfluorooctane sulfonamidoacetic acid | NMeFOSAA | 2355-31-9 | 40-150 |
| N-Methyl perfluorooctanesulfonamide | NMeFOSA | 31506-32-8 | 40-150 |
| N-Ethyl perfluorooctanesulfonamide | NEtFOSA | 4151-50-2 | 40-150 |
| N-Methyl perfluorooctanesulfonamidoethanol | NMeFOSE | 24448-09-7 | 40-150 |
| N-Ethyl perfluorooctanesulfonamidoethanol | NEtFOSE | 1691-99-2 | 40-150 |
| Hexafluoropropylene oxide dimer acid | HFPO-DA | 13252-13-6 | 40-150 |
| Perfluorobutanesulfonic acid | PFBS | 375-73-5 | 40-150 |
| Perfluorobutanoic acid | PFBA | 375-22-4 | 40-150 |
| Perfluorodecanoic acid | PFDA | 335-76-2 | 40-150 |
| Perfluorodecanesulfonic acid | PFDS | 335-77-3 | 40-150 |
| Perfluorododecanoic acid | PFDoA | 307-55-1 | 40-150 |
| Perfluorododecanesulfonic acid | PFDoS | 79780-39-5 | 40-150 |
| Perfluoroheptanoic acid | PFHpA | 375-85-9 | 40-150 |
| Perfluoroheptanesulfonic acid | PFHpS | 375-92-8 | 40-150 |
| Perfluorohexanoic acid | PFHxA | 307-24-4 | 40-150 |
| Perfluorohexanesulfonic acid | PFHxS | 355-46-4 | 40-150 |
| Perfluorononanoic acid | PFNA | 375-95-1 | 40-150 |
| Perfluoronananesulfonic acid | PFNS | 68259-12-1 | 40-150 |
| Perfluorooctanoic acid | PFOA | 335-67-1 | 40-150 |
| Perfluorooctanesulfonic acid | PFOS | 1763-23-1 | 40-150 |
| Perfluorooctanesulfonamide | PFOSA | 754-91-6 | 40-150 |
| Perfluoropentanoic acid | PFPeA | 2706-90-3 | 40-150 |
| Perfluoropentanesulfonic acid | PFPeS | 2706-91-4 | 40-150 |

| Analyte | Acronym | Chemical Abstracts Service (CAS) Number | Accuracy Limits (Percent Recovery [%R]) |
|---|--------------|---|---|
| Perfluorotetradecanoic acid | PFTeDA | 376-06-7 | 40-150 |
| Perfluorotridecanoic acid | PFTrDA | 72629-94-8 | 40-150 |
| Perfluoroundecanoic acid | PFUnA | 2058-94-8 | 40-150 |
| Perfluoro-3-methoxypropanoic acid | PFMPA | 377-73-1 | 40-150 |
| Perfluoro-4-methoxybutanoic acid | PFMBA | 863090-89-5 | 40-150 |
| Nonafluoro-3,6-dioxahheptanoic acid | PFDDHA | 151772-58-6 | 40-150 |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid | 9Cl-PF3ONS | 756426-58-1 | 40-150 |
| 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid | 11Cl-PF3OUdS | 763051-92-9 | 40-150 |
| Perfluoro(2-ethoxyethane)sulfonic acid | PFEESA | 113507-82-7 | 40-150 |
| 3-Perfluoropropyl propanoic acid | 3:3FTCA | 356-02-5 | 40-150 |
| 2H,2H,3H,3H-Perfluorooctanoic acid | 5:3FTCA | 914637-49-3 | 40-150 |
| 3-Perfluoroheptyl propanoic acid | 7:3FTCA | 812-70-4 | 40-150 |

WORKSHEET #12-2: MEASUREMENT PERFORMANCE CRITERIA - PFAS IN GROUNDWATER AND SURFACE WATER
 (UFP-QAPP Manual Section 2.6.2)
 (USEPA 2106-G-05 Section 2.2.6)

| Matrix | | Groundwater and Surface Water | |
|--|---|--|--|
| Analytical Group/Method/SOP | | PFAS per USEPA Draft Method 1633 compliant with DoD QSM 5.4 Table B-24 / AEL SVOC-043 | |
| Concentration Level - Low | | | |
| DQI | QC Sample or Measurement Performance Activity | MPC | QC Sample Assesses Error for S, A, or both S&A |
| Precision | FDs | RPD ≤ 30% | S&A |
| Accuracy/Bias | EIS (Isotope Dilution Analogues) | %R must be within in-house limits. Preliminary inhouse acceptance criteria of 20-150% must be used until in-house limits are generated in accordance USEPA 1633. The lower limit of inhouse acceptance criteria cannot be < 20%. | A |
| Accuracy/Bias | LCS | See table below | A |
| Analytical Accuracy/Bias (matrix interference) | MS/MSD | %R same as LCS | S&A |
| Precision | LCSD and MSD | RPD ≤ 30% | S&A |

| Matrix | | Groundwater and Surface Water | |
|-------------------------------|---|--|--|
| Analytical Group/Method/SOP | | PFAS per USEPA Draft Method 1633 compliant with DoD QSM 5.4 Table B-24 / AEL SVOC-043 | |
| Concentration Level - Low | | | |
| DQI | QC Sample or Measurement Performance Activity | MPC | QC Sample Assesses Error for S, A, or both S&A |
| Accuracy/Bias (contamination) | MB, FB, or EBs as appropriate | No analytes detected > ½ LOQ or > 1/10th the amount measured in any associated sample or 1/10th the regulatory limit, whichever is greater | A |
| Completeness | Useable data (not rejected) | > 90% | S&A |
| Sensitivity | Instrument Sensitivity Check | All analyte concentrations must be within ± 30% of their true values. | A |

Notes: The table above complies with the requirements of USEPA Draft Method 1633 compliant with DoD QSM 5.4 Table B-24.

- ± - plus or minus
- % - percent
- > greater than
- < less than
- ≤ less than or equal to
- ± plus or minus
- %R – percent recovery
- A – Analytical
- DoD – Department of Defense
- DQI – data quality indicator
- EB – equipment blank
- EIS - extracted internal standard
- FB – Field Blank.
- FD – field duplicate
- MB – method blank
- RPD – relative percent difference
- LCS – laboratory control spike
- LCSD – laboratory control spike duplicate
- LOQ – limit of quantitation
- MPC – measurement performance criteria
- MS – matrix spike
- MSD – matrix spike duplicate
- PFAS – Per- and Polyfluoroalkyl substances
- QC – Quality Control
- QSM – Quality Systems Manual
- RPD – relative percent difference
- S – Sampling
- SOP – Standard Operating Procedure
- USEPA – United States Environmental Protection Agency

| PFAS Accuracy Limits – USEPA Draft Method 1633 compliant with DoD QSM 5.4 Table B-24 | | | |
|--|----------|--|-----------------|
| Analyte | Acronym | Chemical Abstract Service (CAS) Number | Accuracy Limits |
| <i>PFAS – Groundwater and Surface Water</i> | | | |
| 1H,1H,2H,2H-Perfluorohexanesulfonic acid | 4:2FTS | 757124-72-4 | 40-150 |
| 1H,1H,2H,2H-Perfluorooctanesulfonic acid | 6:2 FTS | 27619-97-2 | 40-150 |
| 1H,1H,2H,2H-Perfluorodecanesulfonic acid | 8:2 FTS | 39108-34-4 | 40-150 |
| 4,8-Dioxa-3H-perfluorononanoic acid | ADONA | 919005-14-4 | 40-150 |
| N-Ethyl-perfluorooctane sulfonamidoacetic acid | NEtFOSAA | 2991-50-6 | 40-150 |
| N-Methyl-perfluorooctane sulfonamidoacetic acid | NMeFOSAA | 2355-31-9 | 40-150 |
| N-Methyl perfluorooctanesulfonamide | NMeFOSA | 31506-32-8 | 40-150 |
| N-Ethyl perfluorooctanesulfonamide | NEtFOSA | 4151-50-2 | 40-150 |

| PFAS Accuracy Limits – USEPA Draft Method 1633 compliant with DoD QSM 5.4 Table B-24 | | | |
|--|--------------|--|-----------------|
| Analyte | Acronym | Chemical Abstract Service (CAS) Number | Accuracy Limits |
| N-Methyl perfluorooctanesulfonamidoethanol | NMeFOSE | 24448-09-7 | 40-150 |
| N-Ethyl perfluorooctanesulfonamidoethanol | NEtFOSE | 1691-99-2 | 40-150 |
| Hexafluoropropylene oxide dimer acid | HFPO-DA | 13252-13-6 | 40-150 |
| Perfluorobutanoic acid | PFBA | 375-22-4 | 40-150 |
| Perfluorobutanesulfonic acid | PFBS | 375-73-5 | 40-150 |
| Perfluorodecanoic acid | PFDA | 335-76-2 | 40-150 |
| Perfluorodecanesulfonic acid | PFDS | 335-77-3 | 40-150 |
| Perfluorododecanoic acid | PFDoA | 307-55-1 | 40-150 |
| Perfluorododecanesulfonic acid | PFDoS | 79780-39-5 | 40-150 |
| Perfluoroheptanoic acid | PFHpA | 375-85-9 | 40-150 |
| Perfluoroheptanesulfonic acid | PFHpS | 375-92-8 | 40-150 |
| Perfluorohexanoic acid | PFHxA | 307-24-4 | 40-150 |
| Perfluorohexanesulfonic acid | PFHxS | 355-46-4 | 40-150 |
| Perfluorononanoic acid | PFNA | 375-95-1 | 40-150 |
| Perfluorononanesulfonic acid | PFNS | 68259-12-1 | 40-150 |
| Perfluorooctanoic acid | PFOA | 335-67-1 | 40-150 |
| Perfluorooctanesulfonic acid | PFOS | 1763-23-1 | 40-150 |
| Perfluorooctanesulfonamide | PFOSA | 754-91-6 | 40-150 |
| Perfluoropentanoic acid | PFPeA | 2706-90-3 | 40-150 |
| Perfluoropentanesulfonic acid | PFPeS | 2706-91-4 | 40-150 |
| Perfluorotetradecanoic acid | PFTeDA | 376-06-7 | 40-150 |
| Perfluorotridecanoic acid | PFTrDA | 72629-94-8 | 40-150 |
| Perfluoroundecanoic acid | PFUnA | 2058-94-8 | 40-150 |
| Perfluoro-3-methoxypropanoic acid | PFMPA | 377-73-1 | 40-150 |
| Perfluoro-4-methoxybutanoic acid | PFMBA | 863090-89-5 | 40-150 |
| Nonafluoro-3,6-dioxahheptanoic acid | PFDDHA | 151772-58-6 | 40-150 |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid | 9Cl-PF3ONS | 756426-58-1 | 40-150 |
| 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid | 11Cl-PF3OUdS | 763051-92-9 | 40-150 |
| Perfluoro(2-ethoxyethane)sulfonic acid | PFEESA | 113507-82-7 | 40-150 |
| 3-Perfluoropropyl propanoic acid | 3:3FTCA | 356-02-5 | 40-150 |

| PFAS Accuracy Limits – USEPA Draft Method 1633 compliant with DoD QSM 5.4 Table B-24 | | | |
|--|---------|--|-----------------|
| Analyte | Acronym | Chemical Abstract Service (CAS) Number | Accuracy Limits |
| 2H,2H,3H,3H-Perfluorooctanoic acid | 5:3FTCA | 914637-49-3 | 40-150 |
| 3-Perfluoroheptyl propanoic acid | 7:3FTCA | 812-70-4 | 40-150 |

WORKSHEET #12-3: MEASUREMENT PERFORMANCE CRITERIA - TOTAL ORGANIC CARBON IN SOIL

| Matrix | Soil | | |
|--|---|------------------------------------|--|
| Analytical Group/Method/SOP | TOC/SW846 9060A/ AEL WC-021 | | |
| Concentration Level | Low | | |
| DQI | QC Sample or Measurement Performance Activity | MPC | QC Sample Assesses Error for S, A, or both S&A |
| Precision | FDs | RPD ≤ 50% | S&A |
| Accuracy/Bias | LCS | %R 80-120% | A |
| Analytical Accuracy/Bias (matrix interference) | MS/MSD | %R 75-125% | S&A |
| Precision | LCSD and MSD | RPD ≤ 35% | A |
| Accuracy/Bias (contamination) | MB and/or EBs | No detections > LOQ | A |
| Completeness | Useable data (not rejected) | > 90% | S&A |
| Sensitivity | LOQ Verification Sample (spiked at LOQ) | Recovery within ±30% of true value | A |

Notes:

- | | | |
|---|---|------------------------------------|
| ± plus or minus | EB – equipment blank | MS – matrix spike |
| < less than | EIS - extracted internal standard | MSD – matrix spike duplicate |
| ≤ less than or equal to | FD – field duplicate | QC – Quality Control |
| > greater than | MB – method blank | S – Sampling |
| % – percent | RPD – relative percent difference | SOP – Standard Operating Procedure |
| %R – percent recovery | LCS – laboratory control spike | RPD – relative percent difference |
| A – Analytical | LCSD – laboratory control spike duplicate | TOC – total organic carbon |
| AEL – Advanced Environmental Laboratories | LOQ – limit of quantitation | |
| DQI – data quality indicator | MPC – measurement performance criteria | |

WORKSHEET #12-4: MEASUREMENT PERFORMANCE CRITERIA - TOTAL ORGANIC CARBON IN GROUNDWATER AND SURFACE WATER

| Matrix | Groundwater and Surface Water | | |
|--|---|------------------------------------|--|
| Analytical Group/Method/SOP | TOC by SM 5310C / AEL WC-022 | | |
| Concentration Level | Low | | |
| DQI | QC Sample or Measurement Performance Activity | MPC | QC Sample Assesses Error for S, A, or both S&A |
| Precision | FDs | RPD ≤ 30% | S&A |
| Accuracy/Bias | LCS | %R 80-120% | A |
| Analytical Accuracy/Bias (matrix interference) | MS/MSD | %R 75-125% | S&A |
| Precision | LCSD and MSD | RPD ≤ 35% | A |
| Accuracy/Bias (contamination) | MB and/or EBs | No detections > LOQ | A |
| Completeness | Useable data (not rejected) | > 90% | S&A |
| Sensitivity | LOQ Verification Sample (spiked at LOQ) | Recovery within ±30% of true value | A |

Notes:

| | | |
|---|---|--|
| % – Percent | EB – equipment blank | MPC – measurement performance criteria |
| ± – plus or minus | EIS - extracted internal standard | MS – matrix spike |
| < – less than | FD – field duplicate | MSD – matrix spike duplicate |
| ≤ – less than or equal to | MB – method blank | QC – Quality Control |
| > – greater than | RPD – relative percent difference | RPD – relative percent difference |
| %R – percent recovery | LCS – laboratory control spike | SOP – Standard Operating Procedure |
| AEL – Advanced Environmental Laboratories | LCSD – laboratory control spike duplicate | |
| DQI – data quality indicator | LOQ – limit of quantitation | |

WORKSHEET #12-5: MEASUREMENT PERFORMANCE CRITERIA – Corrosivity/pH IN SOIL

| Matrix | Soil | | |
|-----------------------------|---|-------------------------------|--|
| Analytical Group/Method/SOP | pH - SW846 9045D/ AEL WC-057 | | |
| Concentration Level | Low | | |
| DQI | QC Sample or Measurement Performance Activity | MPC | QC Sample Assesses Error for S, A, or both S&A |
| Precision | Lab duplicates and LCS/LCSD | RPD from Worksheet #28 | A |
| Accuracy/Bias | LCS | %R from Worksheet #28 | A |
| Completeness | Useable data (not rejected) | > 90% | S&A |

Notes:

| | | |
|---|---|------------------------------------|
| % - percent | DQI – data quality indicator | QC – Quality Control |
| > greater than | RPD – relative percent difference | S - Sampling |
| %R – percent recovery | LCS – laboratory control spike | SOP – Standard Operating Procedure |
| A – Analytical | LCSD – laboratory control spike duplicate | |
| AEL – Advanced Environmental Laboratories | MPC – measurement performance criteria | |

WORKSHEET #12-6: MEASUREMENT PERFORMANCE CRITERIA - pH IN GROUNDWATER AND SURFACE WATER

| Matrix | Groundwater and Surface Water | | |
|---------------------|---|---------------------------|---|
| pH by SM9040 | pH by SM9040 / AEL WC-002 | | |
| Concentration Level | Low | | |
| DQI | QC Sample or Measurement Performance Activity | MPC | QC Sample Assesses Error for S, Analytical A, or both S&A |
| Precision | Lab duplicates and LCS/LCSD | RPD from Worksheet #28 | A |
| Accuracy/Bias | LCS | % Rec. from Worksheet #28 | A |
| Completeness | Useable data (not rejected) | > 90% | S&A |

Notes:

| | |
|---|---|
| > greater than | LCS – laboratory control spike |
| %R – percent recovery | LCSD – laboratory control spike duplicate |
| AEL – Advanced Environmental Laboratories | MPC – measurement performance criteria |
| DQI – data quality indicator | QC – Quality Control |
| RPD – relative percent difference | |

WORKSHEET #12-7: MEASUREMENT PERFORMANCE CRITERIA – GRAIN SIZE

| Matrix | Soil | |
|---------------------------------------|---|---|
| Analytical Group/Method/SOP | Grain Size/ (ASTM) D422 / (No Laboratory SOP associated with Grain Size Analysis) | |
| Concentration Level | Low | |
| DQI | QC Sample or Measurement Performance Activity | MPC |
| Analytical Accuracy/Bias (laboratory) | LCS | Meet limits of known reference material |
| Analytical Precision (laboratory) | DUP (optional DUP; optional) | 30% RPD |

Notes:

| | |
|---|------------------------------|
| % – percent | DQI – data quality indicator |
| ASTM – American Society for Testing and Materials International | DUP – duplicate (laboratory) |

LCS – laboratory control spike
MPC – measurement performance criteria
QC – Quality Control

RPD – relative percent difference
SOP – Standard Operating Procedure

WORKSHEET #13: SECONDARY DATA USES AND LIMITATIONS
(UFP-QAPP Manual Section 2.7)
(USEPA 2106-G-05 Chapter 3: QAPP Elements for Evaluating Existing Data)

| Data type | Source | Data Uses Relative to Current Project | Factors affecting the reliability of data and limitations on data use |
|-----------------------------------|--|---|---|
| Aerial Imagery | Esri™, ArcGIS Online Aerial Imagery | Provided georeferenced aerial photos for figure backdrops. | There are no known limitations on aerial imagery. |
| Past PFAS Site Investigations | Final PA and SI of PFAS, YTC, Washington (Arcadis, 2021) PA/SI Addendum – Off-post Private Well Investigations of PFAS. Yakima Training Center. Yakima, Washington. (SERES-Arcadis, 2023) PFAS data for primary water supply wells collected under (UCMR3) and IMCOM Operations Order 16-088 in 2013 and 2014, included in PA/SI (Arcadis, 2021) | Provided regional site conditions, historical site usage, historical contaminant identification and concentrations, and remedial actions. | Site usage histories may omit records of AFFF procurement and use. SI sample collection and laboratory analysis for PFAS constituents were conducted in accordance with approved Analytical Methods, SOPs, and Technical Guidance available at the time of sampling. |
| Installation Personnel Interviews | Various – collected during PA | Provided anecdotal histories of site use, AFFF use, and remedial actions completed. | Several installation personnel who would have worked on site during the peak of AFFF use are retired or out of contact. |

Notes:

- AFFF – aqueous film forming foam
- Arcadis – Arcadis, U.S., Inc.
- IMCOM – United States Army Installation Management Command
- PA – Preliminary Assessment
- PFAS – pre- and polyfluoroalkyl substances
- SERES - SERES Engineering & Services LLC
- SI – Site Investigation
- SOP – Standard Operating Procedures
- UCMR3 – Third Unregulated Contaminant Monitoring Rule
- YTC – Yakima Training Center

WORKSHEET #14 & 16: PROJECT TASKS & SCHEDULE
(UFP-QAPP Manual Section 2.8.2)
(USEPA 2106-G-05 Section 2.2.4)

The schedule will be updated as needed as part of the RI process. A formal project schedule is provided in **Appendix I**.

| Activity | Responsible Party | Planned Start Date | Planned Completion Date | Deliverable(s) | Deliverable Due Date |
|--|--------------------------------|-----------------------|--|--|------------------------|
| Project kick-off meeting | ECC/Arcadis | October 2022 | October 2022 | Meeting Minutes | Complete |
| Draft UFP-QAPP and SSHP | ECC/Arcadis | March 2023 | July 2023 (following POP extension to incorporate groundwater analytical data). | Draft UFP-QAPP and SSHP | September 2023 |
| Technical Project Scoping Meetings | ECC/Arcadis | January-February 2023 | January-February 2023 | Meeting Minutes | February 2023 |
| Baseline groundwater, surface water and sediment sampling | ECC/Arcadis | February 2023 | February 2023 | Analytical data package and electronic data deliverable; Data validation report | April 2023 |
| Mobilization and setup for Boundary Investigation | ECC/Arcadis and subcontractors | February 2023 | March 2023 | Permits, Utility Clearance | March 2023 |
| Boundary Well installation, development, and sampling | ECC/Arcadis and subcontractors | March 2023 | April 2023 | Analytical data package and electronic data deliverable; Data validation report | July 2023 |
| Final UFP-QAPP and SSHP | ECC/Arcadis | September 2023 | September 2023 | Final UFP-QAPP and SSHP | September 2023 |
| Coordinating/permitting for prescriptive sampling (RI Phase 1 Field Event) | ECC/Arcadis | September 2023 | November 2024 | Permits, ROEs and Utility Clearance | October 2024 |
| Mobilization and setup for RI Phase 1 Field Event | ECC/Arcadis and subcontractors | October 2023 | November 2024 | Field notes (included in RI Report) | October 2023 |
| Surface water and sediment sampling, soil boring advancement, sample collection of soil and groundwater, permanent well installation and boring abandonment for RI Phase 1 Field Event | ECC/Arcadis and subcontractors | October 2023 | November 2023 | Field notes and measurements (included in RI Report) | Submitted in RI Report |

| Activity | Responsible Party | Planned Start Date | Planned Completion Date | Deliverable(s) | Deliverable Due Date |
|---|--------------------------------|--------------------|-------------------------|--|------------------------|
| Laboratory analysis and Data Validation | ECC/Arcadis and laboratory | November 2023 | December 2023 | Analytical data package and EDD; DVR | Submitted in RI Report |
| Data analysis | ECC/Arcadis | January 2024 | January 2024 | TPP meeting presentation | February 2024 |
| Prescriptive Phase Data Review and Adaptive Phase Scoping Teleconference (TPP) | ECC/Arcadis | February 2024 | February 2024 | Meeting Minutes | February 2024 |
| Coordinating/permitting for adaptive sampling (RI Phase 2 Field Event) | ECC/Arcadis | March 2024 | April 2024 | Permits, ROEs and Utility Clearance | April 2024 |
| Mobilization and setup for RI Phase 2 Field Event | ECC/Arcadis and subcontractors | April 2024 | April 2024 | Field notes (included in RI Report) | April 2024 |
| Surface water and sediment sampling, soil boring advancement, collection of soil and groundwater samples, permanent well installation, boring abandonment. Locations dependent on results from RI Phase 2 Field Event | ECC/Arcadis and subcontractors | April 2024 | May 2024 | Field notes and measurements (included in RI Report) | Submitted in RI Report |
| Laboratory analysis and Data validation | ECC/Arcadis and Laboratory | May 2024 | June 2024 | Analytical data package and EDD; DVR | Submitted in RI Report |
| Data analysis | ECC/Arcadis | June 2024 | July 2024 | TPP meeting presentation | July 2024 |
| Adaptive Phase Data Review and Final Phase Scoping Teleconference (TPP) | ECC/Arcadis | August 2024 | August 2024 | Meeting Minutes | August 2024 |
| Coordinating/permitting for final phase sampling (RI Phase 3 Field Event) | ECC/Arcadis | August 2024 | September 2024 | Site permits | September 2024 |
| Mobilization and setup for RI Phase 3 Field Event | ECC/Arcadis and subcontractors | September 2024 | September 2024 | Field notes (included in RI Report) | September 2024 |
| Surface water and sediment sampling, soil boring advancement, collection of soil and groundwater samples, permanent well installation, boring abandonment. Scope dependent on requirements for final delineation and completion of monitoring well network based on results from RI Phase 3 Field Event. Sample collection from monitoring wells for sitewide monitoring. | ECC/Arcadis and subcontractors | September 2024 | September 2024 | Field notes and measurements (included in RI Report) | Submitted in RI Report |
| Laboratory analysis and Data validation | ECC/Arcadis and Laboratory | September 2024 | October 2024 | Analytical data package and EDD; DVR | Submitted in RI Report |

| Activity | Responsible Party | Planned Start Date | Planned Completion Date | Deliverable(s) | Deliverable Due Date |
|---|-------------------|--------------------|-------------------------|---|------------------------|
| Data analysis | ECC/Arcadis | October 2024 | November 2024 | TPP meeting presentation | December 2024 |
| Draft Refined CSM Tech Memo | ECC/Arcadis | January 2025 | March 2025 | Draft Refined CSM Tech Memo | March 2025 |
| Final Refined CSM Tech Memo | ECC/Arcadis | April 2025 | May 2025 | Final Refined CSM Tech Memo | June 2025 |
| Quarterly Groundwater Monitoring, Lab Analysis, and Data Validation | ECC/Arcadis | July 2025 | August 2025 | Field notes and measurements. Analytical data package and electronic data deliverable; Data validation report (included in RI Report) | Submitted in RI Report |
| | ECC/Arcadis | October 2025 | November 2025 | | |
| | ECC/Arcadis | January 2026 | February 2026 | | |
| | ECC/Arcadis | April 2026 | May 2026 | | |
| Draft RI Report | ECC/Arcadis | June 2026 | January 2027 | Draft RI Report | January 2027 |
| TPP Draft Final RI Report for regulatory review if necessary | ECC/Arcadis | April 2027 | June 2027 | Draft Final RI Report | July 2027 |
| Final RI Report | ECC/Arcadis | June 2027 | August 2027 | Final RI Report | September 2027 |

Notes:

- Arcadis – Arcadis U.S., Inc.
- CSM – Conceptual Site Model
- DVR – Data Validation Report
- ECC – Environmental Chemical Corporation
- EDD – electronic data deliverable
- QAPP – Quality Assurance Project Plan
- RI – Remedial Investigation
- ROE – Right of Entry
- SSHP – Site Safety and Health Plan
- TPP – technical project planning
- UFP – Uniform Federal Policy

**WORKSHEET #15: PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC
 DETECTION/QUANTITATION LIMITS
 (UFP-QAPP Manual Section 2.6.2.3)
 (USEPA 2106-G-05 Section 2.2.6)**

The following **Worksheet #15** tables identify the project action limits (PALs) and provide a comparison of the PALs to analytical laboratory reference limits (i.e., LODs and LOQs) for groundwater/surface water, and soil/sediment per analytical method. The objective is for the laboratory to achieve LODs low enough to measure analytes at concentrations less than the PALs to obtain a dataset of known quality and sufficient sensitivity to meet project DQOs. The PALs represent the lowest of the relevant human health screening levels and other applicable criteria that may be used in the RI and later stages of the CERCLA process.

As discussed in Worksheet 10.3, on 6 July 2022, the OSD issued a revised memorandum (OSD 2022) that provided technical guidance related to the May 2022 updated USEPA Regional Screening Levels (RSLs) for PFOS, PFOA, PFNA, PFHxS, and HFPO-DA, with PFBS remaining unchanged since the previous update (OSD 2021; USEPA, 2022b).

There are currently no human health screening criteria for PFOS, PFOA, PFBS, PFNA, PFHxS, or HFPO-DA in surface water or sediment; therefore, for reference, surface water and sediment results will be compared to groundwater and soil criteria (respectively).

PFAS results for soil, groundwater, surface water, and sediment will be evaluated based on the 2022 OSD risk screening levels for residential exposure using a hazard quotient of 0.1 for the purposes of this RI. A summary of the OSD residential screening levels that will be referenced during this RI is included as **Table 15-1**.

Table 15-1: Summary of OSD Risk Screening Levels

| Chemical of Concern | Matrix | Units | OSD Risk Screening Level (Residential ¹) |
|---------------------|---------------|-------|--|
| PFOS | Soil/sediment | mg/kg | 0.013 |
| PFOA | Soil/sediment | mg/kg | 0.019 |
| PFBS | Soil/sediment | mg/kg | 1.9 |
| PFNA | Soil/sediment | mg/kg | 0.019 |
| PFHxS | Soil/sediment | mg/kg | 0.13 |
| HFPO-DA | Soil/sediment | mg/kg | 0.023 |
| PFOS | Water | ng/L | 4 |
| PFOA | Water | ng/L | 6 |
| PFBS | Water | ng/L | 601 |
| PFNA | Water | ng/L | 6 |
| PFHxS | Water | ng/L | 39 |
| HFPO-DA | Water | ng/L | 6 |

Note:

¹Risk screening levels for tap water and soil/sediment provided by the OSD 2022. Memorandum: Investigation Per- and Polyfluoroalkyl Substances within the DoD Cleanup Program. September 15. (OSD 2022). These standards are not applicable for sediment or surface water but can be used as comparison values for reference for these environmental media. There are currently no human health screening criteria for PFOS, PFOA, PFBS, PFNA, PFHxS, or HFPO-DA in surface water or sediment; therefore, for reference, surface water and sediment results will be compared to groundwater and soil criteria (respectively).

The PALs are not intended to be used as cleanup levels. Concentrations above the PALs would not automatically trigger a response action but would suggest that further site-specific consideration is appropriate.

The analytical laboratory reference limits presented in **Worksheet #15** tables are as follows:

- LOD – The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.
- LOQ – The lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias.

The following worksheets provide Project Screening Levels and Laboratory-Specific Detection/Quantitation Limits for AEL:

- Worksheet #15-1: Reference Limits and Evaluation Tables (Drinking Water, Groundwater and Surface Water)
- Worksheet #15-2: Reference Limits and Evaluation Tables (Soil and Sediment)
- Worksheet #15-3: Reference Limits and Evaluation Tables (Specific Parameters in Soil)

Note: ELLE UFP-QAPP Worksheets (#15-1B through #15-6B) are provided in **Appendix G**.

WORKSHEET #15-1: REFERENCE LIMITS AND EVALUATION TABLES (GROUNDWATER AND SURFACE WATER)

**AEL
 (UFP-QAPP Manual Section 2.6.2.3)
 (USEPA 2106-G-05 Section 2.2.6)**

| Analyte | Acronym | CAS Number | Screening Standard ¹ (ng/L) | LOQ (ng/L) | LOD (ng/L) | DL (ng/L) |
|--|----------------|-------------|--|------------|------------|-----------|
| <i>PFAS – Drinking Water USEPA Method 537.1²</i> | | | | | | |
| Perfluorobutanesulfonic acid | PFBS | 375-73-5 | 601 ⁴ | 8 | 4 | 2 |
| Perfluorooctanoic acid | PFOA | 335-67-1 | 6 ⁴ | 8 | 4 | 2 |
| Perfluorooctanesulfonic acid ² | PFOS | 1763-23-1 | 4 ⁴ | 8 | 4 | 2 |
| N-Ethyl-perfluorooctane sulfonamidoacetic acid | NEtFOSAA | 2991-50-6 | 4 | 8 | 4 | 2 |
| N-Methyl-perfluorooctane sulfonamidoacetic acid | NMeFOSAA | 2355-31-9 | 4 | 8 | 4 | 2 |
| Perfluorodecanoic acid | PFDA | 335-76-2 | 4 | 8 | 4 | 2 |
| Perfluorododecanoic acid | PFDoA | 307-55-1 | 4 | 8 | 4 | 2 |
| Perfluoroheptanoic acid | PFHpA | 375-85-9 | 4 | 8 | 4 | 2 |
| Perfluorohexanoic acid | PFHxA | 307-24-4 | 4 | 8 | 4 | 2 |
| Perfluorohexanesulfonic acid | PFHxS | 355-46-4 | 39 ⁴ | 8 | 4 | 2 |
| Perfluorononanoic acid | PFNA | 375-95-1 | 6 ⁴ | 8 | 4 | 2 |
| Perfluorotetradecanoic acid | PFTA | 376-06-7 | 4 | 8 | 4 | 2 |
| Perfluorotridecanoic acid | PFTrDA | 72629-94-8 | 4 | 8 | 4 | 2 |
| Perfluoroundecanoic acid | PFUnA | 2058-94-8 | 4 | 8 | 4 | 2 |
| 11-Chloroeicosfluoro-3-oxaundecane-1-sulfonic acid | 11Cl-PF3Uds | 763051-92-9 | 4 | 8 | 4 | 2 |
| 9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid | 9Cl-PF3ONS | 756426-58-1 | 4 | 8 | 4 | 2 |
| 4,8-Dioxa-3H-perfluorononanoic acid | ADONA | 919005-14-4 | 4 | 8 | 4 | 2 |
| Hexafluoropropylene oxide dimer acid | HFPO-DA (GenX) | 13252-13-6 | 6 ⁴ | 8 | 4 | 2 |
| <i>PFAS – Groundwater and Surface Water Draft USEPA 1633 and DoD QSM 5.4 Table B-24²</i> | | | | | | |
| Perfluorobutanesulfonic acid | PFBS | 375-73-5 | 601 ⁴ | 10 | 5 | 2.5 |
| Perfluorooctanoic acid | PFOA | 335-67-1 | 6 ⁴ | 10 | 5 | 2.5 |
| Perfluorooctanesulfonic acid ² | PFOS | 1763-23-1 | 4 ⁴ | 10 | 5 | 2.5 |
| 1H,1H,2H,2H-Perfluorohexanesulfonic acid | 4:2 FTS | 757124-72-4 | 5 | 10 | 5 | 2.5 |
| 1H,1H,2H,2H-Perfluorooctanesulfonic acid | 6:2 FTS | 27619-97-2 | 5 | 10 | 5 | 2.5 |

| Analyte | Acronym | CAS Number | Screening Standard ¹ (ng/L) | LOQ (ng/L) | LOD (ng/L) | DL (ng/L) |
|--|----------------|--------------|--|------------|------------|-----------|
| 1H,1H,2H,2H-Perfluorodecanesulfonic acid | 8:2 FTS | 39108-34-4 | 5 | 10 | 5 | 2.5 |
| Perfluorooctane sulphonamide | FOSA | 754-91-6 | 5 | 10 | 5 | 2.5 |
| N-Ethyl-perfluorooctane sulfonamidoacetic acid | NEtFOSAA | 2991-50-6 | 5 | 10 | 5 | 2.5 |
| N-Methyl-perfluorooctane sulfonamidoacetic acid | NMeFOSAA | 2355-31-9 | 5 | 10 | 5 | 2.5 |
| Perfluorobutanoic acid | PFBA | 375-22-4 | 5 | 10 | 5 | 2.5 |
| Perfluoro-4-methoxybutanoic acid | PFMBA | 863090-89-5 | 5 | 10 | 5 | 2.5 |
| Perfluoro-3-methoxypropanoic acid | PFMPA | 377-73-1 | 5 | 10 | 5 | 2.5 |
| Perfluorodecanoic acid | PFDA | 335-76-2 | 5 | 10 | 5 | 2.5 |
| Perfluorodecanesulfonic acid | PFDS | 335-77-3 | 5 | 10 | 5 | 2.5 |
| Perfluorododecanoic acid | PFDoA | 307-55-1 | 5 | 10 | 5 | 2.5 |
| Perfluorododecanesulfonic acid | PFDoS | 79780-39-5 | 5 | 10 | 5 | 3 |
| Perfluoroheptanoic acid | PFHpA | 375-85-9 | 5 | 10 | 5 | 2.5 |
| Perfluoroheptanesulfonic acid | PFHpS | 375-92-8 | 5 | 10 | 5 | 2.5 |
| Perfluorohexanoic acid | PFHxA | 307-24-4 | 5 | 10 | 5 | 2.5 |
| Perfluorohexanesulfonic acid | PFHxS | 355-46-4 | 39 ⁴ | 10 | 5 | 2.5 |
| Perfluorononanoic acid | PFNA | 375-95-1 | 6 ⁴ | 10 | 5 | 2.5 |
| Perfluoronananesulfonic acid | PFNS | 68259-12-1 | 5 | 10 | 5 | 2.5 |
| Perfluoropentanoic acid | PFPeA | 2706-90-3 | 5 | 10 | 5 | 2.5 |
| Perfluoropentanesulfonic acid | PFPeS | 2706-91-4 | 5 | 10 | 5 | 2.5 |
| Perfluorotetradecanoic acid | PFTA | 376-06-7 | 5 | 10 | 5 | 2.5 |
| Perfluorotridecanoic acid | PFTrDA | 72629-94-8 | 5 | 10 | 5 | 2.5 |
| Perfluoroundecanoic acid | PFUnA | 2058-94-8 | 5 | 10 | 5 | 2.5 |
| 3-Perfluoropropyl propanoic acid | 3:3FTCA | 356-02-5 | 20 | 40 | 20 | 10 |
| 2H,2H,3H,3H-Perfluorooctanoic acid | 5:3FTCA | 914637-493-3 | 100 | 200 | 100 | 50 |
| 3-Perfluoroheptyl propanoic acid | 7:3FTCA | 812-70-4 | 100 | 200 | 100 | 50 |
| Perfluoro(2-ethoxyethane)sulfonic acid | PFEESA | 113507-82-7 | 5 | 10 | 5 | 2.5 |
| Nonafluoro-3,6-dioxahexanoic acid | NFDHA | 151772-58-6 | 5 | 10 | 5 | 4 |
| Hexafluoropropylene oxide dimer acid | HFPO-DA (GenX) | 13252-13-6 | 6 ⁴ | 10 | 5 | 2.5 |
| 4,8-Dioxa-3H-perfluorononanoic acid | ADONA | 919005-14-4 | 5 | 10 | 5 | 2.5 |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid | 9C1-PF3ONS | 7569426-58-1 | 5 | 10 | 5 | 2.5 |

| Analyte | Acronym | CAS Number | Screening Standard ¹ (ng/L) | LOQ (ng/L) | LOD (ng/L) | DL (ng/L) |
|---|--------------|-------------|--|------------|------------|-----------|
| 11-Chloroeicosafiuoro-3-oxaundecane-1-sulfonic acid | 11C1-PF3OUdS | 763051-92-9 | 5 | 10 | 5 | 2.5 |
| N-methyl perfluorooctanesulfonamidoethanol | N-Me-FOSE | 24448-09-7 | 50 | 100 | 50 | 25 |
| N-methyl perfluorooctanesulfonamide | N-Me-FOSA | 31506-32-8 | 5 | 10 | 5 | 2.5 |
| N-ethyl perfluorooctanesulfonamidoethanol | N-Et-FOSE | 1691-99-2 | 50 | 100 | 50 | 25 |
| N-ethyl perfluorooctanesulfonamide | N-Et-FOSA | 4151-50-2 | 5 | 10 | 5 | 2.5 |

Notes:

1. PFAS Screening Standard is based on LOD unless a DoD- or state-specific human health screening standard is used. Freshwater screening levels developed by Argonne National Laboratory (ANL 2021) to be used in the screening-level ecological risk assessment are greater than the human health risk-based values for PFBS, PFOA, and PFOS. Therefore, the ecological screening levels are also greater than the laboratory limits.
2. DoD-ELAP compliant method.
3. The branched chain isomers are included in the integrations and quantification of standards and field samples for PFOA, PFOS, PFHxS, NMeFOSAA and NEtFOSAA. Branch chained isomers of PFOS, PFHxS, NMeFOSAA and NEtFOSAA are included in the calibration standards for these compounds and therefore the areas of the linear and branched isomers are integrated together for these compounds in the calibration curves.
4. DoD Memorandum (06 July 2022) Investigating Per- and Polyfluoroalkyl Substances within the Department of Defense Cleanup Program. Screening criteria are risk screening levels for tap water provided by the OSD residential tap water regional screening levels calculated using USEPA online calculator (May 2022), where the hazard quotient equals 0.1.

CAS – Chemical Abstract Service
 DoD – Department of Defense
 DL – detection limit
 ELAP – Environmental Laboratory Accreditation Program
 LOD – limit of detection
 LOQ – limit of quantitation

N/A – not applicable
 ng/L – nanograms per Liter
 OSD – Office of the Secretary of Defense
 PFAS – Per and Polyfluoroalkyl substances
 QSM – Quality Systems Manual
 USEPA – United States Environmental Protection Agency

WORKSHEET #15-2: REFERENCE LIMITS AND EVALUATION TABLES (SOIL AND SEDIMENT)

AEL

**(UFP-QAPP Manual Section 2.6.2.3)
 (USEPA 2106-G-05 Section 2.2.6)**

| Analyte | Acronym | CAS Number | Screening Standard ¹ (µg/kg) | LOQ (µg/kg) | LOD (µg/kg) | DL (µg/kg) |
|--|----------|-------------|---|-------------|-------------|------------|
| <i>PFAS – Solids Draft EPA 1633 and DoD QSM 5.4 Table B-24²</i> | | | | | | |
| Perfluorobutanesulfonic acid | PFBS | 375-73-5 | 1,900 ⁴ | 1.0 | 0.5 | 0.25 |
| Perfluorooctanoic acid | PFOA | 335-67-1 | 19 ⁴ | 1.0 | 0.5 | 0.25 |
| Perfluorooctanesulfonic acid ² | PFOS | 1763-23-1 | 13 ⁴ | 1.0 | 0.5 | 0.35 |
| 1H,1H,2H,2H-Perfluorohexanesulfonic acid | 4:2 FTS | 757124-72-4 | 0.5 | 1.0 | 0.5 | 0.25 |
| 1H,1H,2H,2H-Perfluorooctanesulfonic acid | 6:2FTS | 27619-97-2 | 0.5 | 1.0 | 0.5 | 0.3 |
| 1H,1H,2H,2H-Perfluorodecanesulfonic acid | 8:2FTS | 39108-34-4 | 0.5 | 1.0 | 0.5 | 0.25 |
| Perfluorooctane sulphonamide | PFOSA | 754-91-6 | 0.5 | 1 | 0.5 | 0.25 |
| N-Ethyl-perfluorooctane sulfonamidoacetic acid | NEtFOSAA | 2991-50-6 | 0.5 | 1 | 0.5 | 0.25 |
| N-Methyl-perfluorooctane sulfonamidoacetic acid | NMeFOSAA | 2355-31-9 | 0.5 | 1 | 0.5 | 0.25 |
| Perfluorobutanoic acid | PFBA | 375-22-4 | 0.5 | 1 | 0.5 | 0.25 |
| Perfluorodecanoic acid | PFDA | 335-76-2 | 0.5 | 1 | 0.5 | 0.25 |
| Perfluorodecanesulfonic acid | PFDS | 335-77-3 | 0.5 | 1 | 0.5 | 0.25 |
| Perfluorododecanoic acid | PFDoA | 307-55-1 | 0.5 | 1.0 | 0.5 | 0.25 |
| Perfluorododecanesulfonic acid | PFDoS | 79780-39-5 | 1.0 | 2.0 | 1.0 | 0.5 |
| Perfluoroheptanoic acid | PFHpA | 375-85-9 | 0.5 | 1.0 | 0.5 | 0.25 |
| Perfluoroheptanesulfonic acid | PFHpS | 375-92-8 | 0.5 | 1.0 | 0.5 | 0.35 |
| Perfluorohexanoic acid | PFHxA | 307-24-4 | 0.5 | 1.0 | 0.5 | 0.25 |
| Perfluorohexanesulfonic acid | PFHxS | 355-46-4 | 130 ⁴ | 1.0 | 0.5 | 0.25 |
| Perfluorononanoic acid | PFNA | 375-95-1 | 19 ⁴ | 1.0 | 0.5 | 0.25 |
| Perfluorononanesulfonic acid | PFNS | 68259-12-1 | 0.5 | 1.0 | 0.5 | 0.4 |
| Perfluoropentanoic acid | PFPeA | 2706-90-3 | 0.5 | 1.0 | 0.5 | 0.25 |
| Perfluoropentanesulfonic acid | PFPeS | 2706-91-4 | 0.5 | 1.0 | 0.5 | 0.25 |
| Perfluorotetradecanoic acid | PFTeDA | 376-06-7 | 0.5 | 1.0 | 0.5 | 0.25 |
| Perfluorotridecanoic acid | PFTrDA | 72629-94-8 | 0.5 | 1.0 | 0.5 | 0.25 |

| Analyte | Acronym | CAS Number | Screening Standard ¹ (µg/kg) | LOQ (µg/kg) | LOD (µg/kg) | DL (µg/kg) |
|---|----------------|--------------|---|-------------|-------------|------------|
| Perfluoroundecanoic acid | PFUnA | 2058-94-8 | 0.5 | 1 | 0.5 | 0.25 |
| Hexafluoropropylene oxide dimer acid | HFPO-DA (GenX) | 13232-13-6 | 23 ⁴ | 1.0 | 0.5 | 0.25 |
| 4,8-Dioxa-3H-perfluorononanoic acid | ADONA | 919005-14-4 | 0.5 | 1.0 | 0.5 | 0.25 |
| Perfluoro-3-methoxypropanoic acid | PFMPA | 377-73-1 | 0.5 | 1.0 | 0.5 | 0.25 |
| 3-Perfluoropropyl propanoic acid | 3:3 FTCA | 356-02-5 | 2.0 | 4.0 | 2.0 | 1.0 |
| 2H,2H,3H,3H-Perfluorooctanoic acid | 5:3FTCA | 914637-493-3 | 10 | 20 | 10 | 5.0 |
| 3-Perfluoroheptyl propanoic acid | 7:3FTCA | 812-70-4 | 10 | 20 | 10 | 5.0 |
| Perfluoro-4-methoxybutanoic acid | PFMBA | 863090-89-5 | 0.5 | 1.0 | 0.5 | 0.25 |
| Perfluoro(2-ethoxyethane)sulfonic acid | PFEESA | 113507-82-7 | 0.5 | 1.0 | 0.5 | 0.25 |
| Nonafluoro-3,6-dioxaheptanoic acid | NFDHA | 151772-58-6 | 1.0 | 2.0 | 1.0 | 0.5 |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid | 9C1-PF3ONS | 7569426-58-1 | 0.5 | 1.0 | 0.5 | 0.25 |
| 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid | 11C1-PF3OUdS | 763051-92-9 | 0.5 | 1.0 | 0.5 | 0.25 |
| N-methyl perfluorooctanesulfonamidoethanol | N-Me-FOSE | 24448-09-7 | 5.0 | 10 | 5.0 | 2.5 |
| N-methyl perfluorooctanesulfonamide | N-Me-FOSA | 31506-32-8 | 0.5 | 1.0 | 0.5 | 0.25 |
| N-ethyl perfluorooctanesulfonamidoethanol | N-Et-FOSE | 1691-99-2 | 5.0 | 10 | 5.0 | 2.5 |
| N-ethyl perfluorooctanesulfonamide | N-Et-FOSA | 4151-50-2 | 0.5 | 1.0 | 0.5 | 0.25 |

Notes:

1. PFAS Screening Standard is based on LOD unless a DoD- or state-specific human health screening standard is used. Soil screening levels developed by Argonne National Laboratory (ANL 2021) to be used in the screening-level ecological risk assessment are greater than the human health risk-based values for PFBS and PFOA. For PFOS, the soil screening level for terrestrial mammals is 8.7 µg/kg. This ecological screening level is below the human health risk-based value but still greater than the laboratory limits.
2. DoD- ELAP compliant method.
3. The branched chain isomers are included in the integrations and quantification of standards and field samples for PFOA, PFOS, PFHxS, NMeFOSAA and NEtFOSAA. Branch chained isomers of PFOS, PFHxS, NMeFOSAA and NEtFOSAA are included in the calibration standards for these compounds and therefore the areas of the linear and branched isomers are integrated together for these compounds in the calibration curves.
4. DoD Memorandum (06 July 2022) Investigating Per- and Polyfluoroalkyl Substances within the Department of Defense Cleanup Program. Screening criteria are risk screening levels for soil provided by the OSD residential tap water regional screening levels calculated using USEPA online calculator (May 2022), where the hazard quotient equals 0.1.

CAS – Chemical Abstract Service
 DL – detection limit
 DoD – Department of Defense

ELAP – Environmental Laboratory Accreditation Program
 LOD – limit of detection
 LOQ – limit of quantitation
 µg/kg – micrograms per kilogram
 N/A – not applicable
 OSD – Office of the Secretary of Defense
 PFAS – per- and polyfluoroalkyl substances
 SU – standard units
 TOC – total organic carbon
 USEPA – United States Environmental Protection Agency

WORKSHEET #15-3: REFERENCE LIMITS AND EVALUATION TABLES (SPECIFIC PARAMETERS IN SOIL)

AEL

| Analyte | Acronym | CAS Number | Screening Standard (mg/kg) | LOQ (mg/kg) | LOD (mg/kg) ⁵ | DL (mg/kg) |
|--|---------|------------|----------------------------|-------------|--------------------------|------------|
| <i>ASTM D422 Grain Size</i> | | | | | | |
| Grain Size | N/A | N/A | N/A | N/A | N/A | N/A |
| <i>pH – Solids USEPA 9045D¹</i> | | | | | | |
| pH | pH | N/A | N/A | 0.1 su | N/A | N/A |
| <i>TOC – USEPA 9060A¹</i> | | | | | | |
| Total Organic Carbon | TOC | N/A | N/A | 10,000 | 5,000 | 1,620 |

Notes:

1. DoD- ELAP compliant method.
2. The primary goal is to obtain data for those analytes with promulgated limits to be reported within the instrument calibration range (i.e., no diluted-out data or ‘E’ qualified [exceeding calibration range hits] and that dilutions will raise the LOD (hence, the QAPP screening level) for non-promulgated compounds.

ASTM – American Society for Testing and Materials
 CAS – Chemical Abstract Service
 DL – detection limit
 LOD – limit of detection
 LOQ – limit of quantitation
 mg/kg – milligrams per kilogram

N/A – not applicable
SU – standard units
TOC – total organic carbon

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WORKSHEET #17: GENERAL INVESTIGATION DESIGN AND WORKFLOW

PFAS Sampling Activities

(UFP-QAPP Manual Section 3.1.1)/(USEPA 2106-G-05 Section 2.3.1)

This worksheet describes the design of the field investigation to determine the nature and extent of PFAS impacts in the subsurface.

17.1 Overall Approach Rationale

Releases of PFAS-containing materials at YTC have led to the presence of PFAS in environmental media at concentrations that could potentially pose a threat of adverse effects to human health and/or ecological receptors. The results of the 2020 SI sampling event identified seven AOIs that have PFOS, PFOA, PFBS, PFNA, PFHxS, and/or HFPO-DA above applicable screening criteria in soil and groundwater attributed to the use of AFFF and/or other sources of PFAS-containing materials (Arcadis 2021). The objectives of the sampling approaches for this RI are to provide data to determine the nature and extent of PFOS, PFOA, PFBS, PFNA, PFHxS, and/or HFPO-DA for each AOI, refine the preliminary CSMs for each AOI, and assess and quantify potential impacts to human health posed by PFAS in environmental media. The general investigation design for this RI was developed to ensure that the amount, type, and quality of data are sufficient to meet these objectives.

As stated in **Worksheet #11**, the site characterization approach includes systematic planning and sequencing of work that uses a combination of prescriptive locations (pre-determined based on available site data and completed as part of the initial mobilization for this RI) and adaptive locations, which include “step-outs” to complete delineation of lateral and vertical impacts and “step-ins” to zoom in on source hotspots or the core of the groundwater impacts. Adaptive locations will be finalized following review of analytical data generated during the preceding phase and documented in a Field Change Form (**Appendix J**). Field Change Forms will be shared with regulators and stakeholders to present the rationale for further investigation methods and/or locations, as needed. The flow of work proposed for site characterization activities facilitates the progression from the existing base CSM to an informed flux-based CSM, as illustrated in the flow chart included as **Exhibit 2**.

17.2 Field Activities

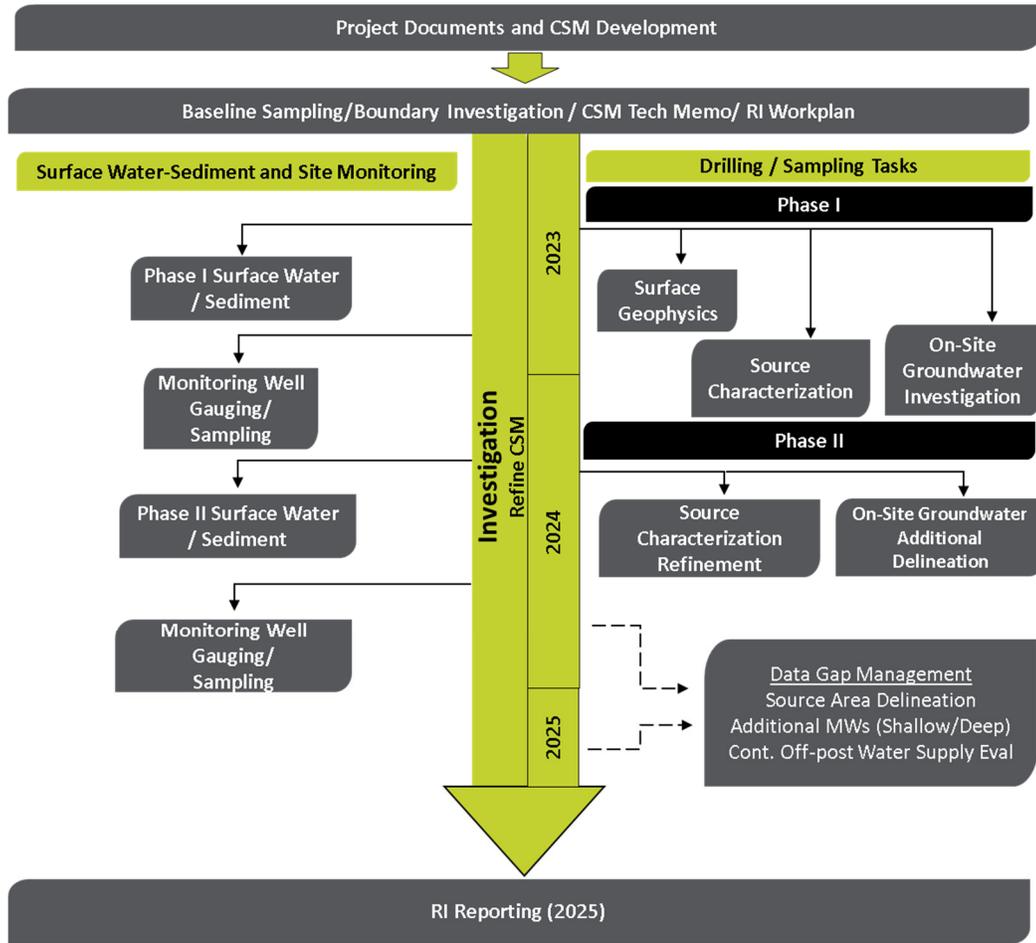
The field team will collect soil, groundwater, surface water, and sediment samples (if appropriate based on AOI-specific CSMs) to fill data gaps identified during the PA/SI, define the nature and extent of contamination, and support the risk assessment. Methods for sampling each medium are provided in the subsequent sections.

The investigation is sequenced to accommodate a data-driven approach, wherein each step is guided by data obtained from completed work, resulting in a more efficient characterization based on a systematic evaluation of information inputs. The field work is sequenced into phases (Phases I, II, and III) to capitalize on readily obtainable data, focus more intrusive data collection, and continuously refine the CSM. Each phase of the investigation is intended to be completed during a separate mobilization.

Phase I

The RI Phase I Field Event will include surface geophysical surveys, source area soil borings, monitoring well installations, and shallow groundwater sampling at locations selected based on evaluation of existing data. Locations will be strategically selected with the intent to resolve existing data gaps identified in the 2023 CSM Technical Memo (ECC/Arcadis 2023a).

Exhibit 2. Project Workflow Chart



A sitewide surface water and sediment sampling event will be completed in parallel with the Phase I drilling program based on analysis of the results of the baseline surface water and sediment data collected during November 2022, and will include additional locations with step-out samples located 1,000 to 2,000 feet downstream of baseline exceedance locations or at the nearest confluences. These samples will be collected to account for seasonality. Additional surface water and sediment sampling may be completed during the adaptive phase of sampling if further delineation is necessary.

AOI-specific investigation approaches were developed for AOIs or AOI groupings, as summarized below and in **Table 17-1**.

Table 17- 1: RI Phase I Sampling Scope of Work

| Area | Sampling Scope of Work |
|--|--|
| Main Cantonment Area (Non-AOI Specific) Figure 17-1 | <ul style="list-style-type: none"> Surface Geophysics – An electromagnetic induction survey will be completed to investigate the extent of the Pomona Basalt in the area shown on Figure 17-1. Surface water – Surface water samples will be collected from two locations within the irrigation canal (Figure 17-1). Sediment samples were collected from these locations during the baseline event; however, the canal was dry at the time of sampling. |

| Area | Sampling Scope of Work |
|---|--|
| Former Fire Training Pit (AOI 1) and Bird Bath Wash Rack (AOI 2) Figure 17-2 | <ul style="list-style-type: none"> Soil – Surface soil samples will be collected from 12 locations (Figure 17-2) to define the lateral extent of the PFAS release areas. Ten soil borings will be advanced to bedrock (approximately 6 feet bgs) and two soil samples will be collected from each boring (surface soil [0 to 0.5 ft bgs] and a 1-foot interval directly above bedrock). Data will supplement existing data, delineate source impacts, and support risk assessment evaluations. Groundwater – Monitoring wells will be installed at four locations (Figure 17-2) to intersect the first water bearing zone within the basalt (fractured/vesiculated zone between 15 and 30 feet bgs) using air rotary drilling methods. Monitoring wells will be sampled for PFAS. |
| AFFF Storage Area (Building 821) (AOI 3) Figure 17-3 | <ul style="list-style-type: none"> Soil – Six soil borings (Figure 17-3) will be advanced to a depth of 10 feet bgs and three soil samples will be collected from each boring (surface soil [0 to 0.5 ft bgs] and two subsurface soils at nominal 5-foot intervals). Data will supplement existing data, delineate source impacts, and support risk assessment evaluations. Groundwater – Monitoring wells will be installed at two locations (Figure 17-3) to intersect the first water bearing zone within the basalt (fracture zone between 80 and 100 feet bgs) using air rotary drilling methods. Monitoring wells will be sampled for PFAS. Surface water – If water is present at locations reported as dry during the baseline event, a surface water sample will be collected. |
| Refractometer Solutions Test Area (AOI 4), Fire Station 29 (Building 346, AOI 5), and AFFF Storage Area (Building 321, AOI 6) Figure 17-4 | <ul style="list-style-type: none"> Soil – Surface soil samples will be collected from up to 22 locations (Figure 17-4) to define the lateral extent of the PFAS release areas. Two soil borings will be advanced to bedrock (approximately 30 to 40 feet bgs) and soil samples will be collected at nominal 5-ft intervals from ground surface to 20 feet bgs and at nominal 10-foot intervals from 20 ft bgs until bedrock is encountered. Data will supplement existing data, delineate source impacts, and support risk assessment evaluations. Groundwater – Monitoring wells will be installed at two locations (Figure 17-4) to intersect the first water bearing zone within the basalt (fracture zone between 80 and 100 feet bgs) using air rotary drilling methods. Monitoring wells will be sampled for PFAS. Surface water/Sediment – Surface water and sediment samples will be collected from approximately four locations (includes resampling of two baseline sample locations to evaluate seasonal variability). |
| Mettie Airstrip (AOI 7) (formerly Selah Airstrip) Figure 17-5 | <ul style="list-style-type: none"> Surface Geophysics – Two surface resistivity transects (Figure 17-5) will be completed to characterize the lithology, identify water bearing zones and potential preferential pathways and guide the placement and installation of monitoring wells during the RI Phase II Field Event. Soil – Surface soil samples will be collected from up to 10 locations (Figure 17-5) to define the lateral extent of the PFAS release areas and determine soil boring locations to provide vertical delineation during RI Phase II. Groundwater – The RI Phase I Field Event does not include any groundwater sampling at Mettie Airstrip and is intended to refine the CSM allowing for effective placement of monitoring wells during RI Phase II. Surface water/Sediment – Surface water and sediment samples will be collected from approximately four locations (three locations were sampled previously and are included for surface water sampling only; Figure 17-5). |

Notes:

AFFF – aqueous film-forming foam
 AOI – Area of Interest
 bgs – below ground surface

PFAS – per- and polyfluoroalkyl substances
 RI – Remedial Investigation

Phase II

Based on Phase I analytical results, the CSMs will be updated, and step-out/step-in locations (soil borings, surface water/sediment samples, and monitoring well locations) will be selected based on the logic provided in **Section 17.3** to complete vertical and lateral delineation in soil, groundwater, surface water, and sediment. Sampling numbers and locations will be determined based on review of the prescriptive (Phase I) data and discussed with Regulators and stakeholders as applicable during periodic project updates and collaboration calls for input regarding adaptive sampling locations.

Surface Water/Sediment Sampling

A second round of sitewide surface water sampling will be completed approximately 6 months from the Phase I sampling event to account for seasonality. The second sampling event will include any step-out sampling locations, in addition to locations sampled during Phase I. No additional sediment sampling is planned, as sediment samples will be collected during the prescriptive phase, and seasonality is not expected to affect sediment PFAS concentrations.

Phase III

Where necessary, additional monitoring well installation and sampling will be completed, based on data collected during the previous phases of work, to provide sufficient infrastructure to monitor any PFAS plumes in groundwater, and address remaining data gaps. A synoptic groundwater monitoring event will be completed and will include groundwater elevation gauging and sampling of all new monitoring wells and select existing monitoring wells.

17.3 Adaptive Decision Logic

The RI will proceed using an adaptive site characterization approach to systematically define the nature and extent of PFAS impacts at each AOI. The approach will include the following steps: 1) use existing data to develop Installation-wide CSMs and AOI-specific CSMs to understand fate and transport and support a human health risk assessment; 2) develop each phase of investigation work, which may include various surface geophysical surveys in advance of drilling activities, to refine borehole locations and identify intervals to target for well construction and/or sampling and document in a Field Change Form; 3) analyze data from the preceding phase develop additional step-out/step-in locations. **Table 17-2** presents the decision logic for developing the subsequent scopes of work based on findings and interpretations. In addition to the analytical results-based logic presented in Table 7, interpretation of geophysical survey results will also be used to optimize sampling locations during subsequent phases.

Table 17-2: Adaptive Sampling Logic

| PFAS Characterization and/or Delineation Status (based on PFAS concentrations and/or AOI-specific conditions) | Contingency Action(s) |
|--|---|
| <i>Soil Delineation</i> | |
| 1. Delineation soil sample results less than OSD risk screening level(s) | 1. Soil delineation is complete in that direction. No additional soil samples are needed. |
| 2. Delineation soil sample results between 1 to 10 times the OSD risk screening level(s) | 2. Step out one to two times at the same soil boring spacing at representative locations. |
| 3. Delineation soil sample results greater than 10 times the OSD risk screening level(s) | 3. Step out at each location one and a half to two times at the soil boring spacing. |
| <i>Groundwater Delineation</i> | |
| 1. Plume delineation groundwater sample results-less than the OSD risk screening level(s) | 1. Delineation is complete in that direction. No additional groundwater monitoring wells are needed in that direction. Continue to monitor groundwater quality at this or nearby locations to verify results. |

| PFAS Characterization and/or Delineation Status (based on PFAS concentrations and/or AOI-specific conditions) | Contingency Action(s) |
|---|--|
| 2. Plume delineation groundwater sample results greater than OSD risk screening level(s) and associated soil sample results less than OSD risk screening level(s) | 2. Delineation of soil impacts is complete in that direction. Groundwater impacts will require further delineation with additional monitoring wells. Step out locations will be selected based on observed concentration gradients, and the location of known or suspected transport pathways. |
| 3. Plume delineation groundwater sample results and associated soil sample results greater than OSD risk screening level(s) | 3. Collect step-out soil samples from additional borings. Groundwater impacts will require further delineation using samples from existing monitoring wells (if available) and/or step out soil borings converted to monitoring wells. Step out locations will be selected based on observed concentration gradients, and the location of known or suspected transport pathways. Collect one deeper groundwater sample from at least one location to provide vertical delineation. |
| Surface Water Delineation | |
| 1. Surface water sample results less than OSD risk screening level(s) | 1. If surface water results are less than OSD risk screening level(s) for tap water, delineation is complete in that direction. No additional sample locations are needed. |
| 2. Surface water sample results greater than OSD risk screening level(s) | 2. Additional downstream sampling will be completed at locations 1,000 to 2,000 feet downgradient of the exceedance location or at the next confluence with another stream/drainage channel. Downgradient sampling will continue until concentrations are below screening level(s) or potential contributions from off-post sources are identified and complicate delineation of site-related contributions. |
| Sediment Delineation | |
| 1. Sediment sample results less than OSD risk screening level(s) | 1. If sediment results are less than OSD risk screening level(s), delineation is complete in that direction. No additional sample locations are needed. Repeat sampling at this location only if surface water quality changes throughout the RI. |
| 2. Sediment sample results greater than OSD risk screening level(s) | 2. Additional downstream sampling will be completed at locations 1,000 to 2,000 feet downgradient of the exceedance location or at the next confluence with another stream/drainage channel. Downgradient sampling will continue until concentrations are below screening level(s) or potential contributions from off-post sources are identified and complicate delineation of site-related contributions. |

Notes:

AOI – Area of interest

OSD – Office of the Secretary of Defense

PFAS – per- and polyfluoroalkyl substances

RI – Remedial Investigation

17.4 Field Methods

All field activities will be conducted in accordance with the approved Accident Prevention Plan / Site Safety and Health Plan (**Appendix H**). All samples will be collected in accordance with the field SOPs and/or Technical Guidance Instructions (TGIs) listed in **Worksheet #21** and provided in **Appendix K**, which account for PFAS-specific sampling guidelines. The methods described in the SOPs/TGIs will establish equipment requirements, procedures for preparing equipment and containers before sampling, sampling procedures under various conditions, proper procedures for storing and shipping samples, and laboratory analysis. Sampling techniques implemented for PFAS will be consistent with conventional environmental sampling techniques, with special considerations for PFAS-containing materials, equipment, and the high

potential for cross-contamination.

Field Change Forms will be used to document any major changes to analytical and field methodologies. A template for a Field Change Form is provided in **Appendix J**. For example, Field Change Forms will be prepared for the following changes:

- Analytical methods
- Project screening or action levels
- Sample collection methods
- Sample locations shifted more than 50 feet from the originally proposed location.

Surface Geophysics

Two surface geophysical survey methods will be utilized to address the following data gaps identified in the 2023 CSM Technical Memo (ECC/Arcadis 2023a):

- Mettie Airstrip - Migration pathways at and downgradient of the Mettie Airstrip are unknown due to a lack of subsurface data and distance from the other AOIs.
- Main cantonment area and installation boundary - The mechanism for vertical migration of perched groundwater to the underlying sand-rich units of the Ellensburg Fm is not completely understood. Some vertical groundwater transport may occur through fractured zones within the Pomona Member. However, the more massive sections of the Pomona Member likely enhance lateral migration along the surface of the basalt until it encounters the edge of the Pomona Member and infiltrates into the more permeable Ellensburg Fm. If lateral groundwater migration occurs along the surface of the Pomona Member, a refined understanding of the extent of the Pomona Member will be critical to defining the migration pathway.

Electrical resistivity imaging (ERI) methods, previously implemented as a component of the installation boundary geophysical survey, will be used at Mettie Airstrip to develop interpreted subsurface profiles, estimate the depth of the water table, identify different subsurface materials, and identify subsurface anomalies likely to serve as the more permeable zones. Interpreted ERI transect results will be incorporated to refine the CSM for Mettie Airstrip and select appropriate locations and depths for monitoring wells during subsequent mobilizations. ERI will be completed along two transects (shown on **Figure 17-5**), each approximately 3,640 linear feet which will be arrayed to achieve subsurface resolution to an approximate depth of 300 to 400 ft bgs. ERI data collection, and processing will be completed in accordance with the Geophysical Investigation TGI for Investigation Fractured Bedrock (P-13 in **Appendix K**).

A technically sound alternative to ERI within the cantonment area and developed off-base locations such as along public rights of way is electromagnetic induction (EMI). The advantages of EMI are that no ground contact is required, and data can be collected in areas paved with asphalt or packed aggregate. In addition, unlike ERI which requires evenly spaced electrodes on a straight transect, EMI can be collected along non-linear pathways and at irregular spacings allowing working in areas with buildings and other structures.

EMI-based resistivity mapping will be conducted using a Geonics EM34-3 terrain conductivity meter (EM34) in accordance with the TGI for Electromagnetic Inductance Surveys (P-20 in **Appendix K**). The EM34, which is a two-person instrument, consists of a transmitter coil physically separated from the receiver coil by a fixed spacing of either 10, 20 or 40 meters. In addition, the coils may be oriented horizontally or vertically, which allows sampling at two depths for each coil separation. There are six possible measurement depths ranging between a minimum of 25 ft and a maximum of 200 ft bgs. To meet the specific objective of delineating the margin of the Pomona Basalt (**Figure 17-1**), data will be collected at transmitter-receiver coil separations and orientations determined by preliminary testing near the locations

of MW-02 and MW-03 which appear to be near, but on opposing sides of the margin of the Pomona Basalt. Each of the six-coil separation/orientation settings will be collected and measurements will be compared with the 2022 ERI results from Transects B and C (ECC/Arcadis 2023a) and the borehole geophysical logs from MW-03 and MW-02. The optimal combination of EM34 coil separations/orientations will be determined and then data collection will proceed. This optimized combination is intended to provide the greatest amount of contrast between the shallow, resistive Pomona Basalt to the north and the more conductive, groundwater saturated sedimentary materials of the Ellensburg Fm to the south.

A series of transects, generally oriented south to north and straddling the currently mapped margin of the Pomona Basalt will be traversed with the EM34. Data will be collected at discrete stations spaced between 100 and 200 ft apart or as site conditions allow. A data-logging unit attached to the receiver coil will record the EM34 readings. The transmitter and receiver operators will each have a digital global positioning system receiver and will record their positions at each station. At each station the operator at the receiver coil will take a reading with the coils held vertically, then horizontally at each of the selected coil separations. The positional and resistivity data will be recorded in the data logger which, at a minimum at the end of each survey day or more frequently as needed, will be transferred to a laptop computer. The downloaded, pre-processed data will be analyzed in profile and map view to identify and interpret the location(s) of the margin of the Pomona Basalt. As needed, in-fill data points and/or additional transects will be visited with the EM34 as needed to confidently define the margin. When the field activities are complete, a contour map of apparent resistivity will be generated for each coil separation/orientation combination. Accurate mapping of the Pomona Basalt margin will inform the CSM and identify appropriate monitoring well locations for subsequent mobilizations.

Borehole Geophysics

Borehole geophysics will be conducted in select bedrock boreholes, prior to monitoring well installation, in order to supplement physical logging of rock chips. Borehole geophysics can provide a detailed measure of the lithology, identify the presence of fractures, faults, geologic contacts, and other preferential flow pathways, and correlate/compare interpretations from the surface geophysical analyses. Boreholes will be digitally surveyed using tools to measure one or more of the following parameters:

- natural gamma radiation (an indication of mineralogy)
- spontaneous potential and single point resistance (an indication of lithology and fracturing)
- 3-arm caliper (an indication of e.g., fracturing, lithologic contacts, bedding planes, weathering)
- Acoustic televiewer which provides a detailed three-dimensional visualization of the borehole
- Heat-pulse flow meter (HPFM) to identify and characterize transmissive zones.

Field work will be conducted in adherence to borehole logging standards found in ASTM D5753-18 (ASTM 2018).

Soil Sampling

Soil samples will be collected via hand auger, direct push technology (DPT) drilling, or sonic drilling methods in accordance with the TGI for PFAS-Specific Drilling and Monitoring Well Installation (P-07 in **Appendix K**).

Locations designated for surface soil sampling only will be advanced by hand auger to the prescribed sample depth, and lithology will be logged based on the hand auger cuttings. Once the approximate lateral extent of PFAS in soil has been approximated via surface sampling, soil borings will be advanced to bedrock using direct push technology or sonic methods to define the vertical distribution of PFAS in soils. Soil cores will be continuously logged, and discrete soil samples will be collected from the following intervals:

- Surface soil: a 6-inch interval beneath the active surface soil horizon/root zone bgs or beneath concrete, asphalt, or disturbed soil, if present (approximately 0.5 to 1 feet bgs)
- Above the bedrock surface
- At nominal 5-ft intervals from ground surface to 20 ft bgs or bedrock. If the bedrock surface is below 20 ft bgs, additional soil samples will be collected at nominal 10-ft intervals until bedrock is encountered, or refusal. Soil sampling intervals will be biased to changes in lithology.

Soil samples will be collected for analysis of PFAS following the soil sampling protocols (P-10 – TGI – PFAS Field Sampling [all media]) detailed in **Appendix K**. The sampling method details equipment requirements, procedures for equipment and container handling before sampling, sampling procedures under various conditions, collecting equipment blank samples (P-12 – TGI – Equipment and Reagent Blank Sample Collection for PFAS Analysis, **Appendix K**), QA/QC sampling requirements, and storing and shipping of samples. In addition to PFAS, up to three samples per characterization area will be analyzed for TOC and grain size to inform the interpretation of PFAS distribution in the subsurface and to update the CSM (**Worksheet #10**). Soil sampling locations may be adjusted in the field to avoid auguring through concrete, asphalt, or other obstructions.

Monitoring Well Installation and Development

Permanent monitoring wells will be installed at select locations to provide sufficient monitoring points to define plume extent, evaluate concentration trends over time, provide data to support FS remedial alternative evaluation and risk assessment evaluations, and monitor plume stability. Additional monitoring wells may be installed after completion of the prescriptive and adaptive phases to ensure optimal placement to meet DQOs. In general, monitoring wells will be biased to the zone of highest flux and located spatially to include the likely source area, the axis and perimeter of the identified groundwater plume, and the base of the plume at depth. Proposed monitoring well locations and screened intervals will be presented in Field Change Form to Regulators and stakeholders for review prior to installation. Final locations will be documented in the meeting minutes and included in the RI report. If existing Installation monitoring wells satisfy data needs, these wells may be included in the RI monitoring network.

Monitoring wells will be drilled using sonic and/or air-rotary drilling methods. and will typically be installed as 2 inch-diameter screened wells (nominal 6-inch borehole). All equipment and materials used in well construction will be PFAS-free, including water that is introduced as part of the drilling process. Monitoring wells will be constructed with 2-inch polyvinyl chloride (PVC) or stainless-steel screens with polyvinyl chloride risers. Screen slot size and filter pack sand size will be determined based on observed grain size distribution. Screen length will be nominally 5, 10, or 20 ft, to be finalized based on observed lithology and PFAS distribution. Filter packs will consist of washed quartz sand, and generally extend from 1 ft below to 2 ft above the well screen unless conditions dictate otherwise. Grain size data collected during the prescriptive phase will be used to correctly size the filter pack material with native lithology. Downhole annular space material will be installed using a tremie pipe. Potential for bridging in the filter pack will be mitigated by performing pre-development before installing bentonite and grout. Pre-development will be performed by gently surging the well to settle the filter pack. Additional filter pack material may be needed if settlement occurs during pre-development. Following pre-development, 2 feet of bentonite will be placed above the filter pack and hydrated using PFAS-free water. The well will then be pressure-grouted via a tremie pipe with bentonite cement grout beginning no more than 4 feet above the top of the aquifer or 4 feet above the top of the screen for wells with the top of screen positioned above the aquifer. Water used to make bentonite cement grout will be clean (i.e., sourced from the same water source as water used for decontamination), and the amount of bentonite will not exceed 2 pounds of bentonite per 94-pound sack of cement. No more than 7 gallons of water will be used for each 94-pound sack of cement. Each well will be completed with a minimum 2-foot by 2-foot and 4-inch-thick concrete pad. Well casings will be equipped with locks and either traffic-rated flush-mount or steel stickup with three protective bollards. The

monitoring wells will be constructed in accordance with Resource Protection Well Construction Standards for Washington State and the PFAS-Specific Drilling and Monitoring Well Installation TGI (P-07, **Appendix K**).

It is anticipated that vertical delineation may require the installation of monitoring wells which may penetrate shallow aquifers and potential underlying confining units in order to sample deeper water bearing zones. Any monitoring wells which may penetrate multiple aquifers will require specific construction techniques (double cased construction) to avoid potential creation of vertical migration pathways. Double cased wells will be installed in two stages:

- The borehole will be advanced to penetrate approximately 2 to 3 feet into the potential confining unit to facilitate installation of a permanent steel casing. The permanent steel casing will be cemented in place using cement grout and tremie pipe effectively isolating the shallower water bearing zone.
- After allowing the cement to cure for a minimum of 24 hours, the borehole will be advanced through the permanent casing to the target depth and a 2 inch-diameter monitoring well will be installed within the borehole using the same materials and method specified for single cased wells.

Following construction, monitoring wells will be developed in accordance with the TGI for Monitoring Well Development (P-06, **Appendix K**) using a combination of surging and pumping techniques. In general, well screens will undergo two 15 to 30-minute cycles of surging, followed by pumping or bailing to remove accumulated sediments. After the wells have been surged twice, the wells will be pumped at a relatively constant rate until indicator parameters (e.g., pH, specific conductance, and temperature) are stable for three consecutive readings, and the extracted water is clear and free of sediment (i.e., with turbidity less than 50 nephelometric turbidity units [NTUs]). Depth to water and total depth measurements will be collected before, during, and after well development. Development water will be containerized for characterization and disposal.

At the completion of each well installation event, groundwater sampling will be performed at each well using low-flow sampling techniques described in the following section. Samples will be collected at least 48 hours after completion of well development. During sampling, the ECC/Arcadis Team will complete gauging of monitoring wells from the top of casing using an electronic water level meter to within 0.01 foot. Groundwater elevations will be summarized in a groundwater elevation summary table.

Hydraulic Testing

Slug tests will be performed on select monitoring wells located in source areas and along plume axis to provide estimates of hydraulic conductivity and support evaluation of contaminant fate and transport. Slug tests will be completed at each of the selected wells in accordance with the TGI for Slug Testing (P-23, **Appendix K**) using either pneumatic methods or a solid slug. Continuous water level measurements will be recorded during each test using electronic data-recording pressure transducers.

Groundwater Sampling

Groundwater sampling is an integral component for PFAS characterization and refinement of the CSM. Groundwater samples may include grab samples associated with either soil borings and/or the drilling of monitoring wells, in addition to synoptic low-flow sampling events to evaluate water quality throughout the installation and track seasonal variability. Grab samples are a useful tool in evaluating where to expand the existing monitoring network. Techniques that will be implemented for both types of samples are described below:

- Grab groundwater samples: During the advancement of soil borings and monitoring well boreholes, grab groundwater samples may be collected at locations where the perched water is encountered or

where different zones of varying permeability are encountered within the same aquifer. Grab groundwater samples will be collected in accordance with the TGI for PFAS Sampling Procedures (P-11, **Appendix K**) and submitted for laboratory analysis of PFAS.

- Low-flow monitoring well sampling: Monitoring wells will be sampled using low-flow sampling techniques. The intake of the submersible pump will be placed in the approximate center of the saturated screen during purging and sampling. Water quality field parameters (temperature, pH, conductivity, DO, turbidity, and ORP) will be recorded during purging and parameters will be allowed to stabilize prior to sample collection, in accordance with the TGI for PFAS Sampling Procedures and Low-Flow Groundwater Purging for Monitoring Wells (P-11, **Appendix K**). Groundwater samples will be analyzed for PFAS as defined in **Worksheet #15** of this UFP-QAPP.

The sampling methods detailed in **Appendix K** establish equipment requirements, procedures for handling equipment and containers before sampling, sampling procedures under various conditions, collecting equipment blank samples (P-13 – TGI – Equipment and Reagent Blank Sample Collection for PFAS Analysis, **Appendix K**), QA/QC sample requirements, and storing and shipping of samples to ensure that sample contamination does not occur during collection, transport, and analysis.

Surface Water and Sediment Sampling

Surface water samples will be collected using direct fill methods in accordance with the surface water sampling protocol (P-10 – TGI – PFAS Field Sampling [all media]; P-15 – TGI – Sediment, Surface Water, and Stormwater Sample Collection for PFAS Analysis) detailed in **Appendix K**. Grab surface water samples will be collected from midstream and before the collection of collocated sediment samples, to minimize turbidity. The sampling method establishes equipment requirements, procedures for handling equipment and containers before sampling, sampling procedures under various conditions, collection of QA/QC samples (P-13 – TGI – Equipment and Reagent Blank Sample Collection for PFAS Analysis, **Appendix K**), and storing and shipping samples to ensure that sample contamination does not occur during collection, transport, and analysis. All surface water samples will be analyzed for PFAS by Method 1633 (**Worksheet #15**).

Sediment samples will be collected following the sediment sampling protocol (P-10 – TGI – PFAS Substances Field Sampling [all media]; P-15 – TGI – Sediment, Surface Water, and Stormwater Sample Collection for PFAS Analysis) detailed in **Appendix K**. Samples will be collected from the upper 6 inches of the stream or channel bed, using a dedicated, disposable trowel/shovel. The sample will be decanted (if needed) before bottling for laboratory analysis. The sampling method establishes equipment requirements, procedures for equipment and containers before sampling, sampling procedures under various conditions, collection of QA/QC samples (P-13 – TGI – Equipment and Reagent Blank Sample Collection for PFAS Analysis, **Appendix K**), and storing and shipping samples to ensure that sample contamination does not occur during collection, transport, and analysis. All sediment samples will be analyzed for PFAS by Method 1633 (**Worksheet 15**).

Surveying

Monitoring wells will be professionally surveyed to include northing, easting, ground surface elevation, and top of casing elevation using World Geodetic System (WGS) 1984, Universal Transverse Mercator (UTM) Zone 10 North in meters. The horizontal location of each monitoring well will be surveyed to an accuracy of 0.01 foot using a Washington licensed surveyor and will include top of casing and ground surface elevation measurements. Mapping- or survey-grade global positioning systems (GPS) or comparable traditional survey methods will be used to collect geospatial data. Other sampling locations (i.e., soil boring locations) will be recorded with a handheld GPS capable of achieving 0.1 foot of horizontal accuracy.

IDW Management

IDW generated during the RI may include soil and rock cuttings, groundwater sampling purge water, decontamination fluids, hydraulic testing water, and well abandonment materials. IDW will be properly containerized and staged at an approved location at YTC for later waste characterization sampling and off-post disposal at an approved, permitted facility (P-16 – TGI – Investigation-Derived Waste Handling and Storage, **Appendix K**). IDW storage containers may consist of a combination of steel or poly 55-gallon drums, 275 or 330-gallon high-density polyethylene totes, bulk water tanks, and lined roll-off dumpsters, as needed. All waste will be labelled with the description of medium, origin of medium, date the material was placed into the container, contact information, and a statement indicating that the contents are on hold pending analysis. ECC will coordinate with the YTC POCs to arrange for signatures of waste manifests and for compliance with facility generator requirements.

WORKSHEET #18: SAMPLING LOCATIONS AND METHODS

**(UFP-QAPP Manual Section 3.1.1 and 3.1.2)
 (USEPA 2106-G-05 Section 2.3.1 and 2.3.2)**

The proposed sampling analytes and parameters for each medium are summarized in **Table 18-1**. Sampling locations for the RI Phase I sampling event are depicted on **Figures 17-2 through 17-5**, though exact locations and samples are subject to change based on field conditions and observations. Sample nomenclature for subsequent phases of the RI will resume following the Phase I event; however, the number of samples may be adjusted based on the prescriptive phase analytical results. Therefore, the samples are not shown on figures at this time but will be presented in each of the Field Change Form. Parent sample nomenclature will follow the format presented below:

- Soil samples: YTC-SO- [Boring No.]-[Upper Depth]-[Lower Depth]
- Grab groundwater samples associated with soil borings: YTC-GW-[Boring No.]-[MMDDYY]
- Low-flow groundwater monitoring well samples: YTC-[Well ID]-[MMDDYY]
- Grab groundwater samples: YTC-GGW-[Well ID]-[MMDDYY]
- Surface water samples: YTC-SW [Sample No.]-[MMDDYY]
- Sediment samples: YTC-SD [Sample No.]-[Upper Depth]-[Lower Depth]

QC Samples:

- Field Duplicate samples (FD): YTC- [Sample Media Abbreviation]-FD-[Duplicate No.]-[MMDDYY]
- Blank samples: YTC-[FB]-[QC sample type number]-[MMDDYY]

The group of PFAS (including PFOS, PFOA, and PFBS) identified for analysis for groundwater, surface water, soil, and sediment samples in the table below is summarized for all media in **Worksheet #15**. **Worksheet #17** describes the rationale for the various sampling locations and media. Field activities and sampling procedures will be conducted in accordance with the TGI and SOP documents in **Appendix K**. The frequency requirements for QA/QC samples for each medium is noted in **Worksheet #20**; however, the final number and identifications of QA/QC samples listed in the table below are TBD based on progression of daily field activities.

Table 18-1: Sample Summary

| Sample Location ¹ | Matrix | Depth Interval | Sample ID | Sample Type | # Samples | Analytes ² | QC Samples | Total Samples |
|------------------------------|----------------|----------------|-----------|-------------|-----------|--|------------|---------------|
| TBD | Sediment | TBD | TBD | Grab | TBD | PFAS, TOC | TBD | TBD |
| | Soil | | | | | PFAS, TOC, grain size (and total solids) | | |
| | Surface Water | | | | | PFAS, field parameters | | |
| | Groundwater | | | | | | | |
| | Drinking Water | | | | | | | |

Notes:

1 – Sampling locations subject to change based on conditions encountered in the field and consultations with Regulators and stakeholders.

2 – See **Worksheet #15** for individual compounds within the PFAS Method 1633 group. Field parameters include temperature, pH, conductivity, dissolved oxygen (DO), turbidity, and oxidation-reduction potential (ORP).

QC – Quality Control

TOC – total organic carbon

ID – identification

PFAS – per- and polyfluoroalkyl substances

TBD – To be determined

WORKSHEET #19 & 30: SAMPLE CONTAINERS, PRESERVATION, AND HOLD TIMES

This worksheet serves as a reference for field personnel. It is also an aid to completing the chain-of-custody (CoC) form and shipping documents.

Primary Laboratory:

Advanced Environmental Laboratories, Inc.
6681 Southpoint Parkway
Jacksonville, FL 32216
Sample Receipt: Craig Myers, cmyers@aellab.com, (904) 363-9350

List any required accreditations/certifications:

DoD ELAP PJLA 104509 L19-470-R2
Washington State Environmental Laboratory Accreditation (Laboratory ID C1083)

Sample Delivery Method: Federal Express Overnight

Data Package Turnaround: 15 business days

| Analyte Group | Matrix | Preparation Method | Analytical Method | Accreditation Expiration Date | Containers (Number, Size and Type) | Preservation | Preparation Holding Time | Analysis Holding Time |
|---------------|----------------------------|--------------------|---|-------------------------------|--|--|--------------------------|------------------------------|
| TOC | Groundwater, Surface Water | N/A | SM5310C | DoD August 4, 2023 | TOC: 3 x pre-preserved phosphoric acid amber 40 mL VOA vials | TOC: Cool to > 0 to < 6 °C, phosphoric acid preservation to pH < 2 | N/A | 28 days from collection |
| TOC | Soil | | USEPA 9060A | | Filled 4-oz glass jar | cooled to > 0 to < 6 °C | N/A | 28 days from collection |
| pH | Groundwater & Water | | USEPA 9040 | | Filled 8oz glass or plastic jar | | N/A | Within 24 hours of receipt |
| pH | Soil | | USEPA 9045D | | Filled 8oz glass jar | | N/A | Within 24 hours from receipt |
| PFAS | Groundwater & Water | | USEPA 1633 Draft and QSM 5.4 Table B-24 | | 2 - 500mL wide mouth HDPE (3x for designated MS/MSD sample) | None, Cool to ≤ 6°C | 28 days | 28 days |
| PFAS | Soil | | ASTM D422 | 1-4oz HDPE | 90 days | | 28 days | |
| Grain Size | | | | | 16 oz glass jar | | N/A | N/A |

Notes:

°C – degrees Celcius

> – greater than

< – less than

≤ – less than or equal to

ASTM – American Society for Testing and Materials

DoD – Department of Defense

HDPE – high-density polyethylene

mL - milliliters

MS – matrix spike

MSD – matrix spike duplicate

N/A – not applicable

oz - ounce

PFAS – per- and polyfluoroalkyl substances

QSM – Quality Systems Manual

TOC – total organic carbon

USEPA – United States Environmental Protection Agency

VOA – volatile organic analysis

WORKSHEET #20: FIELD QC SUMMARY

**(UFP-QAPP Section 3.1.1 and 3.1.2)
 (USEPA 2106-G-05 Section 2.3.5)**

The Field QC samples are listed below as frequency per number of field samples.

| Matrix | Analyte/ Analytical Group | Field Samples | Field Duplicates | MS | MSD | Field Blanks | Equipment Blanks | Total # analyses for prescriptive sampling |
|--------------------------------|---------------------------------|------------------|---------------------|----------|----------|-----------------------|--|---|
| Soil/Sediment | PFAS | TBD | 1 per 20 | 1 per 20 | 1 per 20 | 1 per 20 (aqueous) | 1 per piece of non-dedicated equipment per sampling event | TBD |
| | TOC | TBD | 1 per 20 | 1 per 20 | 1 per 20 | N/A | N/A | TBD |
| | pH | TBD | N/A | N/A | N/A | N/A | N/A | TBD |
| | Grain Size | TBD | N/A | N/A | N/A | N/A | N/A | TBD |
| Groundwater / Surface Water | PFAS | TBD | 1 per 20 | 1 per 20 | 1 per 20 | 1 per 20 (aqueous) | 1 per piece of non-dedicated equipment per sampling event | TBD |
| IDW | TBD | TBD | N/A | N/A | N/A | N/A | TBD | TBD |

Notes:

IDW – investigation derived waste

MS – matrix spike

MSD – matrix spike duplicate

N/A – not applicable

PFAS – per- and polyfluoroalkyl substances

TBD – to be determined

TOC – total organic carbon

WORKSHEET #21: FIELD SOPS
(UFP-QAPP Manual Section 3.1.2)
(USEPA 2106-G-05 Section 2.3.2)

The Field SOPs/TGIs listed below may not apply to all AOPIs. SOPs/TGIs are included in **Appendix K**.

| Procedure # or Reference ¹ | Title, Revision, Date, and URL (if available) | Originating Organization | Procedure Option or Equipment Type (if procedure provides different options) ² | Modified for Project? Y/N |
|---------------------------------------|--|--------------------------|---|---------------------------|
| ENV-610 | ENV-610 – Field QC Samples; 25 June 2008. | ECC | Sample collection | N |
| P-01 | QP – Field Activities Documentation, Rev. C, November 2016 | Arcadis | Applies to field personnel. | N |
| P-02 | TGI – Sample Chain of Custody, Rev. 3, March 2022 | Arcadis | Applies to field personnel with 40-hour HAZWOPER and Department of Transportation Hazardous Materials #1 training. | N |
| P-03 | QP – Calibration and Control of measuring and test equipment, Rev. C, November 2016 | Arcadis | Applies to field personnel using equipment that is capable of calibration. | N |
| P-04 | QP - Field Sampling, Measurement, and Observation, Rev. D, October 2017 | Arcadis | Applies to field personnel completing field sampling, measurement, and observations. | N |
| P-05 | TGI - Soil Description, Rev. 2, 16 Feb 2018 | Arcadis | Applies to field personnel conducting soil logging. | N |
| P-06 | TGI- Monitoring Well Development, Rev. 0, April 2017 | Arcadis | Applies to field personnel developing monitoring wells. See TGI for specific equipment needs. | N |
| P-07 | TGI – PFAS-Specific Drilling and Monitoring Well Installation, Rev. 0, October 2018 | Arcadis | Applies to field personnel conducting oversight of monitoring well installation. See TGI for specific equipment needs. | N |
| P-08 | TGI- Monitoring Well Inspection Assessment, Rev. 1, June 2022 | Arcadis | See TGI for specific equipment needs. | N |
| P-09 | TGI – Groundwater and Soil Sampling Equipment Decontamination, Rev. 01, 8 May 2020 | Arcadis | Applies to soil sampling tools; groundwater, sediment, and surface water sampling devices; water testing instruments; downhole instruments; and other activity-specific sampling equipment. | N |
| P-10 | TGI - PFAS Field Sampling (all media) Guidance, Rev. 9, 22 October 2021 | Arcadis | Applies to field personnel collecting environmental samples for PFAS analysis. See TGI for specific equipment needs. | N |
| P-11 | TGI – PFAS Sampling Procedures and Low-Flow Groundwater Purging for Monitoring Wells, Rev. 0, 19 June 2018 | Arcadis | Applies to low-flow sampling for PFAS. | N |
| P-12 | TGI – Equipment and Reagent Blank Sample Collection for PFAS Analysis, Rev. 0, October 2018 | Arcadis | Applies to field personnel completing field sampling. See TGI for specific equipment needs. | N |
| P-13 | TGI – Geophysical Investigation TGI for Investigation Fractured Bedrock, Rev. 0, 25 October 2022 | Arcadis | Applies to field personnel completing geophysical data collection. See TGI for specific equipment needs. | N |

| Procedure # or Reference ¹ | Title, Revision, Date, and URL (if available) | Originating Organization | Procedure Option or Equipment Type (if procedure provides different options) ² | Modified for Project? Y/N |
|---------------------------------------|---|--------------------------|---|---------------------------|
| P-14 | TGI – Sediment, Surface Water, and Stormwater Sample Collection for PFAS Analysis, Rev. 2, 27 March 2020 | Arcadis | Applies to field personnel collecting sediment, surface water, and stormwater samples. See TGI for specific equipment needs. | N |
| P-15 | TGI – IDW Handling and Storage, Rev. 01, 15 May 2020 | Arcadis | See TGI for specific equipment needs. | N |
| P-16 | TGI – In-Situ and Ex-Situ Water Quality Parameters – Surface Water and Groundwater | Arcadis | Applies to field personnel using equipment to collected field parameters for surface water and groundwater samples. See TGI for specific equipment needs. | N |
| P-17 | TGI – Soil Drilling and Sampling Collection, Rev. 02, 08 April 2022 | Arcadis | Applies to field personnel using equipment to collect surface and subsurface soil samples. | N |
| P-18 | TGI – Residential Drinking Water Well Sampling Collection, Rev. 01, 08 April 2022 | Arcadis | Applies to field personnel sampling drinking water wells. | N |
| P-19 | TGI – PFAS Potable Water Sampling Guidance | Arcadis | Applies to field personnel sampling potable wells for PFAS. | N |
| P-20 | TGI – Electromagnetic Induction Surveys with Geonics Terrain Conductivity Meters, Rev. 6, 8 February 2023 | Arcadis | Applies to field personnel collecting geophysical data using electromagnetic induction. | N |
| P-21 | EQASOP – Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells, Rev. 04, 19 September 2017 | USEPA | Applies to low-flow sampling for PFAS. | N |
| P-22 | TGI – Manual Water-Level and NAPL Monitoring, Rev. 04, 01 March 2023 | Arcadis | Applies to existing monitoring wells and the presence of NAPL. | N |
| P-23 | TGI – General Slug Testing, Rev. 7, 1 March 2023 | Arcadis | Applies to field personnel conducting slug tests. | N |
| P-24 | TGI – Collection and Logging of Bedrock Chips, Rev. 0, 4 November 2022 | Arcadis | Applies to field personnel logging drill cuttings produced using rotary drilling methods. | N |

Notes:

- Copies of the field SOPs/TGIs are included in **Appendix K**.
- For all TGIs pertaining to the collection of samples for PFAS analysis, there is concern that sampling for PFAS using sampling equipment manufactured from fluoropolymers could result in sample contamination. The materials of construction of all downhole and surface sampling and monitoring equipment — including pumps, packers, transducers, tubing, liners, valves, and wiring - should be free from polytetrafluoroethylene or ethylene tetrafluoroethylene to the maximum extent practicable. In addition, well drilling procedures and completion materials should avoid the use of fluorocarbon-based lubricants; O-rings; and pipe thread pastes, tapes, and sealants. If possible, a confirmation letter with analytical testing results should be obtained from a manufacturer or service provider certifying that the equipment and supplies are PFAS-free.

Arcadis – Arcadis U.S., Inc.

ECC – Environmental Chemical Corporation

EQASOP - USEPA Quality Assurance Standard Operating Procedure

HAZWOPER – Hazardous Waste Operations and Emergency Response

IDW – investigation-derived waste

NAPL – non-aqueous phase liquid

PFAS – per- and poly fluoroalkyl substances

QP – Quality Procedure

Rev. – Revision

SOP – Standard Operating Procedure

TGI – Technical Guidance Instructions

USEPA – United States Environmental Protection Agency

WORKSHEET #22: FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION

**(UFP-QAPP Manual Section 3.1.2.4)
 (USEPA 2106-G-05 Section 2.3.6)**

The equipment listed below may not apply to all AOIs.

| Instrument or Equipment | Description | Field Calibration Procedure | Performance Criteria | Responsible Personnel |
|--|--|--|---|------------------------------|
| Water Quality Meter – YSI® 6-Series Multi-Parameter Instrument or Equivalent | Multi-parameter tool designed for field use with battery operation. Ranges: 0 to 14 pH; -999 to +999 millivolt ORP; -5 to 50 °C Temperature; 0 to 50 micrograms per Liter DO; 0 to 100 mS/cm Conductivity 0 to 1,000 NTU Turbidity | The unit is factory calibrated. Unit responsiveness will be checked before use each day with appropriate standards provided by the supplier. Unit responsiveness is checked against the solution standards provided by each manufacturer. | ± 10% of included standard solutions with meter | Sample Collection Personnel |
| Turbidimeter – Hach® 2100P or Equivalent | Designed for field use with battery operation. Range: 0 to 1,000 NTU. | Each day before use, the turbidimeter is calibrated against the standard solutions provided by each manufacturer. | ±10% of included standard solutions with turbidimeter | Sample Collection Personnel |
| 4-Gas Meter – Multi-RAE® or Equivalent | Designed for field use with battery operation. Ranges: 0 to 100% LEL; 0 to 100 ppm H ₂ S; 0 to 30% (by vol.) O ₂ ; 0 to 2,000 ppm CO; 0.1 to 5,000 ppm VOC | Each day before use, the four-Gas Meter is calibrated against clean (ambient) air and supplier-provided standard (mixed gas cannister). | ± 10% of included standard gas value | Sample Collection Personnel |

Notes:

- ± – plus or minus
- % – percent
- CO – carbon monoxide
- DO – dissolved oxygen
- H₂S – hydrogen sulfide
- LEL – lower explosive limit
- mS/cm – milliSiemens per centimeter
- NTU – nephelometric turbidity units
- O₂ – oxygen
- ORP – oxidation reduction potential
- ppm – parts per million
- VOC – volatile organic compound

WORKSHEET #23: ANALYTICAL SOP REFERENCES

**(UFP-QAPP Manual Section 3.2.1)
 (USEPA 2106-G-05 Section 2.3.4)**

| Analyte Group | Method | AEL SOP # | Title, Revision Date, and/or Number | Definitive or Screening Data | Matrix and Analytical Group | Instrument | Organization Performing Analysis | Modified for Project Work (Y/N) |
|-----------------|---|--------------|--|------------------------------|-----------------------------|--------------|----------------------------------|---------------------------------|
| Samples | Sample Receipt, handling, storage, and Log In | Admin-005 | Sample Receipt, handling, storage, and Log-in Rev.09, Eff. 2022-07-27 | Definitive | Samples | NA | AEL | N |
| Disposal | Sample Disposal | Admin-018 | Waste Disposal and Pollution Prevention Admin-018 Rev.10 Eff 2020-10-09 | NA | Samples | NA | AEL | N |
| Quality Systems | Quality Systems | AEL QSM 10.4 | Quality Manual, Rev. 10.4, Eff 2022-03-31 | Definitive | Quality Systems | NA | AEL | N |
| Wet Chemistry | SM9040C | WC-002 | USEPA Methods 150.1, 9040c, & SM 4500H+B Determination of pH (and Corrosivity) Electrometrically, Rev. 16, Eff: 2022-03-11 | Definitive | Aqueous/pH | pH Meter | AEL | N |
| | USEPA 9060A | WC-021 | Method 9060A: Determination of TOC in Solid and Chemical Materials. Rev. 07, Eff. 2022-09-14 | Definitive | Solid/TOC | TOC Analyzer | AEL | N |
| | SM5310C | WC-022 | Method USEPA 415.1 (1974)/SM5310B/C: Determination of TOC, DOC, and TIC. Rev. 11, Eff. 2022-06-22 | Definitive | Aqueous/TOC | TOC Analyzer | AEL | N |
| | USEPA 9045D | WC-057 | Method 9045D Determination of pH Electrometrically in Soil and Waste, Rev 05, Eff: 2022-03-10 | Definitive | Solid/ pH | pH Meter | AEL | N |
| SVOCs | USEPA 1633 Draft and QSM 5.4 Tables B-24 | SVOC-043 | Method USEPA Draft Method 1633 and PFAS Compliant with Table B-24, DoD, QSM, Version 5.4 Requirements, Rev 01, Eff: 2022-08-26 | Definitive | Solid/ Aqueous/ PFAS | LC-MS/MS | AEL | N |

Notes:

AEL – Advanced Environmental Laboratories, Inc.
 DOC – dissolved organic carbon
 DoD - Department of Defense
 Eff. – Effective
 LC-MS/MS – Liquid chromatography with tandem mass spectrometry

NA – not applicable
 PFAS – per- and polyfluoroalkyl substances
 QSM – Quality Systems Manual
 Rev – Revision
 SOP – Standard Operating Procedure
 SVOC – semivolatile organic compound

TIC – total inorganic carbon
 TOC – total organic carbon
 USEPA – United States Environmental Protection Agency
 WC – wet chemistry

WORKSHEET #24: ANALYTICAL INSTRUMENT CALIBRATION

**(UFP-QAPP Manual Section 3.2.2)
 (USEPA 2106-G-05 Section 2.3.6)**

| Instrument | Calibration Procedure | Calibration Range | Frequency of Calibration | Acceptance Criteria | CA | Person Responsible for CA | SOP Reference |
|--------------|-----------------------|--|--|---|--|---------------------------|--|
| TOC Analyzer | Carryover Check | N/A | For SM5310C: At instrument set-up a blank is analyzed after the UQL (25.0mg/L), prior to the ICAL and before sample analysis | TOC concentration <1/2 LOQ | Correct problem then repeat Carry Over Check | Analyst | SVOC-022 Sections 14,16 |
| | ICAL | For SM 5310C: Prepare calibration standards at a maximum of 8 levels: 0 -25 mg/L. For USEPA 9060A: Prepare calibration standards at a maximum of 6 levels: 0.5 to 20 mg. External calibration is used. | At instrument set-up and after ICV or CCV failure, prior to sample analysis, every six months or as needed | Linear least squares regression for each analyte $R \geq 0.995$ | Correct problem then repeat ICAL | | For SM 5310C WC-022 Sections 13, 16. For USEPA 9060A WC-023 Sections 13, 16 |
| | ICV | At concentration level ± 25 -50% of curve range | Once after ICAL | TOC within $\pm 10\%$ of true value | Correct problem. Rerun ICV. If fails, repeat ICAL | | |
| | ICB | N/A | Once after ICV | TOC concentration < 1/2 LOQ | Correct problem then repeat ICB. If fails, perform instrument maintenance and repeat ICAL. | | |

| Instrument | Calibration Procedure | Calibration Range | Frequency of Calibration | Acceptance Criteria | CA | Person Responsible for CA | SOP Reference |
|--------------|-----------------------|--|---|---|--|---------------------------|--|
| TOC Analyzer | CCV | For SM 5310C: Initial CCV is low concentration (0.1 mg/L), then the CCVs are varied between medium (10 mg/L) and high concentration (25 mg/L). For USEPA 9060A: At concentration level \pm 25-50% of curve range. | Daily before sample analysis, every 10 field samples, and close of analysis | For SM 5310C: All target analytes within \pm 50% of true value for the low concentration CCV, and \pm 15% of true value for the mid and high concentration CCV. For EPA 9060A: All target analytes within \pm 10% of true value. | Immediately analyze two additional CCVs. If both pass, samples can be reported. If either fails, repair problem, rerun samples with new passing CCV. May require new ICAL. If reanalysis not possible, data must be qualified with case narration. | Analyst | For SM 5310C WC-022 Sections 12, 13, 16. For USEPA 9060A WC-023 Sections 12, 13, 16 |
| | CCB | N/A | Analyzed after the CCV every 10 field samples, and close of analysis | TOC concentration $< 1/2$ LOQ | If fails, repair problem, rerun samples with new passing CCV and CCB set. May require new ICAL. If reanalysis not possible, data must be qualified with case narration. | | |
| pH meter | ICAL | 4.00, 7.00, 10.00 SUs | At instrument set-up and after ICV or CCV failure, prior to sample analysis | All levels re-analyzed after calibration, and must be within \pm 0.05 SU of true value | Correct problem then repeat ICAL | Analyst | WC-002, WC-057 |
| | ICV | 7.0 pH SUs | Once after ICAL | Within \pm 0.05 SU of true value | Correct problem. Rerun ICV. If fails, repeat ICAL | | |
| | CCV | 7.0 pH SUs | Daily before sample analysis, and at the end of analysis. | Within \pm 0.05 SU of true value | Correct problem. Rerun CCV. If fails, repeat ICAL | | |

| Instrument | Calibration Procedure | Calibration Range | Frequency of Calibration | Acceptance Criteria | CA | Person Responsible for CA | SOP Reference |
|------------|------------------------------|---|--|---|--|---------------------------|----------------------------------|
| LC-MS/MS | Mass Calibration | N/A | Instrument must have a valid mass calibration prior to any sample analysis. Mass calibration is verified after each mass calibration, prior to ICAL. | Calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. Mass calibration range must bracket the ion masses of interest. The most recent mass calibration must be used for every acquisition in an analytical run. Mass calibration must be verified to be ± 0.5 amu of the true value, by acquiring a full scan continuum mass spectrum of a PFAS stock standard. | If the mass calibration fails, then recalibrate. If it fails again, consult manufacturer instructions on corrective maintenance. | Analyst | SVOC-043 Sections 12, 13, and 18 |
| | Bile Salt Interference Check | TDCA, TCDCa and TUDCA at 1.0 $\mu\text{g/mL}$ | After the initial calibration as a check on the chromatographic conditions, and at the beginning of every DoD sequence. | Each bile salt must not elute within 1 minute of all PFOS isomers. | If an interference is present, the chromatographic conditions must be modified to eliminate the interference from the bile salts (e.g., changing the retention time of the bile salts such that they fall outside the retention time window for any of the linear or branched PFOS isomers in the standard by at least one minute), and the ICAL repeated. | Analyst | |

| Instrument | Calibration Procedure | Calibration Range | Frequency of Calibration | Acceptance Criteria | CA | Person Responsible for CA | SOP Reference |
|------------|---|------------------------------------|---|--|-----|---------------------------|----------------------------------|
| LC-MS/MS | Calibration, Calibration Verification and Spiking Standards | Mid- to high- level concentration. | All analytes. Note: Standards containing both branched and linear isomers are to be used during method validation and when re-establishing retention times, to ensure the total response is quantitated for that analyte. Technical grade standards cannot be used for quantitative analysis. Identify the retention times of the branched isomers of PFOA present in the technical-grade PFOA standard. When PFOA is chromatographed on a reversed-phase column, the branched isomers elute prior to the linear isomer. Repeat the procedure in this section for PFHxS, PFOS, N-Et-FOSA, N-Me-FOSE, and N-Et-FOSE. | Standards containing both branched and linear isomers must be used when commercially available. PFAS method analytes may consist of both branched and linear isomers, but quantitative standards that contain the linear and branched isomers do not exist for all method analytes. For PFAS that do not have a quantitative branched and linear standard, identify the branched isomers by analyzing a qualitative standard that includes both linear and branched isomers and determine retention times, transitions and transition ion ratios. Quantitate samples by integrating the total response (i.e., accounting for peaks that are identified as linear and branched isomers) and relying on the initial calibration that uses the linear isomer quantitative standard. | N/A | Analyst | SVOC-043 Sections 12, 13, and 18 |
| | Mass Spectral Acquisition Rate | | Each analyte, EIS analyte and NIS | A minimum of 10 spectra scans are acquired across each chromatographic peak. | | | SVOC-043 Section 24 |

| Instrument | Calibration Procedure | Calibration Range | Frequency of Calibration | Acceptance Criteria | CA | Person Responsible for CA | SOP Reference |
|------------|---|--|--|--|--|---------------------------|----------------------------------|
| LC-MS/MS | Ion Transitions (Precursor⇒ Product) | NA | Every field sample, standard, blank, and QC sample | In order to avoid biasing results high due to known interferences for some transitions, the transitions presented in SVOC-043 Table 3 are used for quantification. If these transitions are not used, the reason must be technically justified and documented (e.g., alternate transition was used due to observed interferences). | NA | Analyst | SVOC-043 Sections 12, 13, and 24 |
| LC-MS/MS | ICAL typically nine standards; Calibration can be linear (minimum of 6 standards) or quadratic (minimum of 7 standards); weighting is allowed. Forcing the calibration through zero is mandatory. | ICAL 0.1 to 10.0 ng/mL ICV (varied ng/mL) | At instrument set-up and after ICV or CCV failure, prior to sample analysis. | Analytes must be within 70-130% of their true value for each calibration standard. ICAL must meet one of the two options below: Option 1: The %RSD of the RFs for all analytes must be ≤ 20%. Option 2: Linear or non-linear calibrations must have $r^2 \geq 0.99$ for each analyte. Commercial PFAS standards available as salts are acceptable providing the measured mass is corrected to the neutral acid concentration. Results shall be reported as the neutral acid with appropriate CAS number. If a labelled analog is not commercially available, the EIS analyte with the closest retention time or chemical similarity to the analyte must be used for quantitation. (IS Quantitation). | Perform maintenance, if necessary, repeat calibration if criterion is not met. No samples shall be analyzed until ICAL has passed. | Analyst | SVOC-043 Sections 12, 13, and 18 |

| Instrument | Calibration Procedure | Calibration Range | Frequency of Calibration | Acceptance Criteria | CA | Person Responsible for CA | SOP Reference |
|------------|-----------------------|-------------------|---|---|--|---------------------------|----------------------------------|
| LC-MS/MS | IB | N/A | Every analytical sequence, prior to sample analysis, after the high standard of the ICAL, after every CCV and after any field sample with an analyte detection above the range of the calibration curve (if observed in time to edit/update the analytical sequence) the laboratory must analyze an IB. | Concentration of each analyte must be $\leq \frac{1}{2}$ the LOQ. IB must contain EIS and NIS to enable quantitation of contamination | If acceptance criteria are not met after the highest calibration standard, calibration must be performed using a lower concentration for the highest standard until acceptance criteria is met. If sample concentrations exceed the highest allowed standard and the sample(s) following exceed this acceptance criteria ($> \frac{1}{2}$ LOQ), they must be reanalyzed | Analyst | SVOC-043 Sections 12, 13, and 18 |
| | ICV | Varied | Once after each ICAL, analysis of a second source standard prior to sample analysis | Analyte concentrations must be within $\pm 30\%$ of their true value | Correct problem and verify second source standard; rerun second source verification. If that fails, correct problem and repeat ICAL. | | |
| | NIS | Varied | NIS are added to all standards, quality control samples, and sample extracts. | Area recoveries must be greater than 30% of the average of the ICAL. NIS areas corrected for the dilution factor must be greater than 30% of the average area of the calibration standards in diluted samples when additional NIS was not added post dilution of the extract. | Repeat the analysis using a fresh aliquot of the extract. If failure does not confirm, report the second analysis. If the failure confirms, examine the project-specific requirements. Contact the client as to additional measures to be taken. | | |

| Instrument | Calibration Procedure | Calibration Range | Frequency of Calibration | Acceptance Criteria | CA | Person Responsible for CA | SOP Reference |
|------------|-----------------------|---|--|---|--|---------------------------|----------------------------------|
| LC-MS/MS | EIS | Varied | Isotope dilution analogues are added to all samples and quality control samples prior to extraction. | EIS recoveries must be within 20-150% until in-house limits can be created. In-house limits cannot be lower than 20%. | Repeat the analysis using a fresh aliquot of the extract. If failure does not confirm, report the second analysis. If the failure confirms, follow the requirements listed in USEPA Draft Method 1633, Section 15.3.2. If EIS recoveries still fall outside of the acceptance range, the client must be contacted for additional measures to be taken. | Analyst | SVOC-043 Sections 12, 13, and 18 |
| | ISC | Varied. Concentrations of analytes at, or below, LOQ. | Daily prior to analysis and at least once every 12 hours | Analyte concentrations must be at LOQ; concentrations must be within $\pm 30\%$ of their true values | Correct problem, rerun ISC. If problem persists, repeat ICAL. | | |
| | CCV | Varied | Daily prior to sample analysis (ISC); after every 10 field samples; at end of analytical sequence | All analytes must be within $\pm 30\%$ of their true value | Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CCVs cannot be run, perform CAs, and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all samples since the last acceptable CCV. | | |

Notes:

< – less than

\leq – less than or equal to

| | |
|--|---|
| > – greater than | N-Et-FOSA – N-ethylperfluorooctansulfonamid |
| ≥ – greater than or equal to | N-Et-FOSE – N-ethylperfluorooctane sulfonamidoethanol |
| ± – plus or minus | N-Me-FOSE – N-methylperfluorooctanesulfonamidoethanol |
| % – percent | ng/mL – nanograms per milliliter |
| amu – atomic mass unit | NIS - Non-extracted Internal Standards |
| CA – corrective action | PFAS – per- and polyfluoroalkyl substances |
| CAS – Chemical Abstract Service | PFHxS – perfluorohexanesulfonic acid |
| CCB – continuing calibration blank | PFOA – perfluorooctanoic acid |
| CCV – continuing calibration verification | PFOS – perfluorooctane sulfonate |
| EIS - extracted internal standards | r ² – correlation coefficient |
| LC-MS/MS – liquid chromatography with tandem mass spectrometry | RF – response factor |
| LOQ – limit of quantitation | RPD – Relative Standard Deviation |
| IB – instrument blank | SOP – Standard Operating Procedure |
| ICAL – initial calibration | SVOC – semivolatile organic compound |
| ICB – initial calibration blank | SU – standard units |
| ICV – Initial calibration verification | TDCA – taurodeoxycholic acid |
| IS – Internal Standard | TCDCa – sodium taurochenodeoxycholate |
| ISC – Instrument Sensitivity Check | TOC – total organic carbon |
| mg – milligrams | TUDCA – tauroursodeoxycholic acid |
| mg/L – milligrams per liter | USEPA – United States Environmental Protection Agency |
| N/A – non applicable | WC – wet chemistry |

WORKSHEET #25: ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION

**(UFP-QAPP Manual Section 3.2.3)
 (USEPA 2106-G-05 Section 2.3.6)**

| Instrument or Equipment | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | CA | Title/Position Responsible for CA | SOP Reference |
|-------------------------|--|------------------|--|---------------------------|--|---|-----------------------------------|-------------------|
| TOC Analyzer | Gas service Halide scrubber Heater element Dryer tube Permeation tube Reagent reservoirs Syringe pump Reaction Vessel | ICAL and CCVs | <ul style="list-style-type: none"> Gas service replace as needed Replace Halide scrubber every 3,000 samples (or as needed) Replace Heater element as needed Replace Dryer tube as needed Replace Permeation tube every 24,000 samples (or as needed) Refill Reagent reservoirs as needed Replace Syringe pump as needed Replace Reaction Vessel as needed | As Listed with Inspection | MBs and IBs < DL, ICAL, ICV, CCV, peak shape, & LCS, as seen on Worksheet #28 | Normal replacements | Analyst. | WC-022, WC-023 |
| pH meter | Analytical Performance | ICAL and CCVs | <ul style="list-style-type: none"> Checked daily Inspect probe for cleanliness, check tubing, check flow of reagents, calibrate | As Listed with Inspection | QC passing criteria | Perform necessary equipment maintenance and check calibration standards | | WC-002 and WC-057 |

| Instrument or Equipment | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | CA | Title/Position Responsible for CA | SOP Reference |
|-------------------------|--|------------------|---|---------------------------|---|--------------------|-----------------------------------|---------------|
| LC-MS/MS | Mobile Phase Injection site Guard Column Analytical Column Mass Spec | ICAL and CCVs | <ul style="list-style-type: none"> Inspect all tubing connections at time of maintenance to assure no leaks present Check column pressure and mobile phase levels/expiration daily Perform the following as needed: prepare aqueous mobile phase, clean/replace injection needle, replace guard cartridge, backflush/replace column, replace injector seat, clean curtain/orifice plate, retune MS | As Listed with Inspection | MBs and IBs < DL, ICAL, ICV, ICS, CCV, peak shape, & LCS, as seen on Worksheet #28 | Normal replacement | | SVOC-043 |

Notes:

- < – less than
- CA – Corrective Action
- CCV – continuing calibration verification
- DL – detection limit
- IB – instrument blank
- ICAL – initial calibration
- ICV – Initial calibration verification
- ISC – Instrument Sensitivity Check
- LC-MS/MS – Liquid chromatography with tandem mass spectrometry
- LCS – laboratory control sample
- MB – method blank
- MS – Mass spectrometry
- QC – Quality Control
- SOP – Standard Operating Procedure
- SVOC – semivolatile organic compound
- TOC – total organic carbon
- WC – wet chemistry

WORKSHEET #26 & 27: SAMPLE HANDLING, CUSTODY, AND DISPOSAL

Sampling Organization: ECC/Arcadis

Laboratory: Advanced Environmental Laboratories (AEL)

Method of sample delivery (shipper/carrier): Federal Express Overnight

Number of days from reporting until sample disposal: at least 45 days after issuance of data package.

| Activity | Organization and Title/Position of Person Responsible for the Activity | SOP Reference |
|--|--|--|
| Sample labeling | Field Team Leader ECC/Arcadis | See Field SOP P-01 Sample identification will be site-specific. |
| CoC form completion | Field Team Leader ECC/Arcadis | See Field SOPs P-01 and P-02 |
| Packaging | Field Team Leader ECC/Arcadis | See Field SOPs P-01 and P-02 |
| Shipping coordination | Field Team Leader ECC/Arcadis | See Field SOPs P-01 and P-02 |
| Sample receipt, inspection, and log-in | AEL, Sample Receipt Supervisor | QA-QM11872 and S-SA-WI10725 |
| Sample custody and storage | AEL, PFAS Managers | QA-QM11872 and S-SA-WI10725 |
| Sample disposal | AEL, Safety and Regulatory Compliance Officer | QA-QM11872 and S-SA-WI10725 |

Notes:

AEL – Advanced Environmental Laboratories, Inc.
 Arcadis – Arcadis, U.S., Inc.
 ECC – Environmental Chemical Corporation
 CoC – chain of custody
 PFAS – per- and polyfluoroalkyl substances

QA – Quality Assurance
 QM – Quality Manual
 S – sample
 SA – sample analysis
 SOP – Standard Operating Procedure

Laboratory Custody Procedures

A designated sample custodian accepts custody of the samples and verifies that the information on the sample labels matches that on the CoC form(s). The sample custodian will document any discrepancies and will sign and date all appropriate receiving documents. The sample custodian will also document the condition of the samples upon receipt at the laboratory. If a sample container is missing, a sample container is received broken, the sample is in an inappropriate container, or the sample has not been preserved by appropriate means, ECC/Arcadis personnel will be notified as per direction on **Worksheet #6**.

In accordance with laboratory custody and security requirements, the laboratory sample custodian will be responsible for logging the samples in, assigning a unique laboratory identification number to each sample to ensure traceability of samples while in possession of the laboratory, labeling the sample bottle with the laboratory identification number, and moving the sample to an appropriate storage location to await analysis. The project name, field sample code, date sampled, date received, analysis required, storage location and date, and action for final disposition will be recorded in the laboratory tracking system. Relevant custody documentation will be placed in the project file.

The following stages of analysis must be documented by the laboratory:

- Sample
- Sample Analysis
- Data Reduction

- Data Reporting
- Laboratory personnel are responsible for the custody of the samples until they are returned to the sample custodian.

Final Evidence Files

This is the final phase of sample custody. The CoC records and sample analysis request form copies are archived in their respective project files. Laboratory custody forms, sample preparation and analysis logbooks, and data packages will become part of the laboratory final evidence file. Other relevant documentation, including records, reports, correspondence, logs, pictures, and data review reports, will be archived by ECC/Arcadis personnel.

WORKSHEET #28-1: ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION

**(UFP-QAPP Manual Section 3.4)
 (USEPA 2106-G-05 Section 2.3.5)**

| Matrix | | Aqueous | | | |
|--|--|---|--|---|--|
| Analytical Group | | TOC | | | |
| Analytical Method/SOP Reference | | SM5310C / WC-021 | | | |
| QC Sample: | Number / Frequency | Method / SOP QC Acceptance Limits | CA | Title of Person Responsible for CA | Project Specific MPC |
| MB | Once every prep/ analytical batch of 10 or fewer samples | TOC < LOQ | Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank. If not enough sample volume to re- prep, data qualified with case narration. | Analyst with Department Supervisor and QA Officer review. | TOC < LOQ |
| LCS | | %R 80 to 120% | | | Correct problem. If required, reprep and reanalyze LCS and all QC samples and field samples processed in batch. If not enough sample volume to re- prep, data qualified with case narration. |
| MS | | %R 75-125% | When outside limits examine project specific requirements and/or contact client. Flag data appropriately. | Analyst with Department Supervisor review. | %R 75-125% |
| MSD | | RPD ≤ 35% between MS and MSD. | | | RPD ≤ 25% between MS and MSD. |
| DUP | | RPD of all target compounds ≤ 35% between sample and DUP. | | | RPD ≤ 25% |

Notes:

< – less than
 ≤ – less than or equal to
 % – percent
 %R – percent recovery
 CA – corrective action
 DUP – duplicate (laboratory)
 LCS – laboratory control sample
 LOQ – limit of quantitation
 MB – method blank

MPC – Method Performance Criteria
 MS – matrix spike
 MSD – matrix spike duplicate
 QA – Quality Assurance
 QC – Quality Control
 RPD – relative percent difference
 SOP – Standard Operating Procedure
 TOC – total organic carbon
 WC – wet chemistry

WORKSHEET #28-2: ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – TOC IN SOILDS

**(UFP-QAPP Manual Section 3.4)
 (USEPA 2106-G-05 Section 2.3.5)**

| Matrix | | Solids | | | |
|--|---|--|---|---|-----------------------------|
| Analytical Group | | TOC | | | |
| Analytical Method/SOP Reference | | USEPA 9060A / WC-021 | | | |
| QC Sample: | Number / Frequency | Method / SOP QC Acceptance Limits | CA | Title of Person Responsible for CA | Project Specific MPC |
| MB | Once every prep/analytical batch of 20 or fewer samples | TOC < LOQ | Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank. If not enough sample volume to re-prep, data qualified with case narration. | Analyst with Department Supervisor and QA Officer review. | TOC < LOQ |
| LCS | | %R 80-120% | Correct problem. If required, reprep and reanalyze LCS and all QC samples and field samples processed in batch. If not enough sample volume to re-prep, data qualified with case narration. | | %R 90-110% |
| MS | | %R 75-125% | When outside limits examine project specific requirements and/or contact client. Flag data appropriately. | Analyst with Department Supervisor review. | %R 75-125% |
| MSD | | RPD ≤ 20% between MS and MSD. | | | RPD ≤ 25% |
| DUP | | RPD ≤ 20% between sample and DUP. | | | RPD ≤ 25% |

Notes:

< - less than
 ≤ - less than or equal to
 % - percent
 %R – percent recovery
 CA – corrective action
 DUP – duplicate (laboratory)
 LCS – laboratory control sample
 LOQ – limit of quantitation
 MB – method blank

MPC – Method Performance Criteria
 MS – matrix spike
 MSD – matrix spike duplicate
 QA – Quality Assurance
 QC – Quality Control
 RPD – relative percent difference
 SOP – Standard Operating Procedure
 TOC – total organic carbon
 WC – wet chemistry

WORKSHEET #28-3: ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – pH IN GROUND WATER & SURFACE WATER

**(UFP-QAPP Manual Section 3.4) /
 (USEPA 2106-G-05 Section 2.3.5)**

| Matrix | | Aqueous | | | |
|--|--|--|---|---|--------------------------------------|
| Analytical Group | | pH | | | |
| Analytical Method/SOP Reference | | SM9040C/WC-002 | | | |
| QC Sample: | Number / Frequency | Method / SOP QC Acceptance Limits | CA | Title of Person Responsible for CA | Project Specific MPC |
| LCS / CCV | Once every prep/analytical batch of 10 or fewer samples, and at the end of the batch | ± 0.05 SU | Correct problem. If required, reprep and reanalyze LCS/CCV and all QC samples and field samples processed in batch. If not enough sample volume to re-prep, data qualified with case narration. | Analyst with Department Supervisor and QA Officer review. | ± 0.05 SU |
| Sample DUP | Once every prep/analytical batch of 10 or fewer samples | within 0.1 SU of original reading | When outside limits examine project specific requirements and/or contact client. Flag data appropriately. | Analyst with Department Supervisor review. | Within 0.1 pH SU of original reading |

Notes:

± - plus or minus
 CA – corrective action
 CCV – continuing calibration verification
 DUP – duplicate (laboratory)
 LCS – laboratory control sample
 MPC – Method Performance Criteria

QA – Quality Assurance
 QC – Quality Control
 SOP – Standard Operating Procedure
 SU – standard units
 WC – wet chemistry

WORKSHEET #28-4: ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION –pH IN SOILDS

**(UFP-QAPP Manual Section 3.4)
 (USEPA 2106-G-05 Section 2.3.5)**

| Matrix | | Solids – Soil and Sediment | | | |
|--|--|--|---|---|--------------------------------------|
| Analytical Group | | pH | | | |
| Analytical Method/SOP Reference | | SM9045D / WC-057 | | | |
| QC Sample: | Number / Frequency | Method / SOP QC Acceptance Limits | CA | Title of Person Responsible for CA | Project Specific MPC |
| LCS / CCV | Once every prep/analytical batch of 10 or fewer samples, and at the end of the batch | ± 0.05 SU | Correct problem. If required, reprep and reanalyze LCS/CCV and all QC samples and field samples processed in batch. If not enough sample volume to re-prep, data qualified with case narration. | Analyst with Department Supervisor and QA Officer review. | ± 0.05 SU |
| DUP | Once every prep/analytical batch of 10 or fewer samples | Within 0.1 pH SU of original reading | When outside limits examine project specific requirements and/or contact client. Flag data appropriately. | Analyst with Department Supervisor review. | Within 0.1 pH SU of original reading |

Notes:

± - plus or minus
 CA – corrective action
 CCV – continuing calibration verification
 DUP – duplicate (laboratory)
 LCS – laboratory control sample
 MPC – Method Performance Criteria

QA – Quality Assurance
 QC – Quality Control
 SOP – Standard Operating Procedure
 SU – standard units
 WC – wet chemistry

WORKSHEET #28-5: ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – PFAS IN GROUND WATER & SURFACE WATER

**(UFP-QAPP Manual Section 3.4)
 (USEPA 2106-G-05 Section 2.3.5)**

| | |
|--|--|
| Matrix | Aqueous |
| Analytical Group | PFAS |
| Analytical Method/SOP Reference | USEPA 1633 Draft and DoD QSM 5.4 B-24 / SVOC - 043 |

| QC Sample: | Number / Frequency | Method / SOP QC Acceptance Limits | CA | Title of Person Responsible for CA | Project Specific MPC |
|------------|--|--|--|---|--|
| MB | Once every prep/analytical batch of 20 or fewer samples | Must be spiked with EIS and subjected to prep procedure. Must be 1) < 1/2 the LOQ, or 2) < 1/10th the concentration found in any sample in the prep batch, or 3) < 1/10th the regulatory limit, whichever of the three concentrations is greater. | Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank. If not enough sample volume to re-prep, data qualified with case narration. | Analyst with Department Supervisor and QA Officer review. | Must be 1) < 1/2 the LOQ, or 2) < 1/10th the concentration found in any sample in the prep batch, or 3) < 1/10th the regulatory limit, whichever of the three concentrations is greater. |
| EIS | Added to every field sample, standard, blanks, and QC samples. | QC samples and field samples must recover within in-house limits. Preliminary in-house acceptance criteria of 20-150% must be used until in-house limits are generated. The lower limit of inhouse acceptance criteria cannot be < 20% | Repeat the analysis using a fresh aliquot of the extract. If failure does not confirm, report the second analysis. If the failure confirms, follow the requirements listed in USEPA Draft Method 1633, Section 15.3.2. If EIS recoveries still fall outside of the acceptance range, the client must be contacted for additional measures to be taken. | | Preliminary in-house acceptance criteria of 20-150% must be used until in-house limits are generated. The lower limit of inhouse acceptance criteria cannot be < 20% |

| QC Sample: | Number / Frequency | Method / SOP QC Acceptance Limits | CA | Title of Person Responsible for CA | Project Specific MPC |
|--|--|---|--|---|---|
| NIS | Added to every field sample, standard, blanks, and QC samples. | NIS areas must be greater than 30% of the average area of the calibration standards in undiluted sample extracts and sample extracts that required additional NIS to be added. NIS areas corrected for the dilution factor must be greater than 30% of the average area of the calibration standards in diluted samples when additional NIS was not added post dilution of the extract | Repeat the analysis using a fresh aliquot of the extract. If failure does not confirm, report the second analysis. If the failure confirms, examine the project-specific requirements. Contact the client as to additional measures to be taken. | Analyst with Department Supervisor and QA Officer review. | NIS areas must be greater than 30% of the average area of the calibration standards in undiluted sample extracts. |
| IAR | All analytes detected in a sample. | Must meet all of the requirements of USEPA Draft Method 1633. Construct an acceptance window for the IAR of each target analyte as 50% to 150% of the IAR in the mid-point calibration standard or daily CCV standard | Apply I-flag to the result associated with the failure. | Analyst with Department Supervisor review. | Construct an acceptance window for the IAR of each target analyte as 50% to 150% of the IAR in the mid-point calibration standard or daily CCV standard |
| FD | One per 20 field samples | $RPD \leq 30\%$ for results > 5 times the LOQ | Qualify data as appropriate | Data Validator | $RPD \leq 30\%$ for results > 5 times the LOQ |
| LCS and LLCS. (Equivalent to as OPR and LLOPR in method 1633) | Once every prep/analytical batch of 20 or fewer samples | Fortify the LLCS with method analytes at or below the LOQ. Fortify the LCS with method analytes at a concentration near the mid-point of the curve. If not enough sample is provided for MS/MSD, then a LCSD must be performed. Recovery must be within 40-150%, until in-house limits can be created. In-house limits cannot be lower than 40%. | Correct problem. If required, reprep and reanalyze LCS and all QC samples and field samples processed in batch. If not enough sample volume to re-prepare, data qualified with case narration. | Analyst with Department Supervisor and QA Officer review. | Recovery must be within 40-150%, until in-house limits can be created, or if project limits are not provided. In-house limits cannot be lower than 40%. |
| MS | Once every prep/analytical batch of 20 or fewer samples | Fortify the MS with method analytes at a concentration close to but greater than the native concentrations (if known), or near the mid-point of the curve. Recoveries must be within the LCS limits – 40-150%. | When outside limits examine project specific requirements and/or contact client. Flag data appropriately. | Analyst with Department Supervisor review. | Recoveries must be within the LCS limits – 40-150% |

| QC Sample: | Number / Frequency | Method / SOP QC Acceptance Limits | CA | Title of Person Responsible for CA | Project Specific MPC |
|------------|---|---|---|--|---|
| MSD | Once every prep/analytical batch of 20 or fewer samples | Fortify the MSD with method analytes at a concentration close to but greater than the native concentrations (if known), or near the mid-point of the curve. recoveries must be within the LCS limits – 40-150%. % RPD must be $\leq 30\%$. | When outside limits examine project specific requirements and/or contact client. Flag data appropriately. | Analyst with Department Supervisor review. | Recoveries must be within the LCS limits – 40-150% RPD $\leq 30\%$ |

Notes:

> - greater than
 < - less than
 \leq - less than or equal to
 % - percent
 %R – relative percent
 CA – corrective action
 CCV – continuing calibration verification
 DoD – Department of Defense
 EIS – extracted internal standards
 FD – field duplicate
 IAR – ion ratio
 LCS – laboratory control sample
 LCSD – laboratory control sample duplicate
 LLCS – low-level laboratory control sample
 LLOPR – low level ongoing precision and recovery

LOQ – limit of quantitation
 MB – method blank
 MPC – Method Performance Criteria
 MS – matrix spike
 MSD – matrix spike duplicate
 NIS – Non-extracted Internal Standards
 OPR – ongoing precision and recovery
 PFAS – Per- and Polyfluoroalkyl substances
 QA – Quality Assurance
 QC – Quality Control
 QSM – Quality Systems Manual
 RPD – relative percent difference
 SVOC – Semivolatile organic compound
 USEPA – United States Environmental Protection Agency

WORKSHEET #28-6: ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION – PFAS SOLIDS

**(UFP-QAPP Manual Section 3.4)
 (USEPA 2106-G-05 Section 2.3.5)**

| QC Sample: | Number / Frequency | Method / SOP QC Acceptance Limits | CA | Title of Person Responsible for CA | Project Specific MPC |
|------------|--|--|--|---|--|
| MB | Once every prep/analytical batch of 20 or fewer samples | Must be spiked with EIS and subjected to prep procedure. Must be 1) less than ½ the MRL/LOQ, or 2) < 1/10th the concentration found in any sample in the prep batch, or 3) < 1/10th the regulatory limit, whichever of the three concentrations is greater. | Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank. If not enough sample volume to re-prep, data qualified with case narration. | Analyst with Department Supervisor and QA Officer review. | Must be 1) less than ½ the LOQ, or 2) < 1/10th the concentration found in any sample in the prep batch, or 3) < 1/10th the regulatory limit, whichever of the three concentrations is greater. |
| EIS | Added to every field sample, standard, blanks, and QC samples. | QC samples and field samples must recover within in-house limits. Preliminary in-house acceptance criteria of 20-150% must be used until in-house limits are generated. The lower limit of inhouse acceptance criteria cannot be < 20% | Repeat the analysis using a fresh aliquot of the extract. If failure does not confirm, report the second analysis. If the failure confirms, follow the requirements listed in USEPA Draft Method 1633, Section 15.3.2. If EIS recoveries still fall outside of the acceptance range, the client must be contacted for additional measures to be taken. | | Preliminary in-house acceptance criteria of 20-150% must be used until in-house limits are generated. The lower limit of inhouse acceptance criteria cannot be < 20% |
| NIS | | NIS areas must be greater than 30% of the average area of the calibration standards in undiluted sample extracts and sample extracts that required additional NIS to be added. NIS areas corrected for the dilution factor must be greater than 30% of the average area of the calibration standards in diluted samples when additional NIS was not added post dilution of the extract | Repeat the analysis using a fresh aliquot of the extract. If failure does not confirm, report the second analysis. If the failure confirms, examine the project-specific requirements. Contact the client as to additional measures to be taken. | | NIS areas must be greater than 30% of the average area of the calibration standards in undiluted sample extracts. |
| IAR | All analytes detected in a sample. | Must meet all of the requirements of USEPA Draft Method 1633. Construct an acceptance window for the IAR of each target analyte as 50% to 150% of the IAR in the mid-point calibration standard or daily CCV standard | Apply I-flag to the result associated with the failure. | Analyst with Department Supervisor review. | Construct an acceptance window for the IAR of each target analyte as 50% to 150% of the IAR in the mid-point calibration standard or daily CCV standard |

| QC Sample: | Number / Frequency | Method / SOP QC Acceptance Limits | CA | Title of Person Responsible for CA | Project Specific MPC |
|--|---|--|---|---|---|
| FD | One per 20 field samples | RPD \leq 30% for results > 5 times the LOQ | Qualify data as appropriate | Data Validator | RPD \leq 30% for results > 5 times the LOQ |
| LCS and LLCS. (Equivalent to as OPR and LLOPR in method 1633) | Once every prep/analytical batch of 20 or fewer samples | Fortify the LLCS with method analytes at or below the MRL. Fortify the LCS with method analytes at a concentration near the mid-point of the curve. If not enough sample is provided for MS/MSD, then a LCSD must be performed. Recovery must be within 40-150%, until in-house limits can be created. In-house limits cannot be lower than 40%. | Correct problem. If required, reprep and reanalyze LCS and all QC samples and field samples processed in batch. If not enough sample volume to re-prep, data qualified with case narration. | Analyst with Department Supervisor and QA Officer review. | Recovery must be within 40-150%, until in-house limits can be created, or if project limits are not provided. In-house limits cannot be lower than 40%. |
| MS | | Fortify the MS with method analytes at a concentration close to but greater than the native concentrations (if known), or near the mid-point of the curve. recoveries must be within the LCS limits – 40-150%. | When outside limits examine project specific requirements and/or contact client. Flag data appropriately. | Analyst with Department Supervisor review. | Recoveries must be within the LCS limits – 40-150% |
| MSD | | Fortify the MSD with method analytes at a concentration close to but greater than the native concentrations (if known), or near the mid-point of the curve. recoveries must be within the LCS limits – 40-150%. % RPD must be \leq 30%. | | | Recoveries must be within the LCS limits – 40-150% RPD \leq 30% |

Notes:

> - greater than
 < - less than
 \leq - less than or equal to
 % - percent

CA – corrective action
 CCV – Continuing Calibration Verification
 EIS – Extracted Internal Standards
 FD – Field Duplicate
 IAR – ion ratio
 LCS – laboratory control sample
 LLCS – low-level laboratory control sample
 LLOPR – low level ongoing precision and recovery
 LOQ – limit of quantitation

MB – method blank
 MRL – method reporting limit
 MS – matrix spike
 MSD – matrix spike duplicate
 NIS – Non-extracted Internal Standards
 OPR – ongoing precision and recovery
 PFAS – per- and polyfluoroalkyl substances
 QA – Quality Assurance
 QC – Quality Control
 RPD – relative percent difference
 SOP – Standard Operating Procedure
 USEPA – United States Environmental Protection Agency

WORKSHEET #29: PROJECT DOCUMENTS AND RECORDS TABLE

**(UFP-QAPP Manual Sections 3.5.1)
 (USEPA 2106-G-05 Sections 2.2.8)**

| Document | Storage Location |
|--|--|
| Programmatic APP and SSHP | ECC Project SharePoint |
| Field Data Collection Sheets | |
| Analytical Data Packages | |
| DUA and associated DVRs | |
| Field Logs, DQCRs, Air Bills, Communication Logs, Non-Conformance Reports, Corrective Action Reports, Documentation of Deviation from Field Methods | |
| Field CoC Records | |
| Laboratory Quality Assurance Plan | Laboratory Record (AEL) |
| Method Detection Limit Study Information | |
| ELAP | |
| Sample Receipt and Tracking Records | |
| State Laboratory Accreditations Drinking Water | |
| Laboratory CoC Records | |
| Equipment Calibration Logs | |
| Sample Preparation Logs | |
| Corrective Action Forms and Reports and Documentation of Corrective Action Results | |
| Data Summary and Instrument Raw Data for Field Samples, Standards, QC Checks, and QC Samples | |
| Laboratory Internal Data Package Completeness Checklist | |
| Standards Traceability Records, Analytical Audit Checklists | |
| Electronic Copy of Analytical Data Reports | ECC Project SharePoint and Laboratory Record (AEL) |
| Case Narrative, Definition of Laboratory Qualifiers, Documentation of Laboratory Method Deviations, Laboratory Sample Identification Numbers, Signatures for Laboratory Sign-Off | |
| EDDs EQuIS 4-File Format and SEDD 2a | |

Notes:

- Data uploads will be coordinated at the Installation level. ECC will establish a secure project-specific MS Teams site to serve as a repository for all site documents and data. The site will be secure and redundant, allowing for the complete transmittal of files to USAEC at the end of the contract. A Project Portal will also be established for file transfer. DoD's Secure Access File Exchange will also be used for file transmittal.

AEL – Advanced Environmental Laboratories, Inc.

EDD – electronic data deliverable

APP – Accident Prevention Plan

ELAP – Environmental Laboratory Accreditation Program

CoC – chain of custody

QC – Quality Control

DQCR – Data Quality Control Report

SEDD – staged electronic data deliverable

DUA – Data Usability Assessment

SSHP – Site Safety and Health Plan

DVR – Data Validation Report

ECC – Environmental Chemical Corporation

WORKSHEET #31, 32 & 33: ASSESSMENTS AND CORRECTIVE ACTION

**(UFP-QAPP Manual Sections 4.1.1 and 4.1.2)
 (USEPA 2106-G-05 Section 2.4 and 2.5.5)**

| ASSESSMENTS | | | | | |
|---|------------------------------------|-------------------------|--|---|---|
| Assessment Type | Responsible Party/ Organization | Number/ Frequency | Estimated Dates | Assessment Deliverable | Deliverable Due Date |
| Review of UFP-QAPP and SOPs with field staff | ECC/Arcadis | Before sampling startup | Before sampling | Documented in field checklist | Completed at beginning of each type/medium field activity and as needed during sampling |
| Daily logbook and field forms | | Daily | During field activities | Contained within written report | As part of Draft Report |
| Laboratory assessment for appropriate certifications and capacity and UFP-QAPP review with laboratory staff | | Before sampling startup | Before sampling | Receipt of copies of certifications. Email traffic concerning laboratory capacity before sampling startup. UFP-QAPP sign-off sheet received from laboratory. | N/A |
| Field sampling and CoC review against UFP-QAPP requirements | | Daily | During field activities | Communication in the form of an email. | Notify laboratory or field team, as appropriate, of corrections. Last email received no later than 24 hours after last sampling event |
| Laboratory report deliverables and analytical results review against UFP-QAPP requirements | | SDG | Immediately following receipt of laboratory report | | N/A |
| Data verification | Arcadis | Per SDG | | Communication in the form of an email requesting additional laboratory forms, backup data that may be missing, and/or clarification of the analytical report. | Three weeks after receipt of data. |
| Data validation | | | | Data Validation Report | |

Notes:

Arcadis – Arcadis, U.S., Inc.
 CoC – Chain of Custody
 ECC – Environmental Chemical Corporation
 N/A – not applicable
 QAPP – Quality Assurance Project Plan
 SDG – Sample delivery group
 UFP – Uniform Federal Policy

| ASSESSMENT RESPONSE AND CORRECTIVE ACTION | | | | | |
|---|---|---|------------------------------------|---------------------------------------|--|
| Assessment Type | Responsibility for Responding to Assessment Finding | Assessment Response Documentation | Timeframe for Response | Responsibility for Implementing CA | Responsible for Monitoring CA Implementation |
| Review of UFP-QAPP and SOPs with field staff | FTL ECC/Arcadis | Field progress report, non-conformance report, or CA report dependent on significance of finding | Within 24 hours | FTL and/or Regional Lead, ECC/Arcadis | Jackson Kiker, ECC |
| Daily logbook and field forms | | | | FTL ECC/Arcadis | Regional Lead, ECC/Arcadis |
| Laboratory assessment for appropriate certifications and capacity and UFP-QAPP review with laboratory staff | Craig Myers | Response to email | Within 48 hours of notification | Craig Myers | Project Chemist, ECC/Arcadis |
| Field sampling and CoC review against UFP-QAPP requirements | FTL ECC/Arcadis | | Within 24 hours after sampling | FTL ECC/Arcadis | Field Team Leader ECC/Arcadis |
| Laboratory report deliverables and analytical results review against UFP-QAPP requirements | Craig Myers | If required, laboratory reports will be amended and corrections noted in the case narrative. | Within 72 hours after notification | Craig Myers | Sharon Pennington, Project Chemist, or designated validator, Arcadis |
| Data verification | | If required, laboratory reports will be amended and corrections noted in the case narrative and contained within the validation report. | Up to seven days | | |
| Data validation | | | | | |

Notes:

Arcadis – Arcadis, U.S., Inc.
 CA – corrective action
 CoC – chain of custody
 ECC – Environmental Chemical Corporation
 FTL – Field Team Leader
 QAPP – Quality Assurance Project Plan
 SDG – Sample Delivery Group
 SOP – Standard Operating Procedure
 UFP – Uniform Federal Policy

WORKSHEET #34: DATA VERIFICATION AND VALIDATION INPUTS

**(UFP-QAPP Manual Section 5.2.1 and Table 9)
 (USEPA 2106-G-05 Section 2.5.1)**

| Item | Description | Verification (completeness) | Validation (conformance to specifications) |
|-----------------------------------|---|-----------------------------|--|
| <i>Planning Documents/Records</i> | | | |
| 1 | Contract | X | |
| 2 | Field Standard Operating Procedures (SOPs) | X | X |
| 3 | Laboratory SOPs | X | X |
| <i>Field Records</i> | | | |
| 4 | Field logbooks | X | |
| 5 | Equipment calibration records | X | |
| 6 | Chain of Custody (CoC) Forms | X | X |
| 7 | Sampling logs | X | |
| 8 | Drilling logs | X | |
| 9 | Change orders/deviations | X | |
| 10 | Field audit reports | X | |
| 11 | Field Corrective Action Reports/Field Non-conformance Reports | X | |
| <i>Analytical Data Package</i> | | | |
| 12 | Cover sheet (laboratory identifying information) | X | X |
| 13 | Case narrative | X | X |
| 14 | Internal laboratory CoC | X | X |
| 15 | Sample receipt records | X | X |
| 16 | Sample chronology (i.e., dates and times of receipt, preparation, & analysis) | X | X |
| 17 | Communication records | X | X |
| 18 | Limit of Detection/Limit of Quantitation establishment and verification | X | X |
| 19 | Standards Traceability | X | X |
| 20 | Instrument calibration records | X | X |
| 21 | Definition of laboratory qualifiers | X | X |
| 22 | Results reporting forms | X | X |
| 23 | Quality Control sample results | X | X |
| 24 | Corrective Action reports | X | X |
| 25 | Raw data | X | X |
| 26 | Electronic Data Deliverable (EQuIS 4-File Format and Staged Electronic Data Deliverable (SEDD 2a) | X | X |

WORKSHEET #35: DATA VERIFICATION PROCEDURES

(UFP-QAPP Manual Section 5.2.2)
 (USEPA 2106-G-05 Section 2.5.1)

| Records Reviewed | Required Documents | Process Description | Responsible Person, Organization |
|---|--------------------|---|---|
| Field logbook | UFP-QAPP | <ul style="list-style-type: none"> Process Description Verify that records are present and complete for each day of field activities. Verify that all planned samples, including field QC samples, were collected and that sample collection locations are documented. Verify that meteorological data were provided for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements. Verify that any required field monitoring was performed, and results are documented. | <p><i>Daily:</i> Lead/Field Team Leader, ECC/Arcadis</p> |
| CoC forms | UFP-QAPP | <ul style="list-style-type: none"> All samples to be analyzed by the laboratory will be shipped via overnight delivery or will be sent via the laboratory courier service. Upon receipt, the laboratory sample custodian will check the integrity of the custody seals and will sign and date the CoC to acknowledge receipt. The laboratory is responsible for verifying that the CoC and containers agree and that the sample containers are received in good condition. The sample receipt form will be sent to the ECC/Arcadis PM before preparation for analysis. The Laboratory Information Management System will provide evidence of sample custody from receipt by the laboratory until appropriate disposal. | <p><i>Daily:</i> Lead/Field Team Leader, ECC/Arcadis <i>Upon receipt:</i> Steve Warren AEL PM</p> |
| Laboratory Non-conformance/ <u>Corrective Action</u> and report procedure | UFP-QAPP | <ul style="list-style-type: none"> Routine Corrective Actions apply to all analytical QC parameters and analytical system specifications as defined in the laboratory SOPs. Analysts have full responsibility and authority for performing routine Corrective Actions, which are documented as part of the analytical record. Defective processes, holding time violations, systematic errors, and quality defects that occur are to be reported by the analyst to the laboratory supervisor and a non-conformance record initiated. The laboratory PM will then notify the ECC/Arcadis PM and/or Project Chemist. All notifications must be made in a timely manner. The non-conformance record must become part of the analytical record. | <p><i>Before release:</i> Heather Quilal-lan, QA Manager Steve Warren AEL PM <i>Upon receipt:</i> Sharon Pennington, Arcadis or Lyndi Mott, Arcadis</p> |

| Records Reviewed | Required Documents | <ul style="list-style-type: none"> Process Description | Responsible Person, Organization |
|---------------------------------------|--|---|---|
| Analytical Data Package - Laboratory | UFP-QAPP Lab QA Manual Lab SOPs | <ul style="list-style-type: none"> All data produced by the laboratory will be required to undergo several levels of review, which will include two levels of management review at the laboratory. The laboratory will review the data packages internally for completeness and verify that all the required forms and raw data are included for each data package type. The Lab QA Manager may also select to review randomly chosen data packages for additional audits. | <p><i>Before release:</i> Steve Warren AEL PM</p> <p><i>Upon receipt:</i> Sharon Pennington, Arcadis or Lyndi Mott, Arcadis or designated validator</p> |
| Analytical Data Package/Laboratory QC | UFP-QAPP, DoD QSM 5.4 or later versions Lab SOPs Analytical Methods, DoD Data Validation Guidance document | <ul style="list-style-type: none"> The Data Validator will verify that data have been received for all samples sent to the laboratory. These data will be evaluated to determine whether the laboratory met the QC requirements as stated in this UFP-QAPP, DoD QSM 5.4 or later versions, DoD Data Validation Guidance documents, analytical methods, and laboratory SOPs. The QSM version used for data evaluation will be the version for which the lab is certified under DoD ELAP at the time of analysis. | Sharon Pennington, Arcadis or Lyndi Mott, Project Chemist, or designated validator, Arcadis |
| Laboratory EDD | UFP-QAPP | <ul style="list-style-type: none"> The laboratory will provide EDDs. The database manager or designee will review these files for correctness and completeness. EDD format will be EQuIS 4-File Format and SEDD 2a. | Sharon Pennington, Arcadis or Lyndi Mott, Arcadis and Jackson Kiker, ECC |

Notes:

All required data deliverables must be present in the data package to proceed to the next step of data validation (**Worksheet # 36**).

AEL – Advanced Environmental Laboratories, Inc.

Arcadis – Arcadis, U.S., Inc.

CoC – chain of custody

DoD – Department of Defense

ECC – Environmental Chemical Corporation

EDD – electronic data deliverable

PM – Project Manager

QA – Quality Assurance

QAPP – Quality Assurance Project Plan

QC – Quality Control

QSM – Quality Systems Manual

SEDD – Staged Electronic Data Deliverable

SOP – Standard Operating Procedure

UFP – Uniform Federal Policy

WORKSHEET #36: DATA VALIDATION PROCEDURES

**(UFP-QAPP Manual Section 5.2.2)
 (USEPA 2106-G-05 Section 2.5.1)**

| | | |
|--|---|--|
| Data Validator: | Arcadis Project Chemist, or designee | |
| Analytical Group/Method: | PFAS, TOC | pH, Grain Size |
| Data deliverable requirements: | Stage 4 Data Package (pdf) EQuIS 4-File EDD and SEDD 2a | Stage 2A Data Package (pdf); EQuIS 4-file Format and SEDD 2a |
| Analytical specifications: | PFAS per DoD QSM 5.4 Table B-24 (USEPA Method 1633) USEPA 537.1 Drinking Water (Appendix G) TOC by SW-846 9060A and SM 5310C | pH by SW-846 9040C and 9045D Grain Size by ASTM D422 |
| Measurement performance criteria: | DoD QSM 5.4 or later versions; Worksheets #12 and #28 | Laboratory SOPs and QC Control Limits- Worksheets #12 and #28 |
| Percent of data packages to be validated: | 100% Stage 2B manual | 0% |
| Percent of raw data reviewed: | 10% Stage 4 (only PFAS) | 0% |
| Percent of results to be recalculated: | 10% as part of Stage 4 (select PFAS detections) | 0% |
| Validation procedure: | <ul style="list-style-type: none"> • DoD General Data Validation Guidelines. November 2019. • DoD Data Validation Guidelines Module 6: Data Validation Procedure for PFAS Analysis by QSM Table B-24. October 2022. • USEPA Data Review and Validation Guidelines for PFAS Analyzed Using USEPA Method 537. USEPA 910-R-18-001 November 2018. • This UFP-QAPP, DoD QSM 5.4 or later versions • Lab SOPs • DVRs are produced for each sample delivery group. | Not applicable. No DVRs are produced for these methods. |

Notes:

100% of the data will be reviewed and verified. Data validation is not required for the geotechnical parameters (e.g., pH, grain size distribution).

% - percent

ASTM – American Society for Testing and Materials

DoD – Department of Defense

DVR – data validation report

EDD – electronic data deliverable

PFAS – per- and polyfluoroalkyl substances

QC – Quality Control

QSM – Quality Systems Manual

SEDD – Staged Electronic Data Deliverable

SOP – Standard Operating Procedure

TOC – total organic carbon

USEPA – United States Environmental Protection Agency

WORKSHEET #37: DATA USABILITY ASSESSMENT

**(UFP-QAPP Manual Section 5.2.3 including Table 12)
 (USEPA 2106-G-05 Section 2.5.2, 2.5.3, and 2.5.4)**

Data validation will be performed on the field samples collected during the sampling events per **Worksheet #36**. The review will consist of a verification and validation based on completeness and compliance checks of sample receipt conditions and both sample-related and instrument-related QC results, as addressed in **Worksheets #12, #24, and #28**. Any data validation qualifiers that limit the usability of the data will be applied to all applicable samples. DoD General Data Validation Guidelines November 2019 Revision 1, Section 4.8 states: “*The following provides a brief explanation of the DoD data validation qualifiers assigned to results during the data review process by a data validator. The reviewer should use these qualifiers, as applicable, unless other data qualifiers are specified in a project related document, such as a UFP-QAPP. If other qualifiers are used, a complete explanation of those qualifiers should accompany the data validation report.*” Below are the qualifier codes that may be applied to sample results.

| Qualifier | DoD Data Validation Qualifier Description |
|-----------|---|
| J | The reported result was an estimated value with an unknown bias. |
| J+ | The result was an estimated quantity, but the result may be biased high. |
| J- | The result was an estimated quantity, but the result may be biased low. |
| UJ | The analyte was not detected and was reported as less than the LOD or as defined by the customer. However, the associated numerical value is approximate. |
| U | Analyte considered non-detect at the listed value due to associated blank contamination as noted in DVR. |
| J+ | The reported result may be biased high due to associated blank contamination as noted in DVR. |
| R | Rejected. This datum has been deemed by the project team, due to deficiencies in meeting QC criteria and/or DQOs, as unusable for use in making project decisions. |
| X | The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Acceptance or rejection of the data should be decided by the project team (which should include a project chemist), but exclusion of the data is recommended. |

Notes:

DQO – data quality objective
 DVR – data validation report

LOD – limit of detection
 QC – quality control

DUA is performed by all ECC/Arcadis team members and stakeholders. The first usability assessment will be performed by the Project Chemist, who will validate the project data to determine its quality with respect to QAPP MPC. Next, the usability of the data will be reviewed by the ECC/Arcadis team with respect to proper sample collection methods/techniques, proper sample locations selected, proper sample time/season, and further evaluated with respect to applicable data trends and/or models. During data validation, the following DQIs will be evaluated for each method and matrix. Any limitations to the data will be presented in a summary of the Arcadis DVR. Each project report will include a general data usability statement, which will briefly summarize any data affected by serious deficiencies, any systemic laboratory bias, and any matrix bias, and a DUA with associated DVRs will be provided in each report.

Precision: Results of field duplicates will be presented separately in tabular format for each sample pair. For each field duplicate pair, the results will be assessed as stated in **Worksheet #12**. MS/MSD RPDs are calculated by the laboratory and those with RPDs outside the criteria established in **Worksheet #12** will be listed in the DVR. The DVR will summarize all field duplicate results for each analytical method and matrix in a tabular format. A brief discussion will follow summarizing the results of the laboratory precision. Any

conclusions about the precision of the analyses will be drawn, and any limitations on the use of the data will be described.

Accuracy/Bias Contamination: Results for all laboratory MBs, equipment blanks, and continuing calibration blanks will be evaluated, and analytes detected in these blanks will be discussed in the DVR. Laboratory data will be qualified based on the criteria listed in **Worksheet #12**. The DVR will summarize all analytes qualified based on blank contamination for each analytical method and matrix in a tabular format. A brief discussion will follow summarizing the results of the laboratory accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn, and any limitations on the use of the data will be described.

Overall Accuracy/Bias: Results for all LCSs, surrogates, extracted internal standards, non-extracted internal standards, and MS/MSD recoveries that are outside evaluation criteria will be presented in the DVRs. The results will be checked versus those listed in **Worksheet #12**. The DVR will summarize all analytes that were qualified based on LCS, surrogates, extracted internal standards, non-extracted internal standards, and MS/MSD recoveries for each analytical method and matrix. A discussion will follow summarizing the overall accuracy/bias. Any conclusions about the accuracy/bias of the analyses will be drawn, and any limitations on the use of the data will be described.

Representativeness: Representativeness is a qualitative measure of the degree to which data accurately and precisely represent a characteristic of a population and data is mainly addressed in the sample design. A measure of representativeness can also be obtained by assessing holding times and blank data. The DVR will summarize all analytes that were qualified based on holding time or preservation requirements for each analytical method and matrix. Any conclusions about the representativeness of the samples will be drawn, and any limitations on the use of the data will be described.

Comparability: In accordance with this UFP-QAPP, the data are comparable when collection techniques, measurement method, and reporting procedures are the same for each dataset. The DUA will describe issues with comparability.

Completeness: A completeness check will be performed on all data generated by the laboratory. Completeness criteria are presented on **Worksheet #12**. Completeness will be calculated as the number of data points for each analyte that is deemed useable (not rejected) divided by the total number of data points for each analyte. A discussion in the DUA will follow summarizing the results of the calculation of data completeness. Any conclusions about the completeness of the data will be drawn, and any limitations on the use of the data will be described. Data completeness addresses only those samples that are collected and only data that are analyzed by the laboratory.

Reconciliation: Each of the MPCs listed in **Worksheet #12** will be examined to determine if the objective was met. Each analysis will be evaluated separately in terms of the major impacts observed from the data verification/validation, DQIs, and MPC assessments. Based on the results of these assessments, the chemical quality of the data will be determined. Usability of the data will be based on the chemical quality assessment during data validation and the assessment that it is representative of site conditions by being properly collected and satisfying project DQOs.

The Arcadis DVR will include a presentation of method QC reviewed and a presentation of data exceeding a QC check MPC for each reviewed method. Data exceeding an MPC for a DQI will be reconciled with the project DQOs and appropriately qualified. Any conclusions or limitations on the usability of any of the data will be described in the DUA and/or the DVR. Each project report will include general data usability report statements, which will briefly summarize any data affected by serious deficiencies, any systemic laboratory bias, and any matrix bias.

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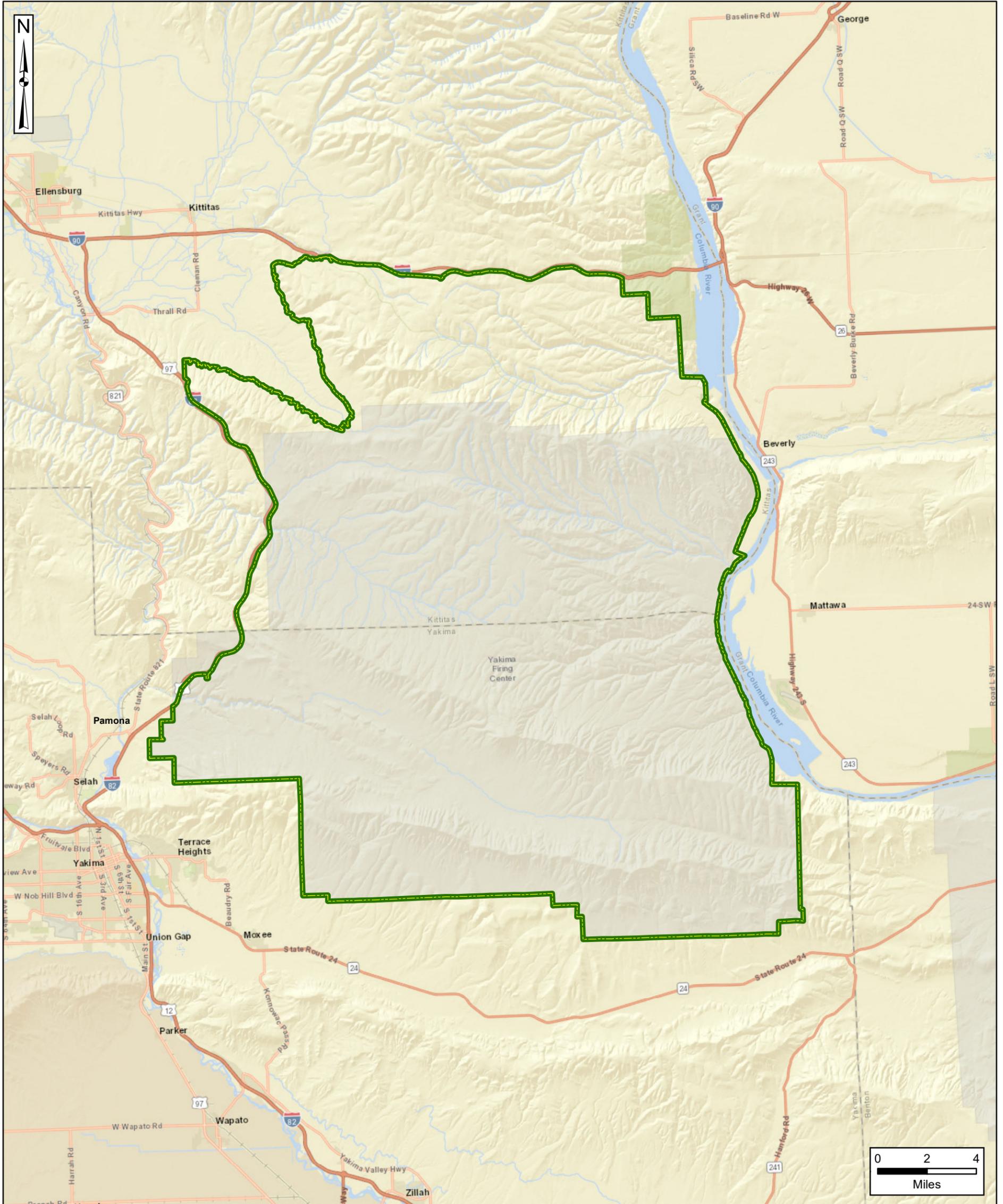
FIGURES



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 Yakima Training Center, WA



Figure 10-1
 Site Location



 Installation Boundary

Data Sources:
 Yakima Training Center, GIS Data, 2018
 ESRI ArcGIS Online, Street Map Data

Coordinate System:
 WGS 1984, UTM Zone 10 North



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 Yakima Training Center, WA

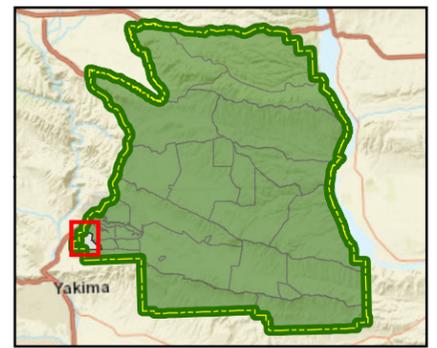
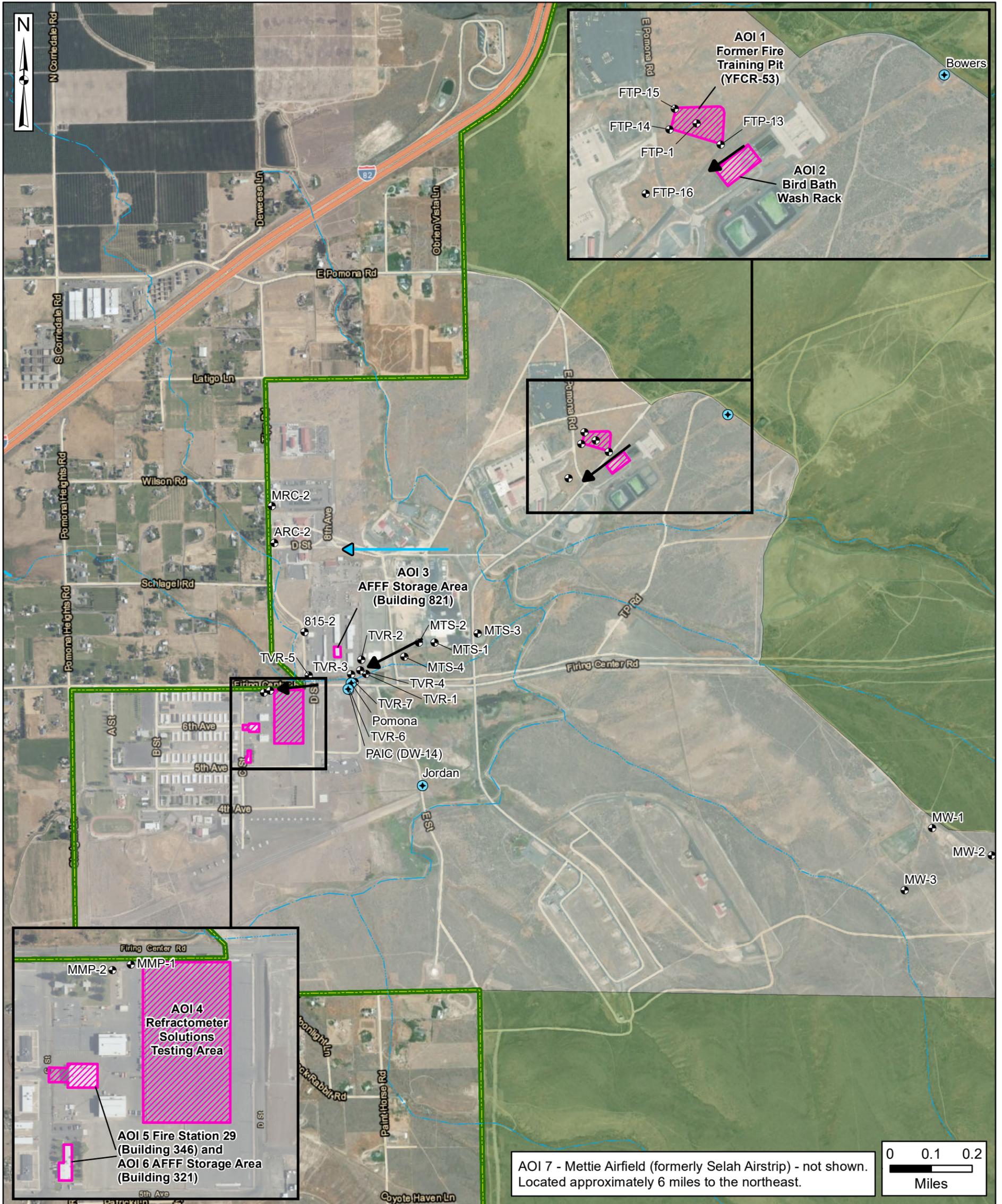


Figure 10-2
 Site Features and AOI Locations



- Installation Boundary
- Potable Water Well (On-Installation)
- Cantonment Area
- River/Stream (Perennial)
- Range/Training Area
- River/Stream (Intermittent)
- AOI
- Canal/Ditch
- AFFF Use Area
- Deep Groundwater (i.e., Used for Installation)
- Potable Supply) Flow Direction (Inferred)
- Monitoring Well
- Perched Groundwater Flow Direction (Inferred)

AFFF = Aqueous Film-Forming Foam
 AOI = Area of Interest

Data Sources:
 Yakima Training Center, GIS Data, 2018
 ESRI ArcGIS Online, Aerial Imagery

Coordinate System:
 WGS 1984, UTM Zone 10 North



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 Yakima Training Center, WA

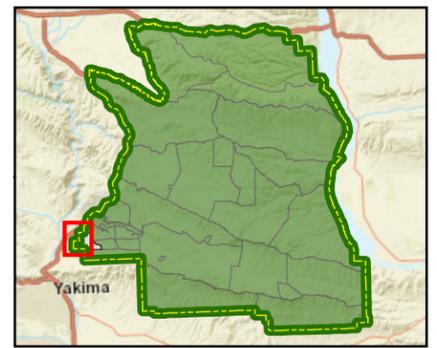
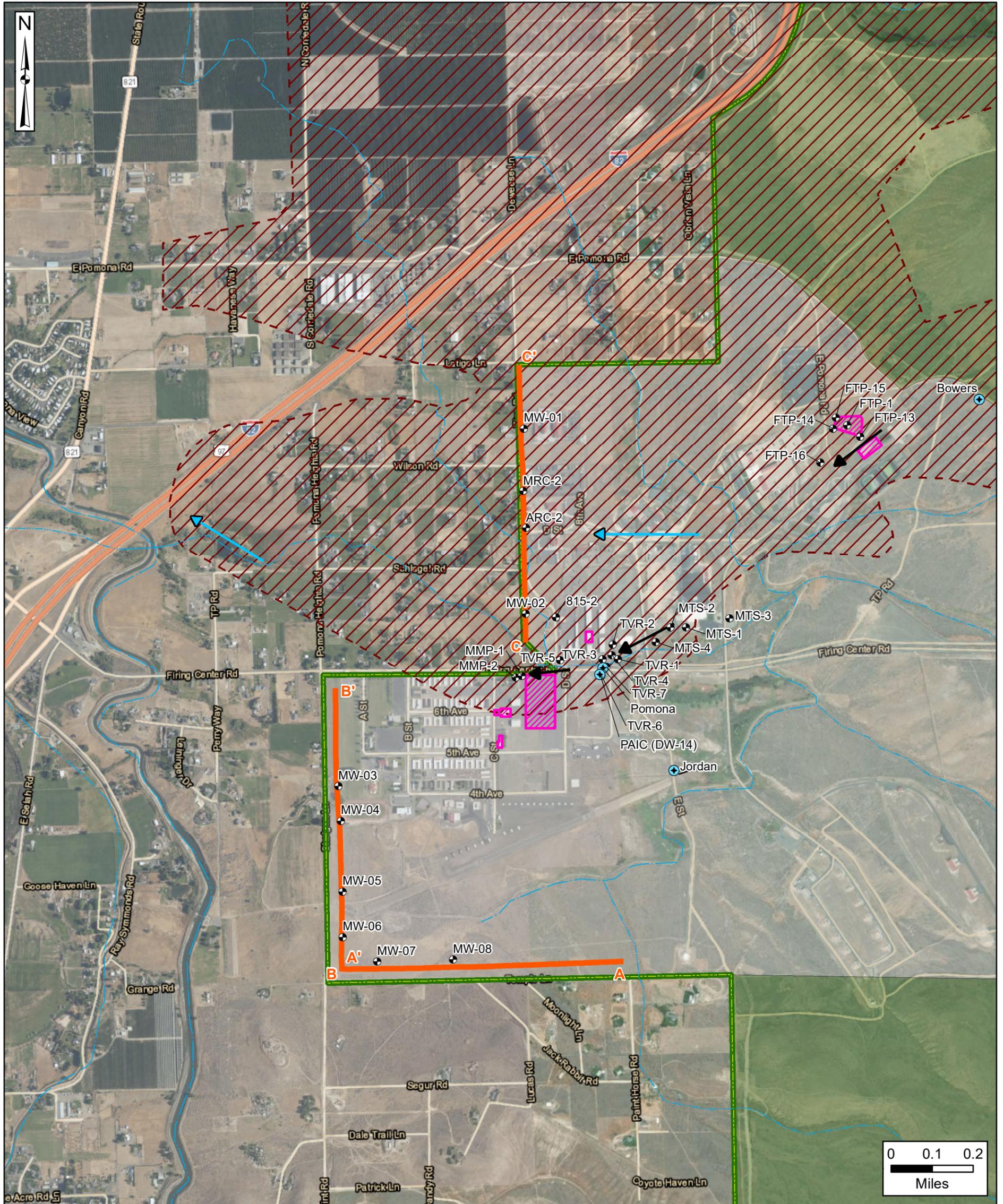


Figure 10-3
Surface Geophysics Transects and
Boundary Well Locations



| | | |
|-----------------------|---|---|
| Installation Boundary | Potable Water Well (On-Installation) | Deep Groundwater (i.e., Used for Installation Potable Supply) Flow Direction (Inferred) |
| Cantonment Area | River/Stream (Perennial) | Perched Groundwater Flow Direction (Inferred) |
| Range/Training Area | River/Stream (Intermittent) | Surface Geophysics Transect |
| AOI | Canal/Ditch | AFFF = Aqueous Film-Forming Foam |
| AFFF Use Area | Approximate Limits of Pomona Basalt (dashed where inferred) | AOI = Area of Interest |
| Monitoring Well | | |

Data Sources:
 Yakima Training Center, GIS Data, 2018
 ESRI ArcGIS Online, Aerial Imagery

Coordinate System:
 WGS 1984, UTM Zone 10 North



Figure 10-4
 Surface Geophysics Results A-A'

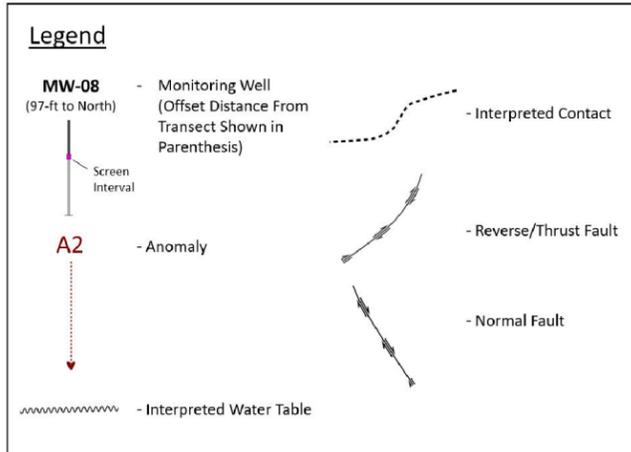
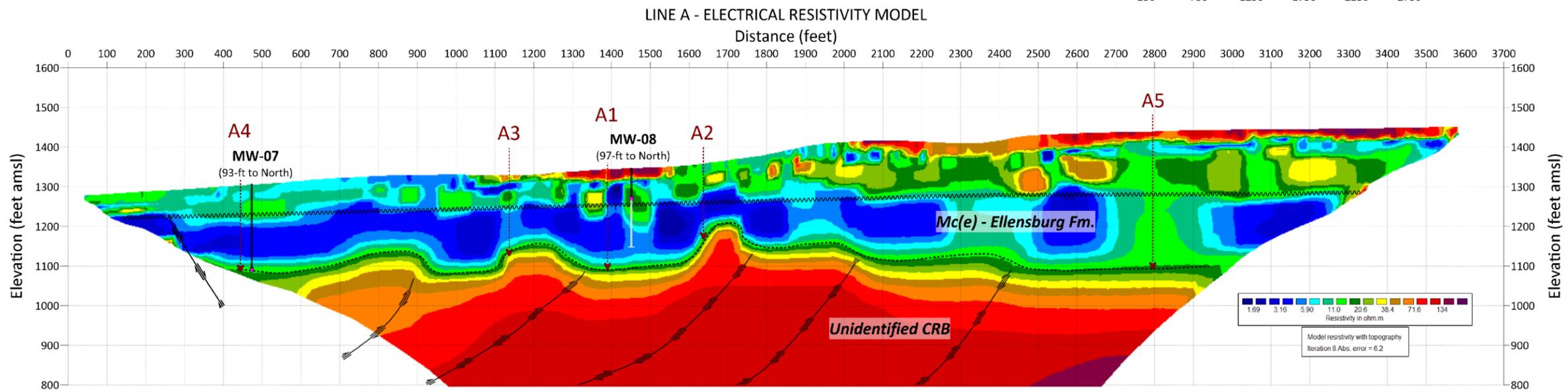
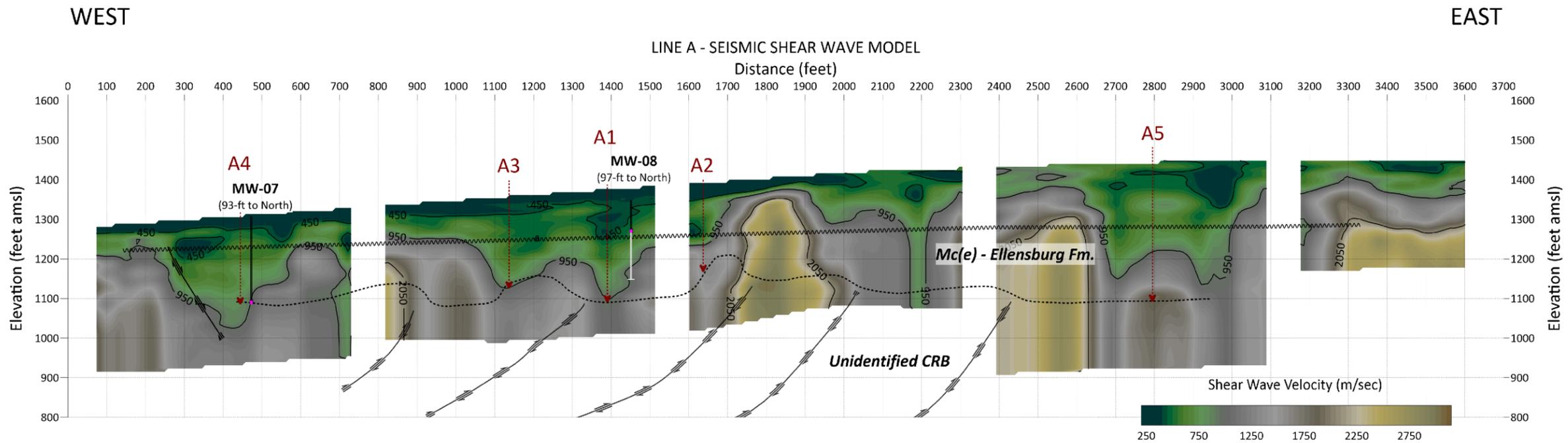




Figure 10-5
 Surface Geophysics Results B-B'

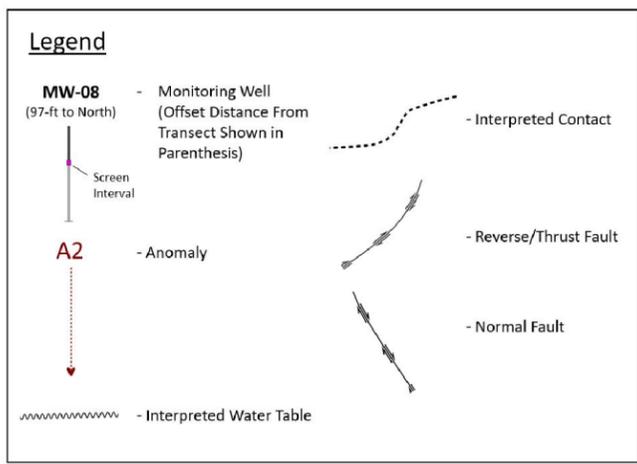
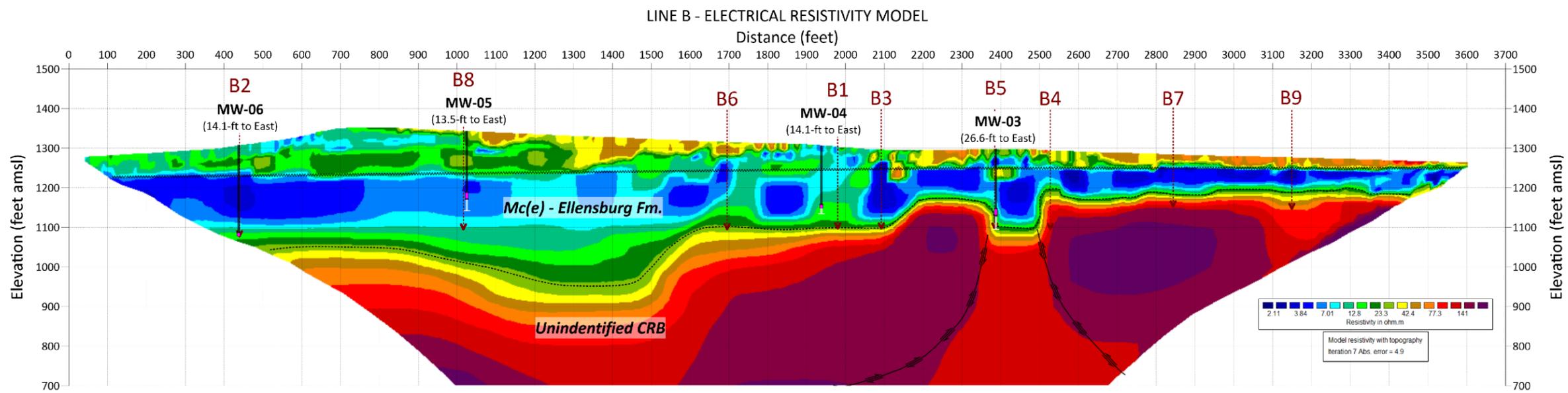
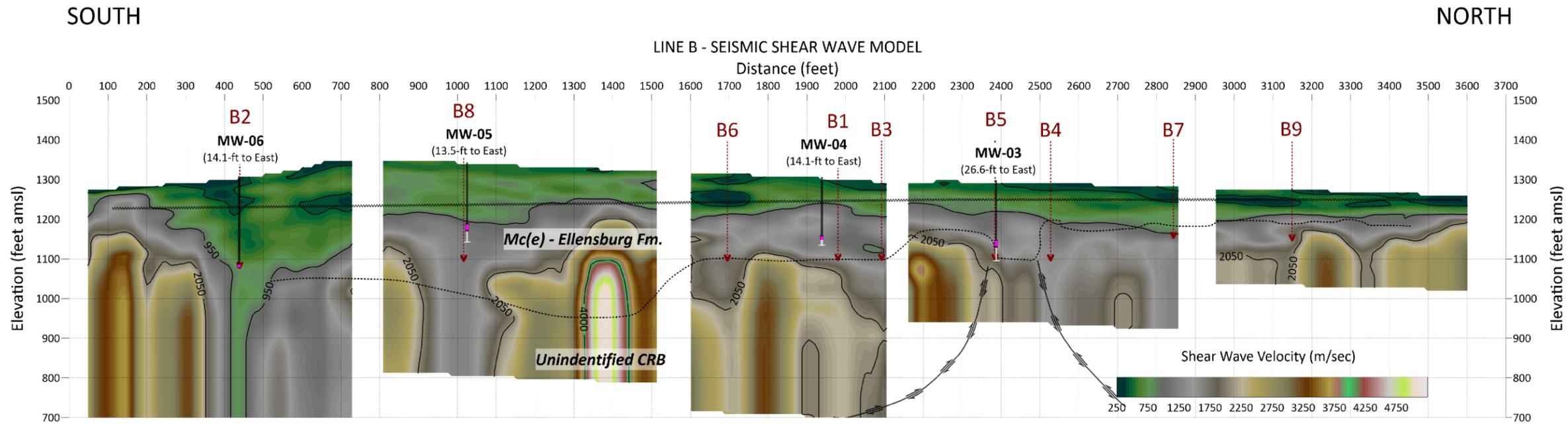
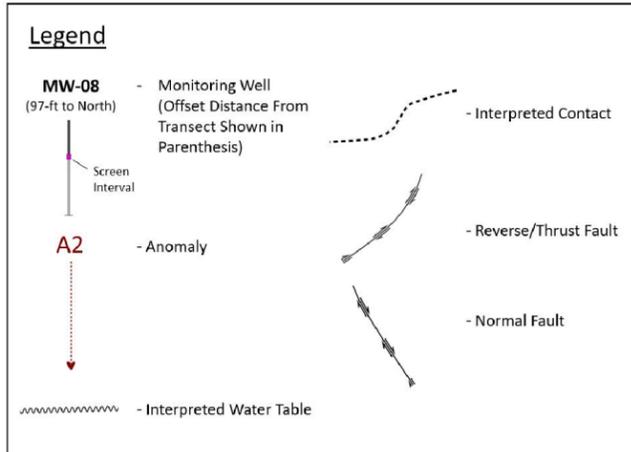
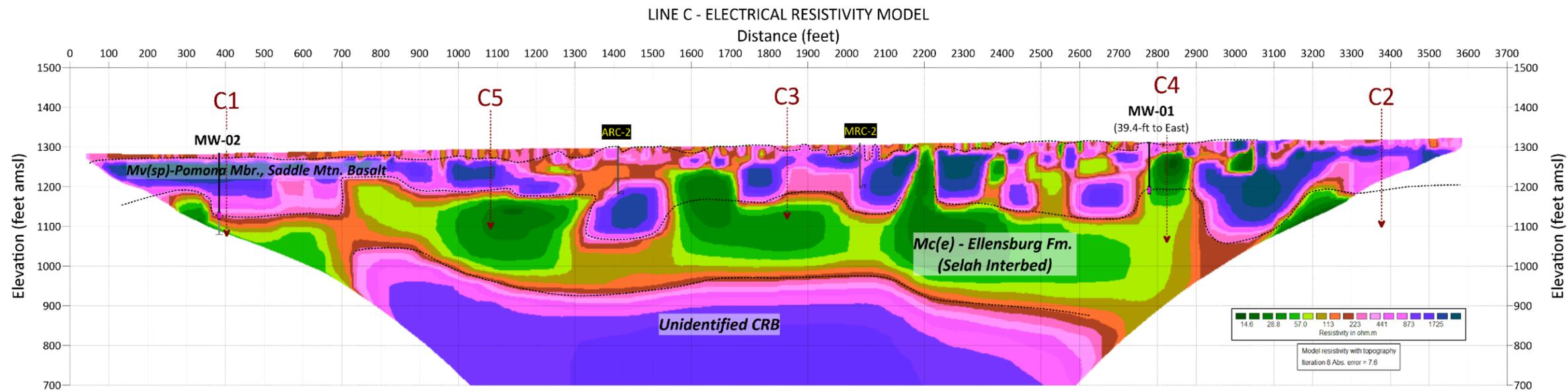
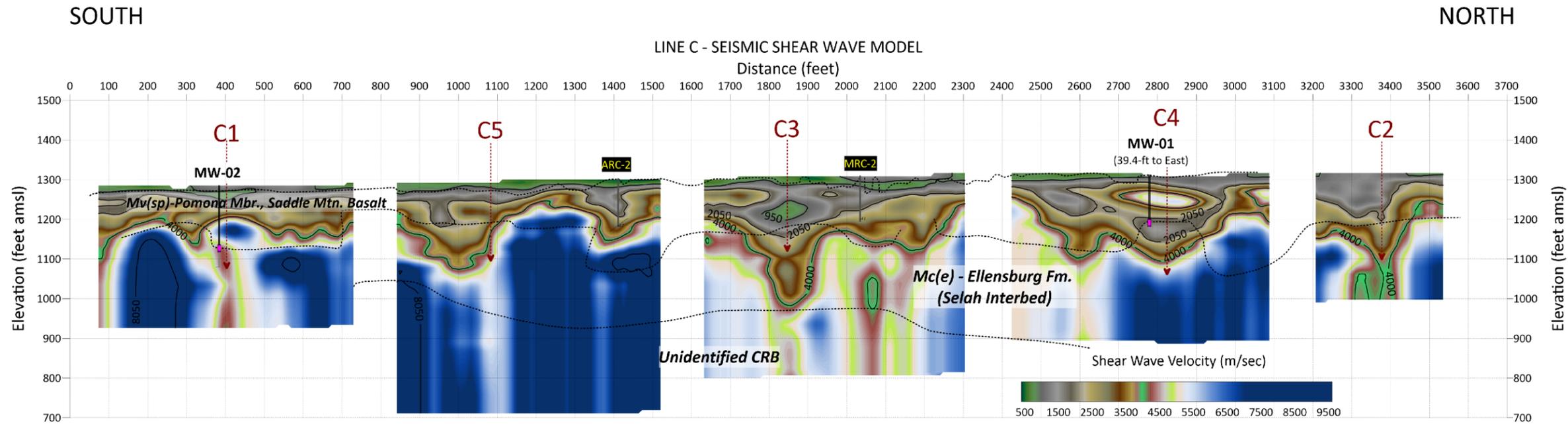




Figure 10-6
 Surface Geophysics Results C-C'

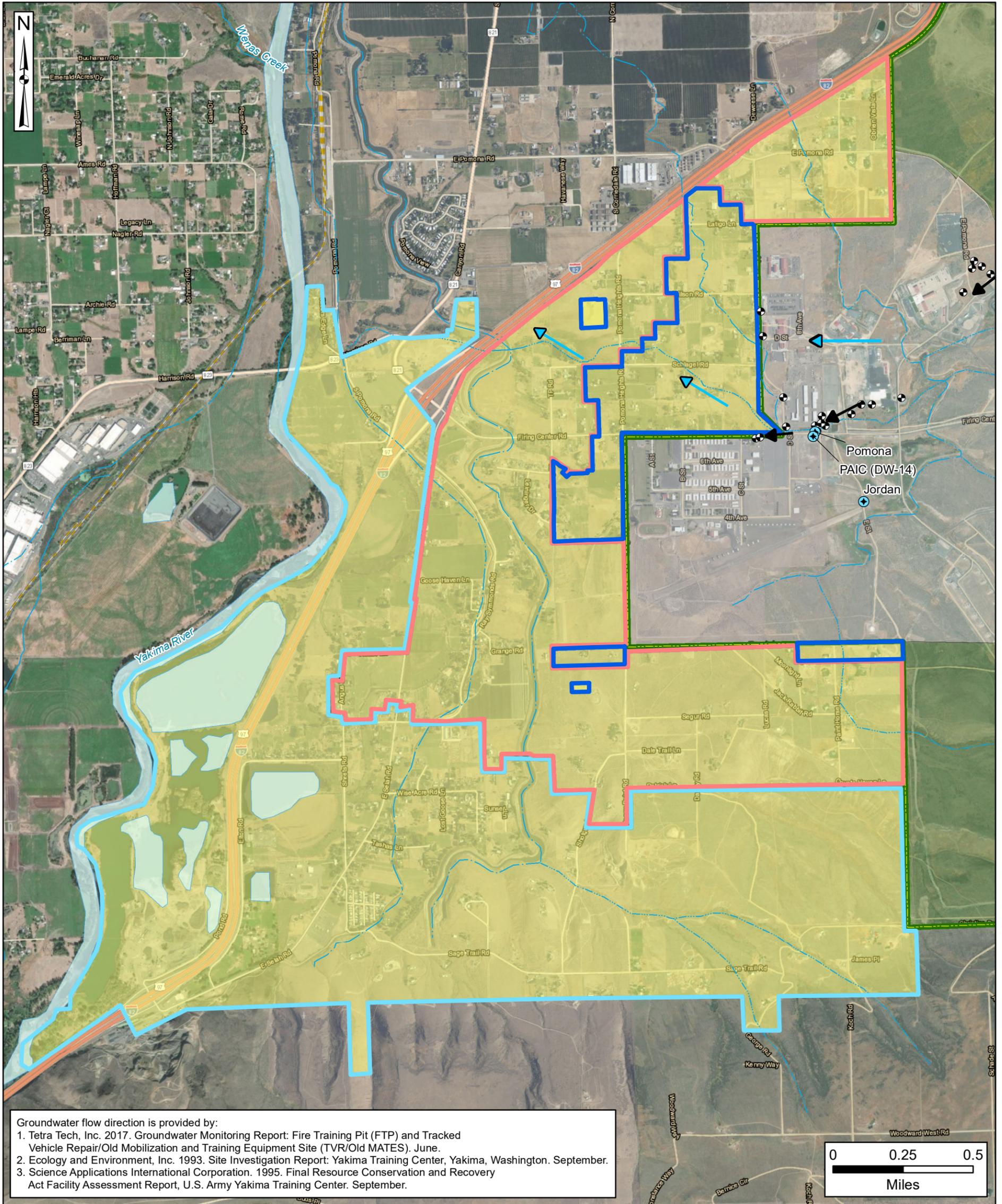
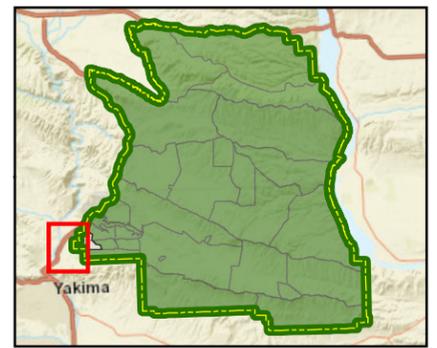




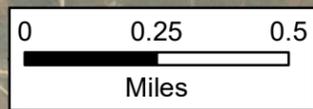
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Figure 10-7
 Off-Post Evaluation Area and
 Potable Supply Wells



Groundwater flow direction is provided by:
 1. Tetra Tech, Inc. 2017. Groundwater Monitoring Report: Fire Training Pit (FTP) and Tracked Vehicle Repair/Old Mobilization and Training Equipment Site (TVR/Old MATES). June.
 2. Ecology and Environment, Inc. 1993. Site Investigation Report: Yakima Training Center, Yakima, Washington. September.
 3. Science Applications International Corporation. 1995. Final Resource Conservation and Recovery Act Facility Assessment Report, U.S. Army Yakima Training Center. September.



- Installation Boundary
- Cantonment Area
- Range/Training Area
- Monitoring Well
- Potable Water Well (On-Installation)
- Water Body
- River/Stream (Perennial)
- River/Stream (Intermittent)
- Canal/Ditch
- Deep Groundwater (i.e., Used for Installation Potable Supply) Flow Direction
- Perched Groundwater Flow Direction (Inferred)
- Phase 1 Outreach Area (September 2021)*
- Phase 2 Outreach Area (January 2022)*
- Phase 3 Outreach Area (July/August 2022)*

*Undeveloped parcels and connections to Class A/B systems included.

Data Sources:
 Yakima Training Center, GIS Data, 2018
 ESRI ArcGIS Online, Aerial Imagery

Coordinate System:
 WGS 1984, UTM Zone 10 North



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Yakima Training Center, WA

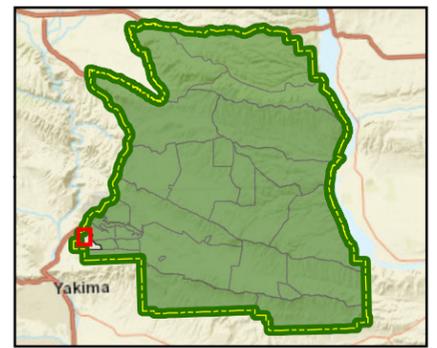
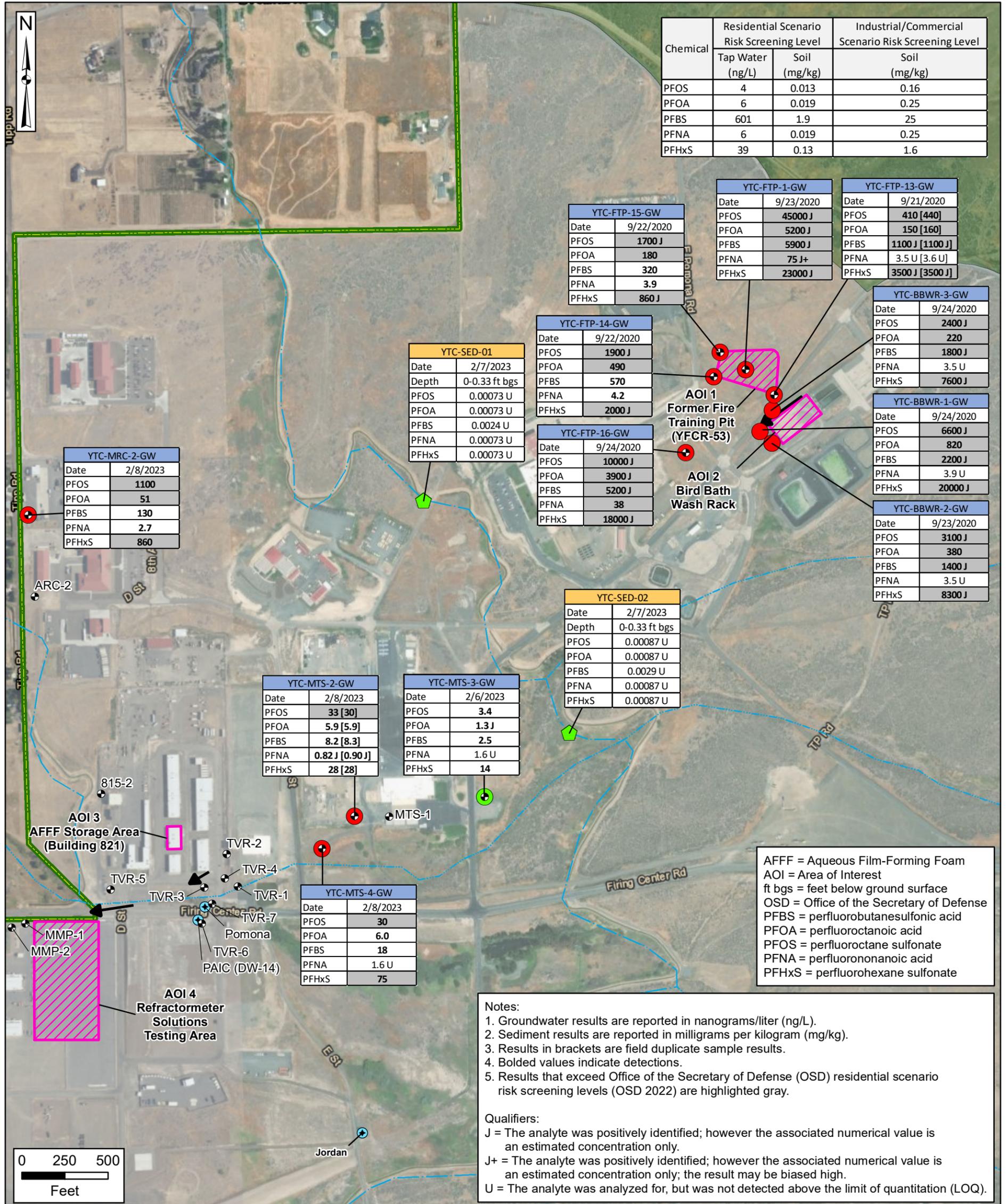


Figure 10-8
Former Fire Training Pit (YFCR-53) and Bird Bath Wash Rack AOI Historical Analytical Results



AFFF = Aqueous Film-Forming Foam
AOI = Area of Interest
ft bgs = feet below ground surface
OSD = Office of the Secretary of Defense
PFBS = perfluorobutanesulfonic acid
PFOA = perfluorooctanoic acid
PFOS = perfluorooctane sulfonate
PFNA = perfluorononanoic acid
PFHxS = perfluorohexane sulfonate

Notes:

- Groundwater results are reported in nanograms/liter (ng/L).
- Sediment results are reported in milligrams per kilogram (mg/kg).
- Results in brackets are field duplicate sample results.
- Bolded values indicate detections.
- Results that exceed Office of the Secretary of Defense (OSD) residential scenario risk screening levels (OSD 2022) are highlighted gray.

Qualifiers:

J = The analyte was positively identified; however the associated numerical value is an estimated concentration only.
J+ = The analyte was positively identified; however the associated numerical value is an estimated concentration only; the result may be biased high.
U = The analyte was analyzed for, but was not detected above the limit of quantitation (LOQ).

| | | |
|-----------------------|---|--|
| Installation Boundary | Monitoring Well | Historical Results |
| Cantonment Area | Potable Water Well (On-Installation) | Groundwater - 2022 OSD Risk Screening Level Exceedance |
| Range/Training Area | River/Stream (Intermittent) | Sediment - No Exceedances |
| AOI | Canal/Ditch | |
| AFFF Use Area | Perched Groundwater Flow Direction (Inferred) | |



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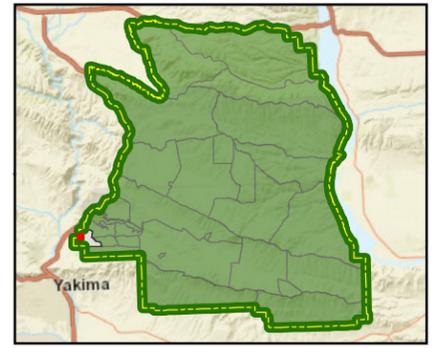
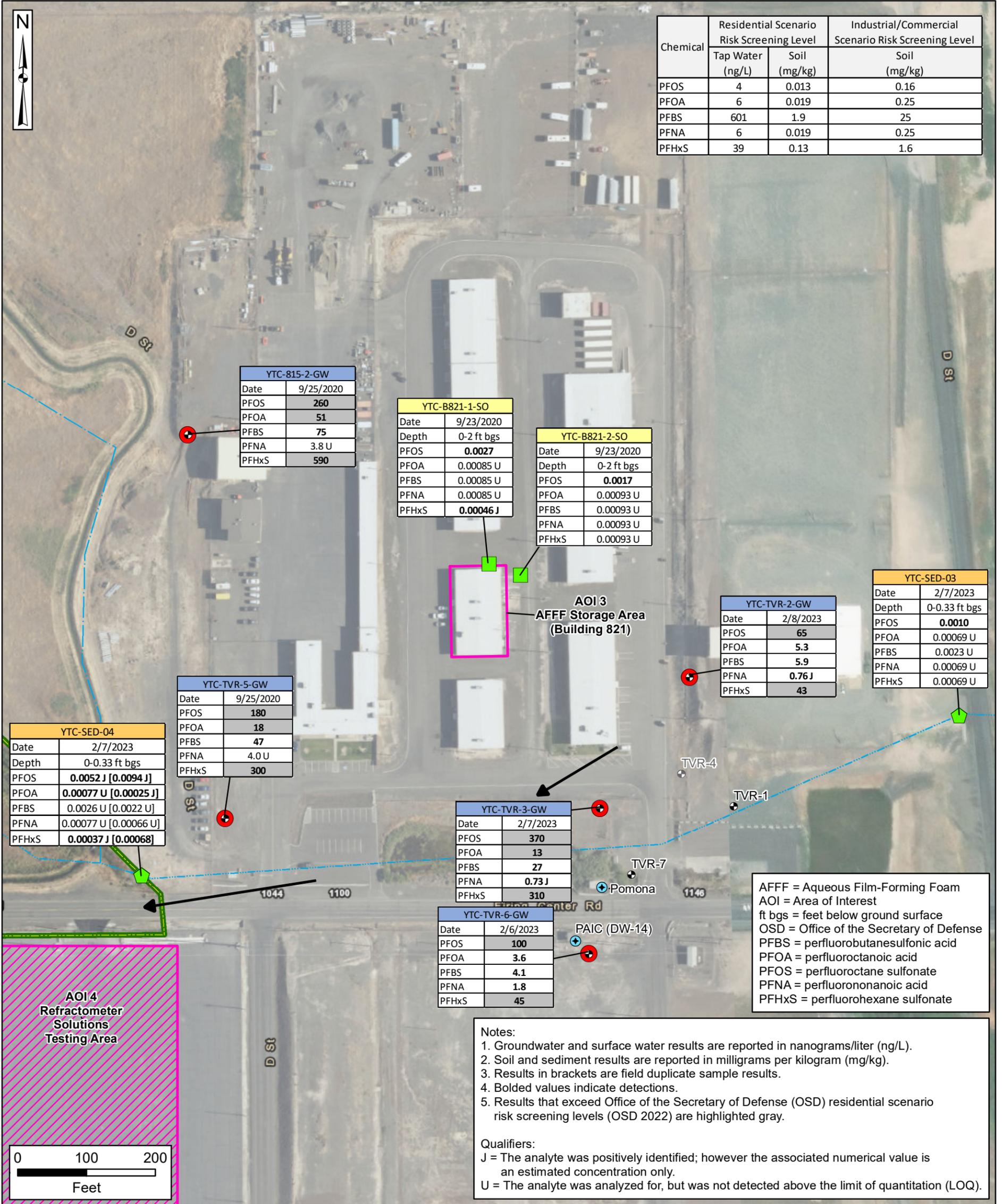


Figure 10-9
AFFF Storage Area (Building 821) AOI
Historical Analytical Results



- Installation Boundary
- Cantonment Area
- Range/Training Area
- AOI
- AFFF Use Area

- Monitoring Well
- Abandoned Monitoring Well Potable
- Water Well (On-Installation) River/
- Stream (Intermittent)
- Canal/Ditch
- Perched Groundwater Flow Direction (Inferred)

- Historical Results**
- Groundwater - 2022 OSD Risk Screening Level Exceedance
 - Soil - No Exceedances
 - Sediment - No Exceedances



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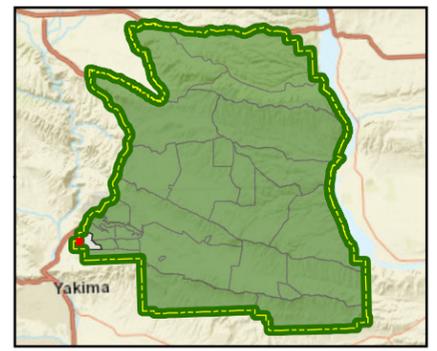
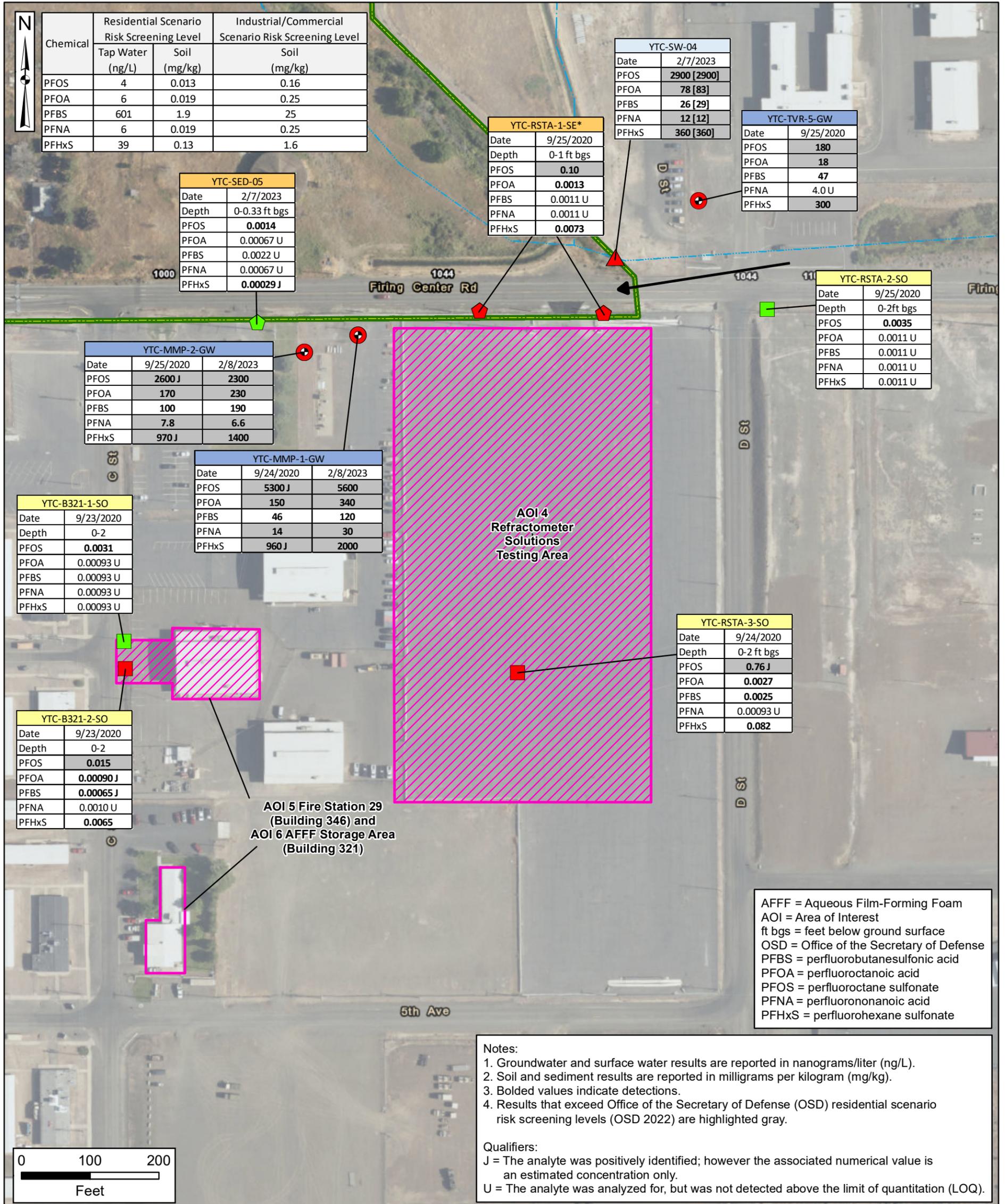


Figure 10-10
Refractometer Solutions Testing Area and Fire Station 29 (Building 346) and AFFF Storage Area (Building 321) AOI Historical Analytical Results



AFFF = Aqueous Film-Forming Foam
 AOI = Area of Interest
 ft bgs = feet below ground surface
 OSD = Office of the Secretary of Defense
 PFBS = perfluorobutanesulfonic acid
 PFOA = perfluorooctanoic acid
 PFOS = perfluorooctane sulfonate
 PFNA = perfluorononanoic acid
 PFHxS = perfluorohexane sulfonate

Notes:
 1. Groundwater and surface water results are reported in nanograms/liter (ng/L).
 2. Soil and sediment results are reported in milligrams per kilogram (mg/kg).
 3. Bolded values indicate detections.
 4. Results that exceed Office of the Secretary of Defense (OSD) residential scenario risk screening levels (OSD 2022) are highlighted gray.

Qualifiers:
 J = The analyte was positively identified; however the associated numerical value is an estimated concentration only.
 U = The analyte was analyzed for, but was not detected above the limit of quantitation (LOQ).

| | | |
|-----------------------------|--|---|
| Installation Boundary | Monitoring Well | Sediment - 2022 OSD Risk Screening Level Exceedance |
| Cantonment Area | Perched Groundwater Flow Direction (Inferred) | Soil - No Exceedances |
| Range/Training Area | Historical Results | Sediment - No Exceedances |
| AOI | Groundwater - 2022 OSD Risk Screening Level Exceedance | |
| AFFF Use Area | Surface Water - 2022 OSD Risk Screening Level Exceedance | |
| River/Stream (Intermittent) | Soil - 2022 OSD Risk Screening Level Exceedance | |
| Canal/Ditch | | |

* Sediment sample was collected as a multi-point composite of loose sediment along the ditch and shallow accumulations of soil at the edge of the asphalt pad.



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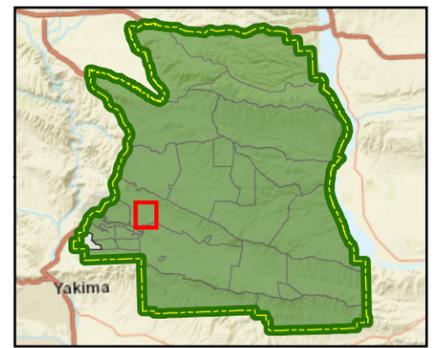
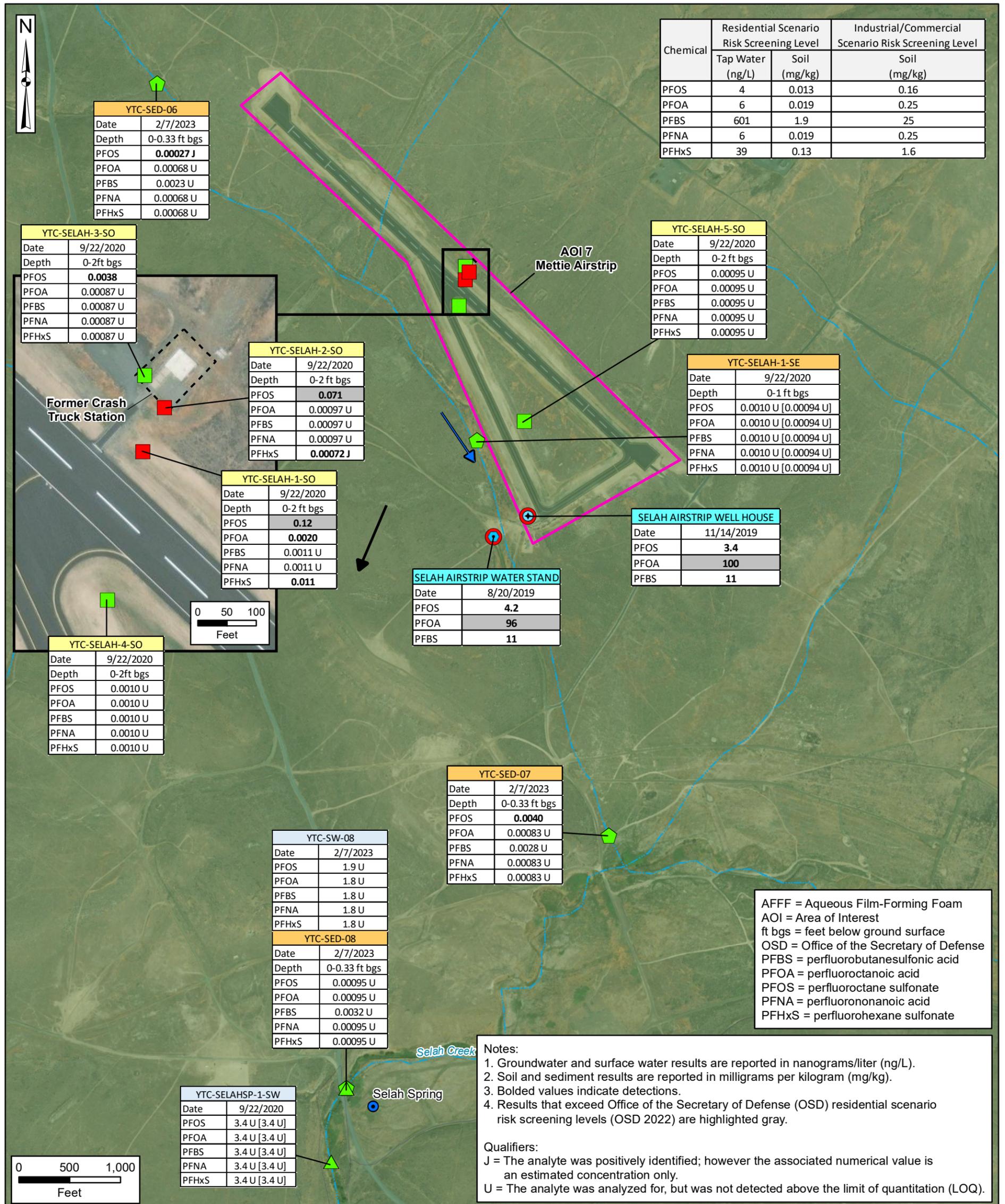


Figure 10-11
 Mettie Airstrip AOI
 Historical Analytical Results



- Installation Boundary
- Cantonment Area
- Range/Training Area
- AOI
- Former Building
- River/Stream (Intermittent)

- Canal/Ditch
- Perched Groundwater Flow Direction (Inferred)
- Surface Water Flow Direction
- Potable Water Well (On-Installation)
- Water Stand
- Spring

- Historical Results**
- Groundwater - 2022 OSD Risk Screening Level Exceedance
 - Soil - 2022 OSD Risk Screening Level Exceedance
 - Soil - No Exceedances
 - Sediment - No Exceedances
 - Surface Water - No Exceedances

Data Sources:
 Yakima Training Center, GIS Data, 2018
 ESRI ArcGIS Online, Aerial Imagery

Coordinate System:
 WGS 1984, UTM Zone 10 North



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Yakima Training Center, WA

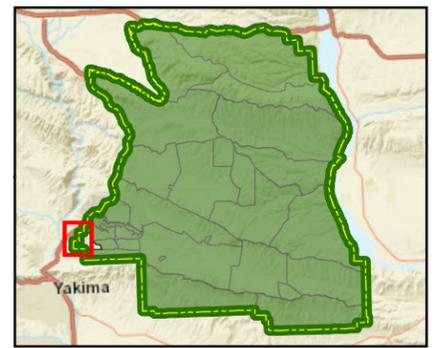
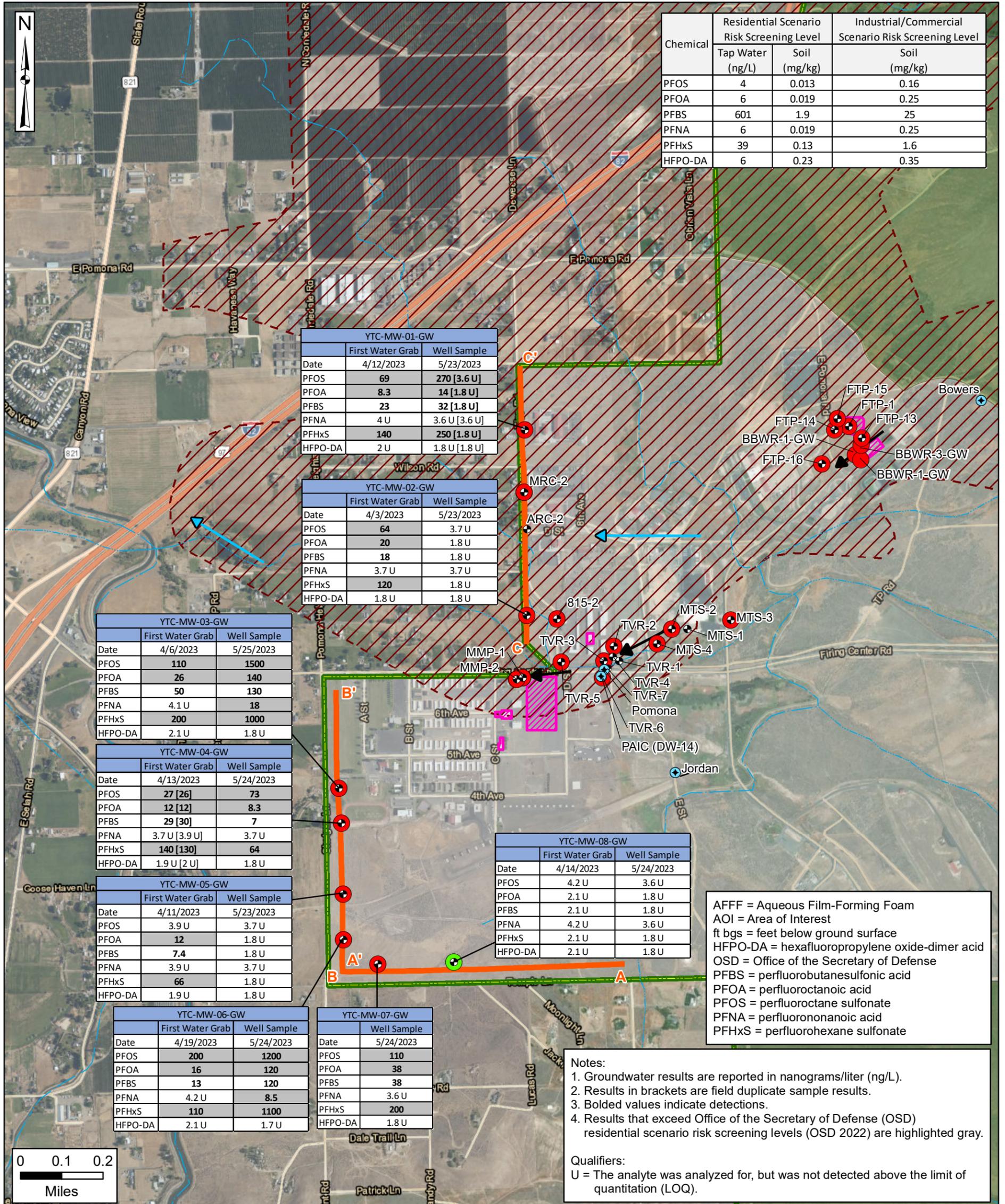


Figure 10-12
Boundary Well Groundwater Analytical Results



AFFF = Aqueous Film-Forming Foam
 AOI = Area of Interest
 ft bgs = feet below ground surface
 HFPO-DA = hexafluoropropylene oxide-dimer acid
 OSD = Office of the Secretary of Defense
 PFBS = perfluorobutanesulfonic acid
 PFOA = perfluorooctanoic acid
 PFOS = perfluorooctane sulfonate
 PFNA = perfluorononanoic acid
 PFHxS = perfluorohexane sulfonate

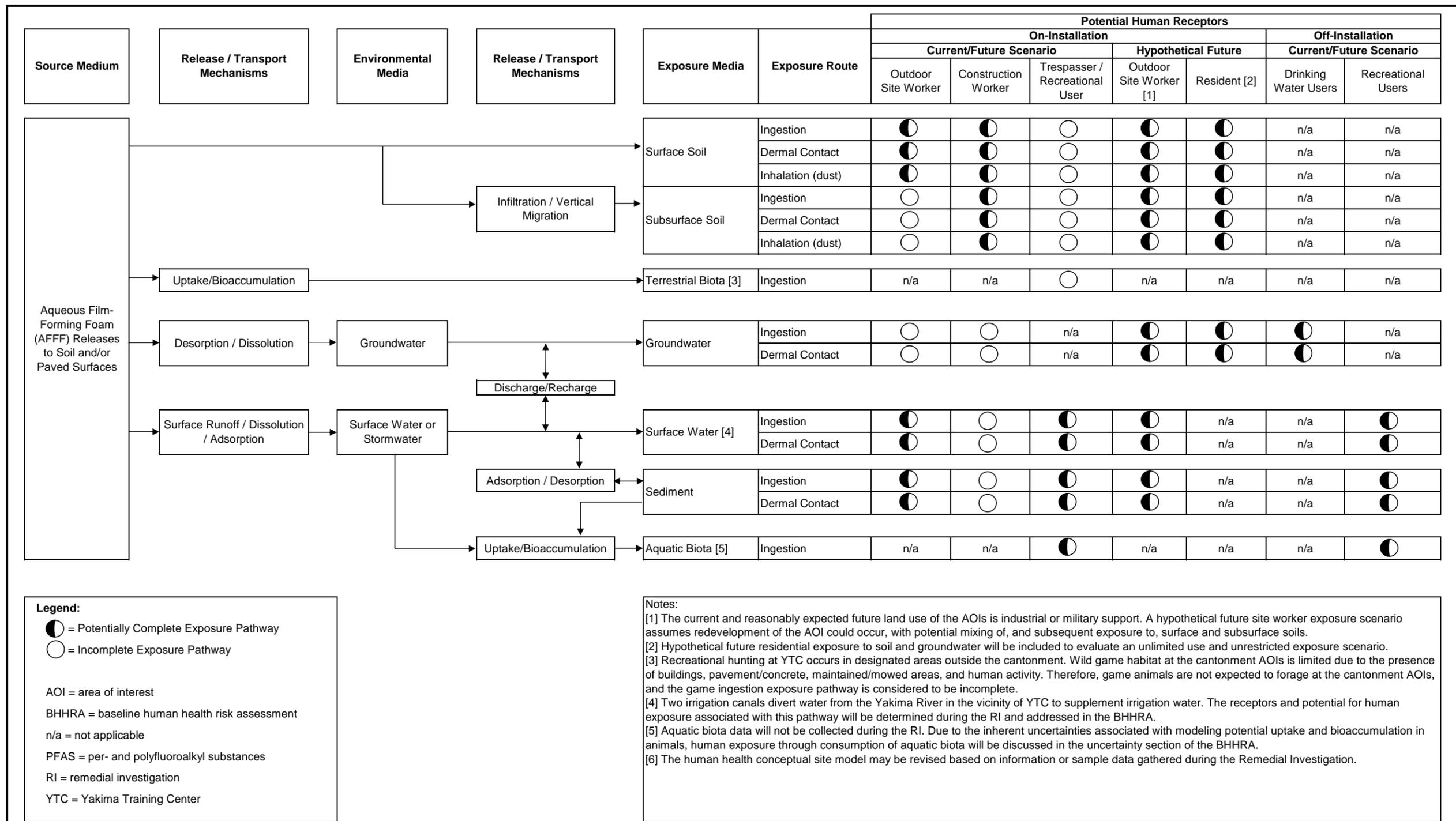
Notes:
 1. Groundwater results are reported in nanograms/liter (ng/L).
 2. Results in brackets are field duplicate sample results.
 3. Bolded values indicate detections.
 4. Results that exceed Office of the Secretary of Defense (OSD) residential scenario risk screening levels (OSD 2022) are highlighted gray.

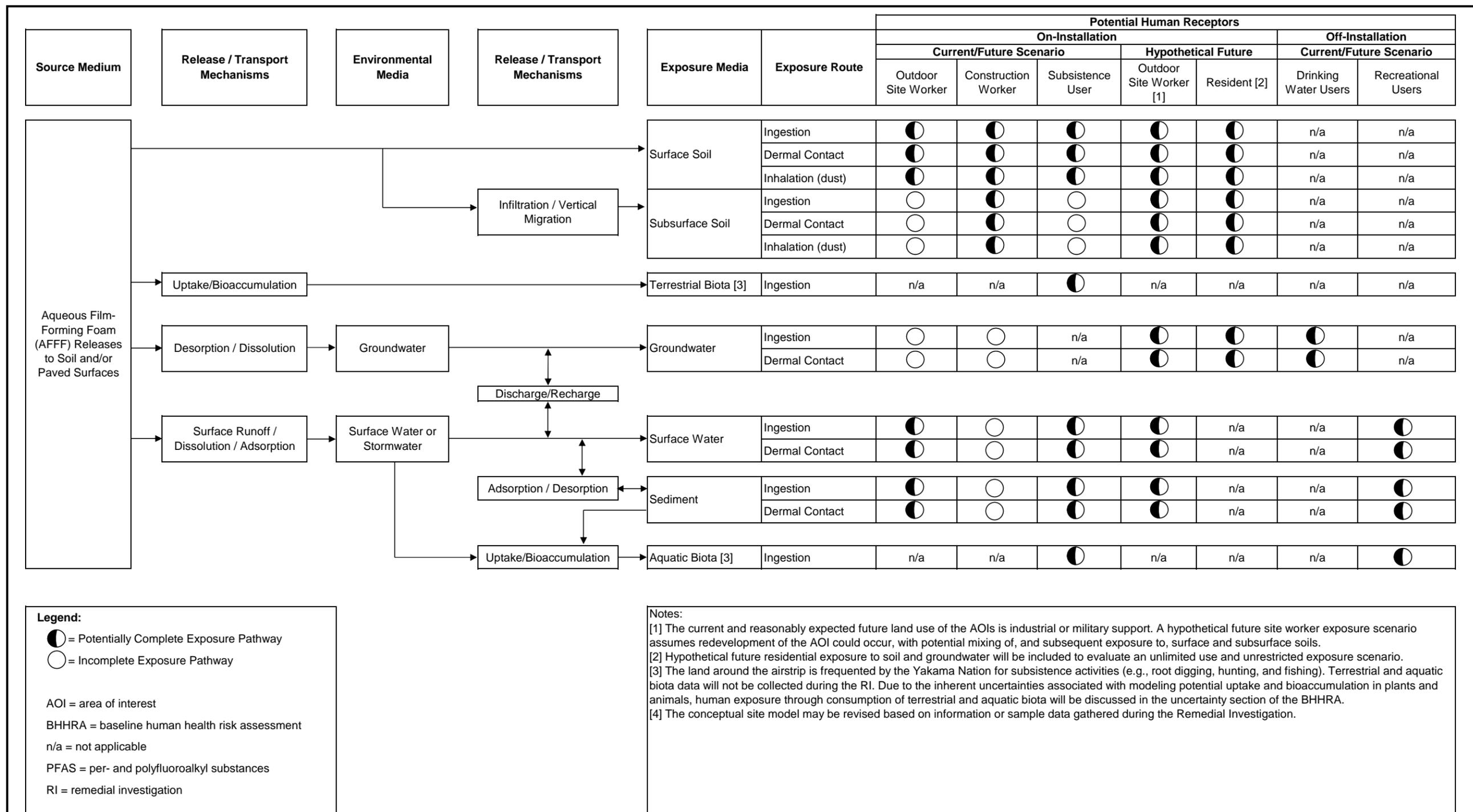
Qualifiers:
 U = The analyte was analyzed for, but was not detected above the limit of quantitation (LOQ).

| | | |
|-----------------------|---|--|
| Installation Boundary | Potable Water Well (On-Installation) | Deep Groundwater (i.e., Used for Installation Potable Supply) Flow Direction |
| Cantonment Area | River/Stream (Perennial) | Perched Groundwater Flow Direction |
| Range/Training Area | River/Stream (Intermittent) | Surface Geophysics Transect |
| AOI | Canal/Ditch | Analytical Results |
| AFFF Use Area | Approximate Limits of Pomona Basalt (dashed where inferred) | Groundwater - 2022 OSD Risk Screening Level Exceedance |
| Monitoring Well | | Groundwater - No Exceedances |

Data Sources:
 Yakima Training Center, GIS Data, 2018
 ESRI ArcGIS Online, Aerial Imagery

Coordinate System:
 WGS 1984, UTM Zone 10 North







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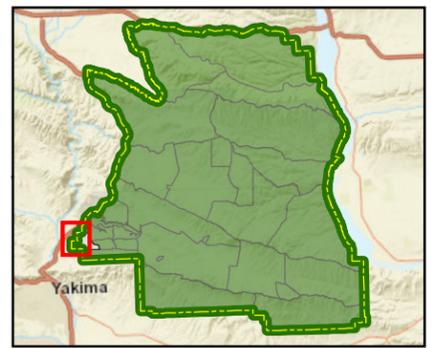
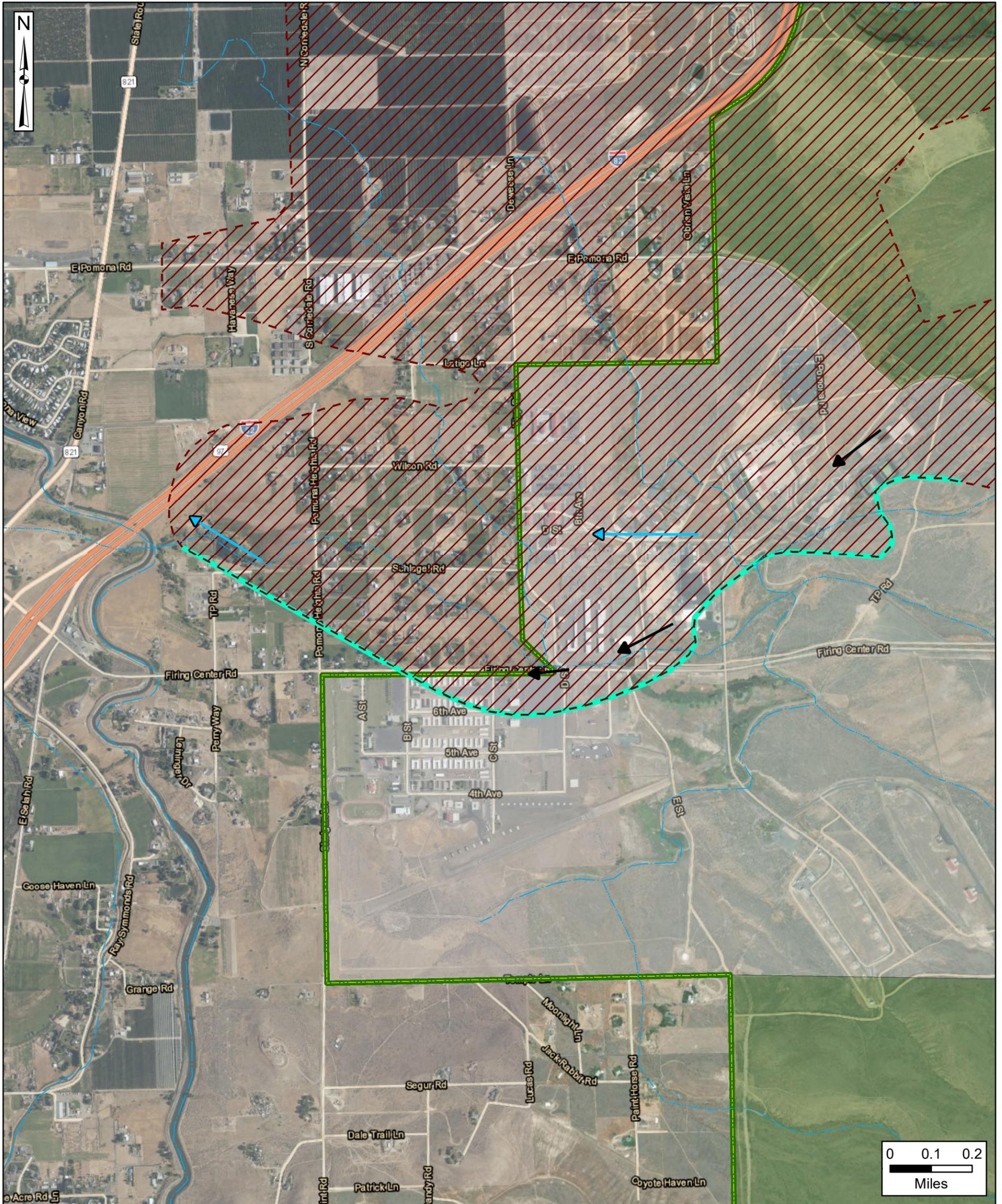


Figure 17-1
 Proposed EM-34 Geophysical Survey Area



- Installation Boundary
- Cantonment Area
- Range/Training Area
- AOI
- AFFF Use Area

- River/Stream (Perennial)
- River/Stream (Intermittent)
- Canal/Ditch
- Deep Groundwater (i.e., Used for Installation Potable Supply) Flow Direction (Inferred)
- Perched Groundwater Flow Direction (Inferred)

- Approximate Limits of Pomona Basalt (dashed where inferred)
- EM-34 Surface Resistivity Survey Focus
- AFFF = Aqueous Film-Forming Foam
- AOI = Area of Interest

Data Sources:
 Yakima Training Center, GIS Data, 2018
 ESRI ArcGIS Online, Aerial Imagery

Coordinate System:
 WGS 1984, UTM Zone 10 North



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 USAEC PFAS Remedial Investigation
 Yakima Training Center, WA

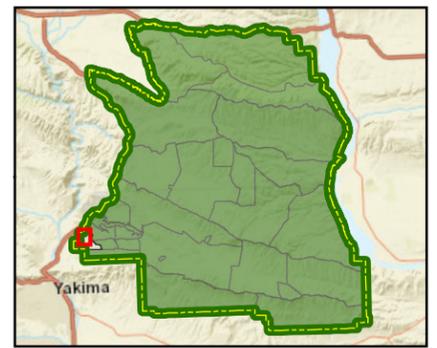
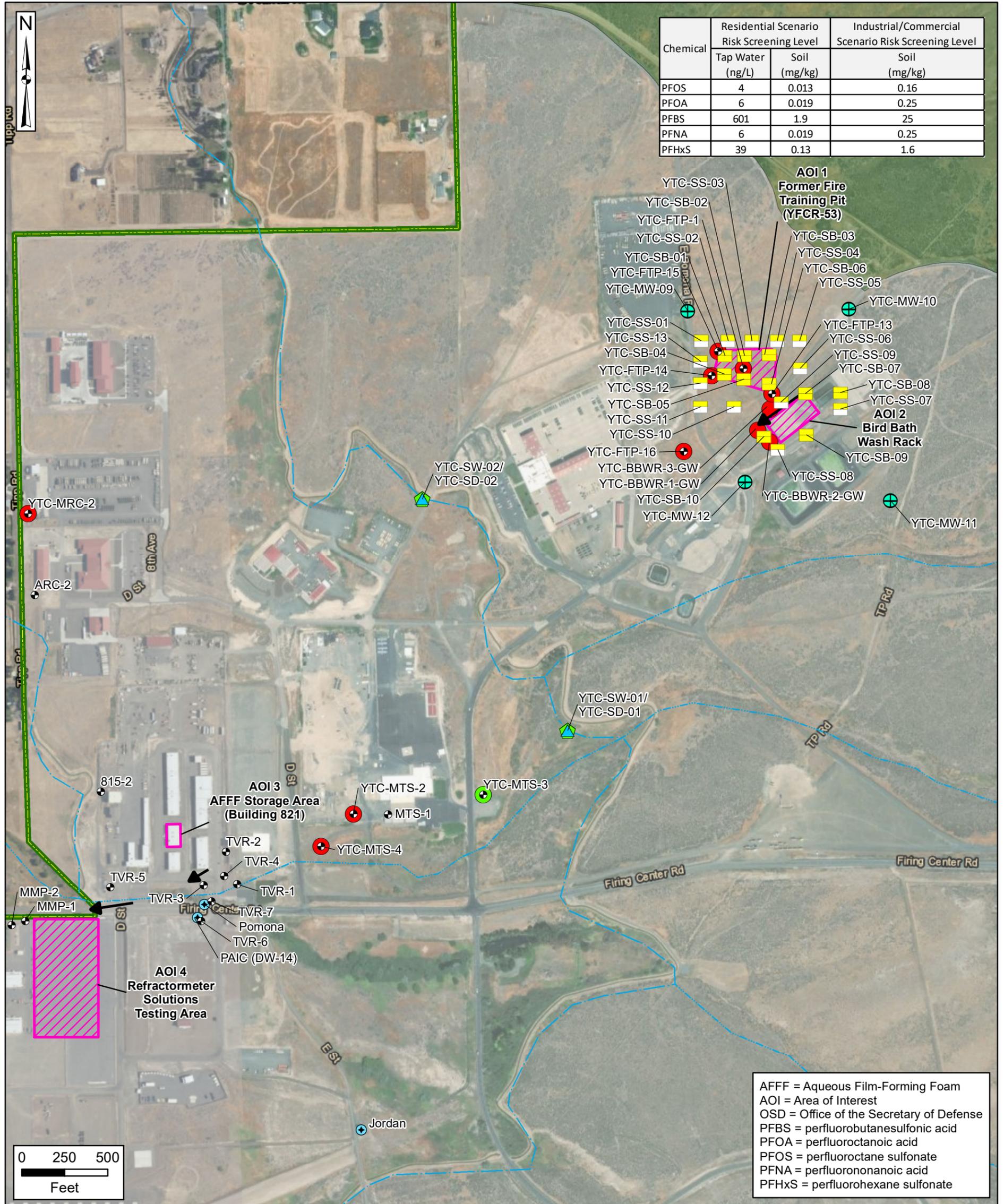


Figure 17-2
Former Fire Training Pit (YFCR-53) and Bird Bath Wash Rack AOI Prescriptive Sampling Locations



AFFF = Aqueous Film-Forming Foam
 AOI = Area of Interest
 OSD = Office of the Secretary of Defense
 PFBS = perfluorobutanesulfonic acid
 PFOA = perfluorooctanoic acid
 PFOS = perfluorooctane sulfonate
 PFNA = perfluorononanoic acid
 PFHxS = perfluorohexane sulfonate

| | | |
|-----------------------------|--|------------------------------------|
| Installation Boundary | Monitoring Well | Proposed Sampling Locations |
| Cantonment Area | Potable Water Well (On-Installation) | Surface Soil (Hand Auger) |
| Range/Training Area | Perched Groundwater Flow Direction (Inferred) | Surface & Deep Soil (Hand Auger) |
| AOI | Historical Results | Surface Water |
| AFFF Use Area | Groundwater - 2022 OSD Risk Screening Level Exceedance | New Shallow Monitoring Well |
| River/Stream (Intermittent) | Groundwater - No Exceedances | |
| Canal/Ditch | Sediment - No Exceedances | |

Data Sources:
 Yakima Training Center, GIS Data, 2018
 ESRI ArcGIS Online, Aerial Imagery
 Coordinate System:
 WGS 1984, UTM Zone 10 North



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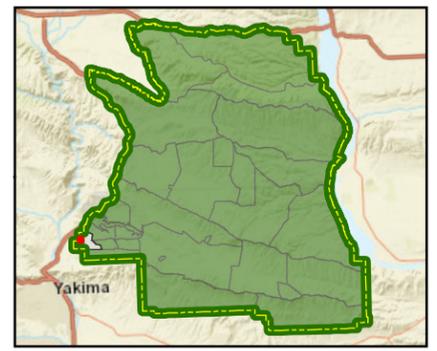
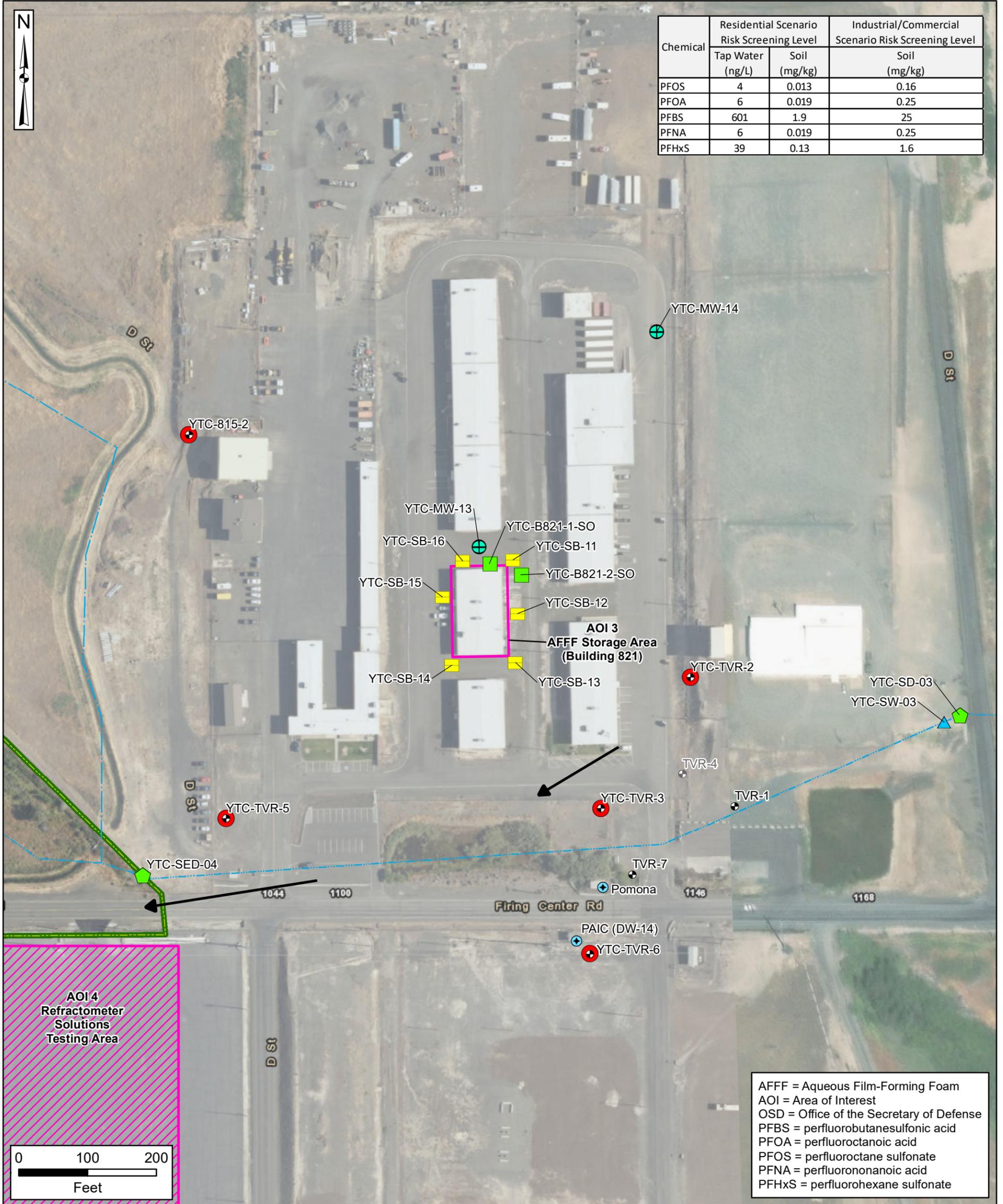


Figure 17-3
AFFF Storage Area (Building 821) AOI
Prescriptive Sampling Locations



| Chemical | Residential Scenario Risk Screening Level | | Industrial/Commercial Scenario Risk Screening Level |
|----------|---|--------------|---|
| | Tap Water (ng/L) | Soil (mg/kg) | Soil (mg/kg) |
| PFOS | 4 | 0.013 | 0.16 |
| PFOA | 6 | 0.019 | 0.25 |
| PFBS | 601 | 1.9 | 25 |
| PFNA | 6 | 0.019 | 0.25 |
| PFHxS | 39 | 0.13 | 1.6 |

AFFF = Aqueous Film-Forming Foam
 AOI = Area of Interest
 OSD = Office of the Secretary of Defense
 PFBS = perfluorobutanesulfonic acid
 PFOA = perfluorooctanoic acid
 PFOS = perfluorooctane sulfonate
 PFNA = perfluorononanoic acid
 PFHxS = perfluorohexane sulfonate

- Installation Boundary
- Cantonment Area
- Range/Training Area
- AOI
- AFFF Use Area
- River/Stream (Intermittent)
- Canal/Ditch
- Monitoring Well
- Abandoned Monitoring Well
- Potable Water Well (On-Installation)
- Perched Groundwater Flow Direction (Inferred)
- Historical Results**
- Groundwater - 2022 OSD Risk Screening Level Exceedance
- Soil - No Exceedances
- Sediment - No Exceedances
- Proposed Sampling Locations**
- Surface & Deep Soil (Hand Auger)
- Surface Water
- New Shallow Monitoring Well

Data Sources:
 Yakima Training Center, GIS Data, 2018
 ESRI ArcGIS Online, Aerial Imagery
 Coordinate System:
 WGS 1984, UTM Zone 10 North



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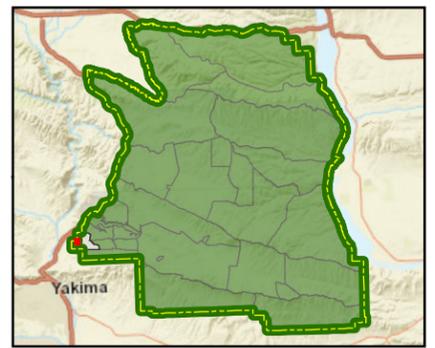
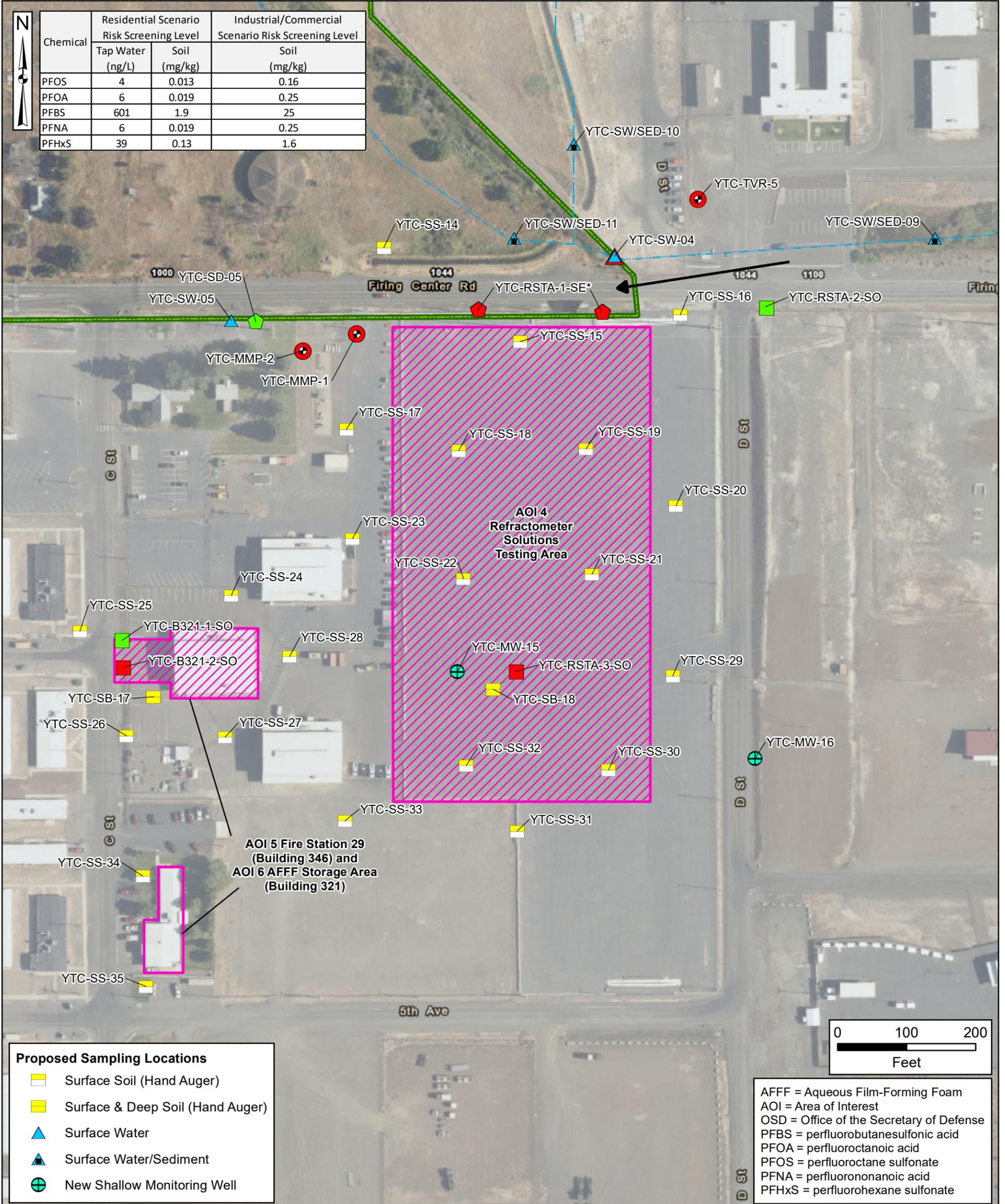


Figure 17-4
Refractometer Solutions Testing Area and Fire Station 29 (Building 346) and AFFF Storage Area (Building 321) AOI Prescriptive Sampling Locations



Proposed Sampling Locations

- Surface Soil (Hand Auger)
- Surface & Deep Soil (Hand Auger)
- Surface Water
- Surface Water/Sediment
- New Shallow Monitoring Well

Historical Results

- Groundwater - 2022 OSD Risk Screening Level Exceedance
- Surface Water - 2022 OSD Risk Screening Level Exceedance
- Soil - 2022 OSD Risk Screening Level Exceedance
- Sediment - 2022 OSD Risk Screening Level Exceedance
- Soil - No Exceedances
- Sediment - No Exceedances

Legend

- Installation Boundary
- Cantonment Area
- Range/Training Area
- AOI
- AFFF Use Area
- River/Stream (Intermittent)
- Canal/Ditch
- Monitoring Well
- Perched Groundwater Flow Direction (Inferred)

Definitions:
 AFFF = Aqueous Film-Forming Foam
 AOI = Area of Interest
 OSD = Office of the Secretary of Defense
 PFBS = perfluorobutanesulfonic acid
 PFOA = perfluorooctanoic acid
 PFOS = perfluorooctane sulfonate
 PFNA = perfluorononanoic acid
 PFHxS = perfluorohexane sulfonate

* Sediment sample was collected as a multi-point composite of loose sediment along the ditch and shallow accumulations of soil at the edge of the asphalt pad.

Data Sources:
 Yakima Training Center, GIS Data, 2018
 ESRI ArcGIS Online, Aerial Imagery

Coordinate System:
 WGS 1984, UTM Zone 10 North



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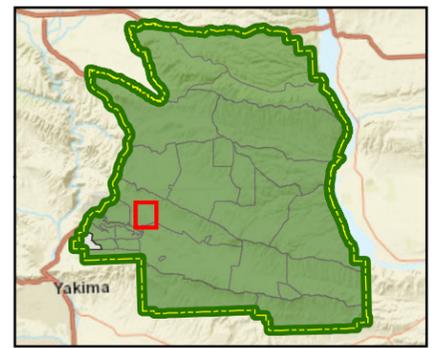
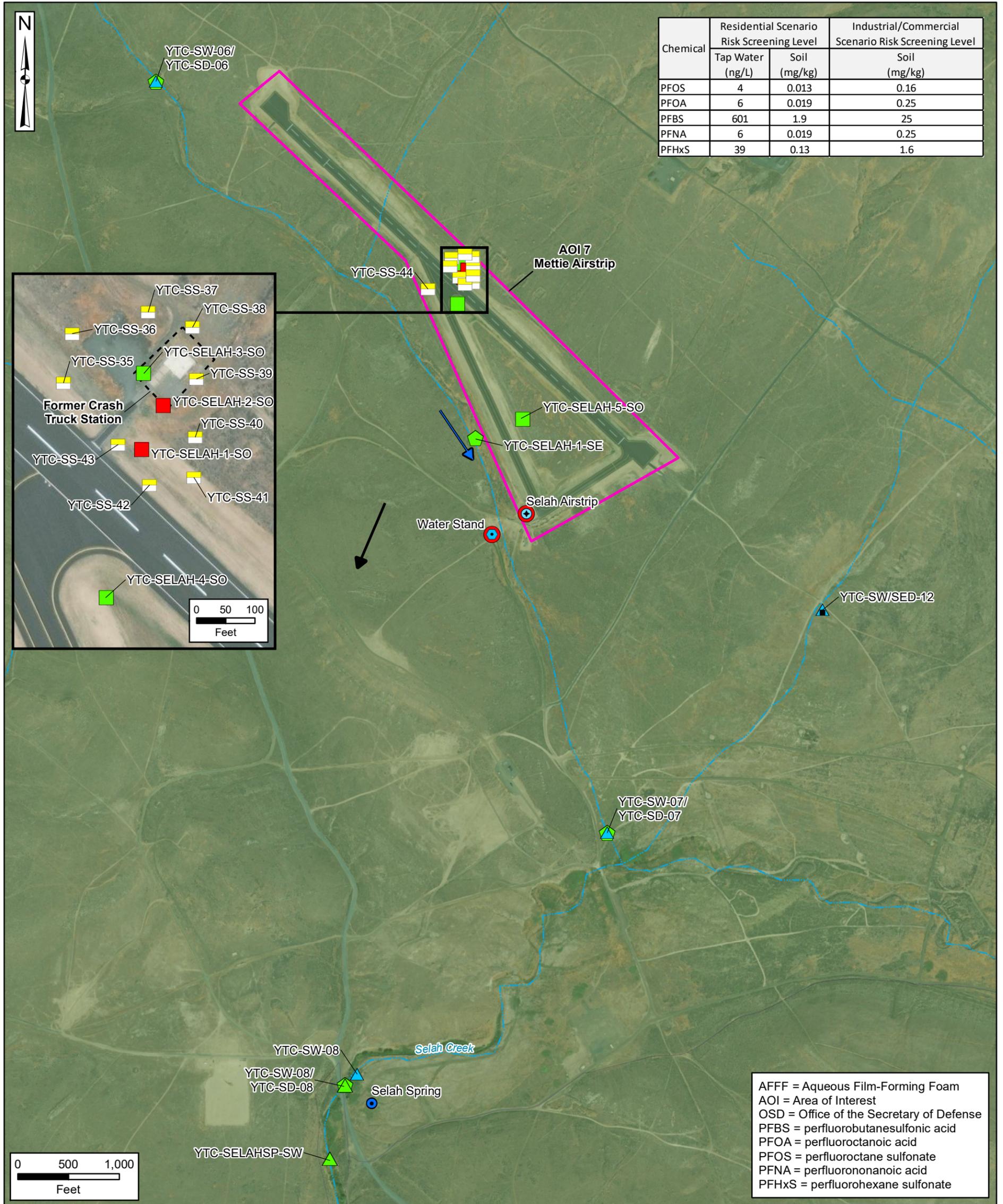


Figure 17-5
Mettie Airstrip AOI
Prescriptive Sampling Locations



| Chemical | Residential Scenario Risk Screening Level | | Industrial/Commercial Scenario Risk Screening Level |
|----------|---|--------------|---|
| | Tap Water (ng/L) | Soil (mg/kg) | Soil (mg/kg) |
| PFOS | 4 | 0.013 | 0.16 |
| PFOA | 6 | 0.019 | 0.25 |
| PFBS | 601 | 1.9 | 25 |
| PFNA | 6 | 0.019 | 0.25 |
| PFHxS | 39 | 0.13 | 1.6 |



AFFF = Aqueous Film-Forming Foam
 AOI = Area of Interest
 OSD = Office of the Secretary of Defense
 PFBS = perfluorobutanesulfonic acid
 PFOA = perfluorooctanoic acid
 PFOS = perfluorooctane sulfonate
 PFNA = perfluorononanoic acid
 PFHxS = perfluorohexane sulfonate

| | | | |
|-----------------------------|---|--|---------------------------|
| Installation Boundary | Perched Groundwater Flow Direction (Inferred) | Historical Results | Surface Soil (Hand Auger) |
| Cantonment Area | Surface Water Flow Direction | Groundwater - 2022 OSD Risk Screening Level Exceedance | Surface Water |
| Range/Training Area | Potable Water Well (On-Installation) | Soil - 2022 OSD Risk Screening Level Exceedance | Surface Water/Sediment |
| AOPI | Water Stand | Soil - No Exceedances | |
| Former Building | Spring | Sediment - No Exceedances | |
| River/Stream (Intermittent) | | Surface Water - No Exceedances | |
| Canal/Ditch | | | |

Data Sources:
 Yakima Training Center, GIS Data, 2018
 ESRI ArcGIS Online, Aerial Imagery
 Coordinate System:
 WGS 1984, UTM Zone 10 North

TABLES

Table 10-1 - On-Post Potable Water Wells Construction Details
PFAS RI QAPP
Yakima Training Center
Yakima, Washington



| Well Identification | Building Location | Basin | Easting | Northing | Casing Depth (ft bgs) | Total Depth (ft bgs) | Approximate Static Water Level (ft bgs) | Notes |
|------------------------------|-------------------|----------|----------------------------|------------------------------|--------------------------------|----------------------|---|---|
| Pomona | P-0829 | Yakima | 694871.1 | 5172244.4 | 353 | 407 | Artesian | Wells provide water for the cantonment area drinking water distribution system. |
| Jordan | P-0551 (P-0550) | Yakima | 695149.8 | 5171842.6 | 365 | 617 | 53 | |
| Bowers | P-0860 | Yakima | 696344.7 | 5173298.5 | 241 | 541 | 162 | |
| YRS | P-1721 | Yakima | 701889.5 | 5172928.0 | 307 | 602 | 324 | Well provides water for YRS drinking water distribution system. |
| MPRC | P-U084B | Columbia | 717131.4 | 5185002.5 | 1300 | 1311 | 1005 | Well provides water for MPRC drinking water distribution system. |
| Badger Pocket/ Badger Gap | P-U084E | Yakima | 708607.6875 707116.5087 | 5192417.0000 5192944.1759 | 490 | 510 | 175 | No additional notes. |
| Dead Truck Farm | P-0020 | Yakima | 707154.0 | 5203249.0 | 40 | 150 | 53 | Wells are pumped and treated as needed (i.e., to supply troops during training exercises); wells are not connected to main cantonment drinking water distribution system. |
| Doris | P-3002 | Columbia | 728443.7 | 5194962.1 | 580 | 580 | 435 | |
| Exit 11 | P-2239 | Yakima | 701394.6 | 5188940.0 | 200 | 580 | 289 | |
| North Filey Road | P-0010 | Columbia | 730923.3 | 5167831.9 | 40 | 950 | 533 | |
| Range 19 | P-2229 | Yakima | 706162.7 | 5184387.0 | 135 | 425 | 93 | |
| Range 55 | P-2555 | Yakima | 718105.8 | 5168615.1 | 105 | 135 | 72 | |
| Hester | NA | Yakima | 703211.0 | 5172606.5 | 315 | 585 | 244 | |
| Range Control | P-1804 | Yakima | 703217.7 | 5172521.4 | 281 | 302 | 266 | |
| Selah Airstrip | P-2060 | Yakima | 704259.8 | 5176188.0 | 73 | 91 | 47 | |
| PAIC | P-0840 | Yakima | 694859.4 | 5172220.4 | (NA - similar to Pomona well*) | NA | NA | |

Acronyms:

- bgs – below ground surface
- ft – feet
- MPRC – Multi-Purpose Range Complex
- NA - not available
- PAIC – Pamona Artesian Irrigation Company
- YRS – Yakima Research Station

Sources:

Data table: Yakima Training Center. 2003. Yakima Training Center Well Data. June.
 *Construction details not provided on source table noted above. Additional information regarding PAIC well construction is as provided by the United States Army Corps of Engineers in the 2012 Periodic Review Report, Yakima Training Center, Yakima, Washington.

Table 10-2 - On-Post Monitoring Well Construction Details
PFAS RI QAPP
Yakima Training Center
Yakima, Washington



| Well ID | Elevation at TOC (ft AMSL) | Ground Surface Elevation (ft AMSL) | Easting UTM (m) | Northing UTM (m) | Total Depth (ft) | Screen Interval (ft bgs) |
|---|----------------------------|------------------------------------|-----------------|------------------|------------------|--------------------------|
| Fire Training Pit Monitoring Wells | | | | | | |
| FTP-1 | 1,467.72 | 1,464.59 | 695828.3 | 5173198.0 | 21 | 8 – 18 |
| FTP-13 | 1,473.07 | 1,470.96 | 695878.5 | 5173153.0 | 25 | 10 – 20 |
| FTP-14 | 1,457.48 | 1,455.35 | 695771.4 | 5173185.2 | 22 | 12 – 22 |
| FTP-15 | 1,460.88 | 1,458.72 | 695783.1 | 5173228.9 | 20 | 10 – 20 |
| FTP-16 | 1,444.81 | 1,442.68 | 695722.0 | 5173050.7 | 30 | 20 – 30 |
| TVR/Old Mates Monitoring Wells | | | | | | |
| 815-2 | 1,304.28 | 1,301.86 | 694687.7 | 5172445.5 | 132 | 115 – 130 |
| MMP-1 | 1,301.37 | 1,298.39 | 694553.4 | 5172215.3 | 101 | 88 – 98 |
| MMP-2 | 1,301.31 | 1,298.55 | 694529.6 | 5172207.9 | 76 | 64 – 74 |
| MRC-2 | 1,312.11 | 1,309.64 | 694558.9 | 5172939.9 | 114 | 101 – 111 |
| MTS-1 | 1,361.02 | 1,359.05 | 695196.9 | 5172404.6 | 127 | 115 – 125 |
| MTS-2 | 1,351.88 | 1,348.79 | 695135.9 | 5172405.4 | 113 | 101 – 111 |
| MTS-3 | 1,362.36 | 1,362.62 | 695366.1 | 5172439.6 | 72 | 62 – 72 |
| MTS-4 | 1,331.88 | 1,332.14 | 695078.6 | 5172347.7 | 97 | 82 – 97 |
| TVR-1 | 1,320.17 | 1,317.32 | 694936.0 | 5172286.6 | 105 | 93 – 103 |
| TVR-2 | 1,317.56 | 1,314.18 | 694910.0 | 5172337.7 | 95 | 83 – 93 |
| TVR-3 | 1,310.60 | 1,310.86 | 694872.9 | 5172282.5 | 158 | 143 – 158 |
| TVR-5 | 1,302.04 | 1,299.42 | 694704.2 | 5172275.0 | 142 | 132 – 142 |
| TVR-6 | 1,310.06 | 1,310.30 | 694866.4 | 5172214.0 | 139 | 139 – 149 |
| TVR-7 | 1,310.95 | 1,311.63 | 694882.5 | 5172255.6 | 140 | 140 – 150 |
| Boundary Monitoring Wells | | | | | | |
| MW-01 | 1,322.95 | 1,320.60 | 694561.3 | 5173185.3 | 140 | 120-140 |
| MW-02 | 1,292.26 | 1,289.70 | 694569.6 | 5172458.2 | 173 | 153-173 |
| MW-03 | 1,307.66 | 1,305.00 | 693835.3 | 5171781.3 | 177 | 157-177 |
| MW-04 | 1,315.69 | 1,314.30 | 693844.8 | 5171644.8 | 166 | 156-166 |
| MW-05 | 1,354.49 | 1,351.80 | 693851.2 | 5171367.2 | 183 | 163-183 |
| MW-06 | 1,315.81 | 1,313.40 | 693852.8 | 5171189.7 | 236 | 226-236 |
| MW-07 | 1,323.93 | 1,321.20 | 693987.1 | 5171092.7 | 235 | 225-235 |
| MW-08 | 1,360.67 | 1,358.00 | 694284.6 | 5171100.3 | 92 | 82-92 |

Abbreviations and Acronyms:

- ft – feet
- ft AMSL – feet above mean sea level
- ft bgs – feet below ground surface
- ID – identification
- m – meter
- Old MATES – Old Mobilization and Training Equipment Site
- TOC – top-of-casing
- TVR – Tracked Vehicle Repair
- UTM – Universal Transverse Mercator

| AOPI Location Sample/Duplicate ID Sample Date Sample Type Matrix | | | | YTC-MMP-1-GW | | YTC-MMP-2-GW | | YTC-MRC-2-GW | | YTC-MTS-2-GW | | | | YTC-MTS-3-GW | |
|---|-------------|-----|------|---------------------------------|-----|---------------------------------|-------|---------------------------------|------|---------------------------------|------|--|------|---------------------------------|------|
| | | | | YTC-MMP-1-GW-020823 | | YTC-MMP-2-GW-020823 | | YTC-MRC-2-GW-020823 | | YTC-MTS-2-GW-020823 | | YTC-MTS-2-GW-020823 / YTC-FD-01-GW-020823 | | YTC-MTS-3-GW-020623 | |
| | | | | 02/08/2023 N Ground Water | | 02/08/2023 N Ground Water | | 02/08/2023 N Ground Water | | 02/08/2023 N Ground Water | | 02/08/2023 FD Ground Water | | 02/06/2023 N Ground Water | |
| | | | | Analyte | CAS | OSD Tapwater | Units | Result | Qual | Result | Qual | Result | Qual | Result | Qual |
| PFAS | | | | | | | | | | | | | | | |
| 11-chloroeicosfluoro-3-oxaundecane-1-sulfonic acid (F-53B Minor) | 763051-92-9 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| 2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA) | 13252-13-6 | 6 | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorodecanoic acid (7:3 FTCA) | 812-70-4 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorohexanoic acid | 356-02-5 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorooctanoic acid (5:3 FTCA) | 914637-49-3 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| 4,8-Dioxa-3H-perfluorononanoic acid (DONA) | 919005-14-4 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| 4:2 Fluorotelomer sulfonate (4:2 FTS) | 757124-72-4 | -- | ng/L | 2.4 | | 0.74 | J | 1.6 | U | 1.6 | U | 1.6 | U | 1.6 | U |
| 6:2 Fluorotelomer sulfonic acid (6:2 FTSA) | 27619-97-2 | -- | ng/L | 1300 | J | 630 | | 3.7 | | 2.4 | U | 2.4 | U | 2.5 | U |
| 8:2 Fluorotelomer sulfonic acid (8:2 FTSA) | 39108-34-4 | -- | ng/L | 300 | | 110 | | 5.4 | | 2.4 | U | 2.4 | U | 2.5 | U |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9CL-PF3ONS) | 756426-58-1 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamide (N-EtFOSA) | 4151-50-2 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamide ethanol (N-EtFOSE) | 1691-99-2 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA) | 2991-50-6 | -- | ng/L | 2.5 | U | 2.6 | U | 2.4 | U | 2.4 | U | 2.4 | U | 2.5 | U |
| N-Methyl perfluorooctane sulfonamide (N-MeFOSA) | 31506-32-8 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| N-Methyl perfluorooctane sulfonamidoethanol (N-MeFOSE) | 24448-09-7 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| N-Methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) | 2355-31-9 | -- | ng/L | 1.7 | U | 1.7 | U | 1.6 | U | 1.6 | U | 1.6 | U | 1.6 | U |
| Nonafluoro-3,6-Dioxaheptonic Acid (NFDHA) | 151772-58-6 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluoro (2-Ethoxyethane) Sulfonic Acid (PFEEESA) | 113507-82-7 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluoro-3-methoxypropanoic aci (PFMPA) | 377-73-1 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluoro-4-Methoxybutanic acid (PFMBA) | 863090-89-5 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluorobutane sulfonic acid (PFBS) | 375-73-5 | 601 | ng/L | 120 | | 190 | | 130 | | 8.2 | | 8.3 | | 2.5 | |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | -- | ng/L | 140 | | 110 | | 53 | | 7.5 | | 7.4 | | 4.1 | U |
| Perfluorodecane sulfonic acid (PFDS) | 335-77-3 | -- | ng/L | 0.91 | J | 1.7 | U | 1.6 | U | 1.6 | U | 1.6 | U | 1.6 | U |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | -- | ng/L | 4.6 | | 1.3 | J | 0.55 | J | 1.6 | U | 1.6 | U | 1.6 | U |
| Perfluorododecane sulfonic acid (PFDOS) | 79780-39-5 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | -- | ng/L | 1.7 | U | 1.7 | U | 1.6 | U | 1.6 | U | 1.6 | U | 1.6 | U |
| Perfluoroheptane sulfonic acid (PFHpS) | 375-92-8 | -- | ng/L | 120 | | 56 | | 25 | | 0.76 | J | 0.67 | J | 1.6 | U |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | -- | ng/L | 360 | | 180 | | 39 | | 5.6 | | 5.5 | | 0.50 | J |
| Perfluorohexane sulfonic acid (PFHxS) | 355-46-4 | 39 | ng/L | 2000 | | 1400 | | 860 | | 28 | | 28 | | 14 | |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | -- | ng/L | 520 | | 530 | | 220 | | 15 | | 15 | | 1.5 | J |
| Perfluorononane sulfonic acid (PFNS) | 68259-12-1 | -- | ng/L | 1.7 | U | 1.7 | U | 2.4 | | 1.6 | U | 1.6 | U | 1.6 | U |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 6 | ng/L | 30 | | 6.6 | | 2.7 | | 0.82 | J | 0.90 | J | 1.6 | U |
| Perfluorooctane sulfonamide (PFOSA) | 754-91-6 | -- | ng/L | 18 | | 11 | | 2.9 | | 1.6 | U | 1.6 | U | 1.6 | U |
| Perfluorooctane sulfonic acid (PFOS) | 1763-23-1 | 4 | ng/L | 5600 | | 2300 | | 1100 | | 33 | | 30 | | 3.4 | |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 6 | ng/L | 340 | | 230 | | 51 | | 5.9 | | 5.9 | | 1.3 | J |
| Perfluoropentane sulfonic acid (PFPeSA) | 2706-91-4 | -- | ng/L | 180 | | 210 | | 150 | | 6.5 | | 6.5 | | 2.0 | |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | -- | ng/L | 450 | | 360 | | 120 | | 13 | | 13 | | 0.99 | J |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | -- | ng/L | 1.7 | U | 1.7 | U | 1.6 | U | 1.6 | U | 1.6 | U | 1.6 | U |
| Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 | -- | ng/L | 1.7 | U | 1.7 | U | 1.6 | U | 1.6 | U | 1.6 | U | 1.6 | U |
| Perfluoroundecanoic acid (PFUdA) | 2058-94-8 | -- | ng/L | 1.7 | U | 1.7 | U | 1.6 | U | 1.6 | U | 1.6 | U | 1.6 | U |

Table 10-3 - Groundwater PFAS SI, Baseline, and Boundary Investigation Analytical Results
 PFAS RI QAPP
 Yakima Training Center
 Yakima, Washington



| AOPI Location Sample/Duplicate ID Sample Date Sample Type Matrix | | | | YTC-MTS-4-GW | | | | | | | | | | | |
|---|-------------|--------------|-------|---------------------------------|------|---------------------------------|------|---------------------------------|------|---------------------------------|------|---------------------------------|------|---|------|
| | | | | YTC-MTS-4-GW | | YTC-MW-01-GW | | | | YTC-MW-02-GW | | | | | |
| | | | | YTC-MTS-4-GW-020823 | | YTC-MW-01-GW-041223 | | YTC-MW-01-GW-052323 | | YTC-MW-02-GW-040323 | | YTC-MW-02-GW-052323 | | YTC-MW-02-GW-052323 / YTC-FD-02-GW-052323 | |
| | | | | 02/08/2023 N Ground Water | | 04/12/2023 N Ground Water | | 05/23/2023 N Ground Water | | 04/03/2023 N Ground Water | | 05/23/2023 N Ground Water | | 05/23/2023 FD Ground Water | |
| Analyte | CAS | OSD Tapwater | Units | Result | Qual | Result | Qual |
| PFAS | | | | | | | | | | | | | | | |
| 11-chloroeicosfluoro-3-oxaundecane-1-sulfonic acid (F-53B Minor) | 763051-92-9 | -- | ng/L | -- | | 2 | U | 1.8 | U | 1.8 | U | 1.8 | U | 1.8 | U |
| 2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA) | 13252-13-6 | 6 | ng/L | -- | | 2 | U | 1.8 | U | 1.8 | U | 1.8 | U | 1.8 | U |
| 2H, 2H, 3H, 3H-perfluorodecanoic acid (7:3 FTCA) | 812-70-4 | -- | ng/L | -- | | 100 | U | 89 | U | 92 | U | 92 | U | 90 | U |
| 2H, 2H, 3H, 3H-perfluorohexanoic acid | 356-02-5 | -- | ng/L | -- | | 20 | U | 18 | U | 18 | U | 18 | U | 18 | U |
| 2H, 2H, 3H, 3H-perfluorooctanoic acid (5:3 FTCA) | 914637-49-3 | -- | ng/L | -- | | 100 | U | 89 | U | 92 | U | 92 | U | 90 | U |
| 4,8-Dioxa-3H-perfluorononanoic acid (DONA) | 919005-14-4 | -- | ng/L | -- | | 2 | U | 1.8 | U | 1.8 | U | 1.8 | U | 1.8 | U |
| 4:2 Fluorotelomer sulfonate (4:2 FTS) | 757124-72-4 | -- | ng/L | 1.6 | U | 4 | U | 3.6 | U | 3.7 | U | 3.7 | U | 3.6 | U |
| 6:2 Fluorotelomer sulfonic acid (6:2 FTSA) | 27619-97-2 | -- | ng/L | 2.4 | U | 4 | U | 3.6 | U | 3.7 | U | 3.7 | U | 3.6 | U |
| 8:2 Fluorotelomer sulfonic acid (8:2 FTSA) | 39108-34-4 | -- | ng/L | 2.4 | U | 5 | U | 4.5 | U | 4.6 | U | 4.6 | U | 4.5 | U |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9CL-PF3ONS) | 756426-58-1 | -- | ng/L | -- | | 4 | U | 3.6 | U | 3.7 | U | 3.7 | U | 3.6 | U |
| N-Ethyl perfluorooctane sulfonamide (N-EtFOSA) | 4151-50-2 | -- | ng/L | -- | | 5 | U | 4.5 | U | 4.6 | U | 4.6 | U | 4.5 | U |
| N-Ethyl perfluorooctane sulfonamide ethanol (N-EtFOSE) | 1691-99-2 | -- | ng/L | -- | | 50 | U | 45 | U | 46 | U | 46 | U | 45 | U |
| N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA) | 2991-50-6 | -- | ng/L | 2.4 | U | 4 | U | 3.6 | U | 3.7 | U | 3.7 | U | 3.6 | U |
| N-Methyl perfluorooctane sulfonamide (N-MeFOSA) | 31506-32-8 | -- | ng/L | -- | | 5 | U | 4.5 | U | 4.6 | U | 4.6 | U | 4.5 | U |
| N-Methyl perfluorooctane sulfonamidoethanol (N-MeFOSE) | 24448-09-7 | -- | ng/L | -- | | 50 | U | 45 | U | 46 | U | 46 | U | 45 | U |
| N-Methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) | 2355-31-9 | -- | ng/L | 1.6 | U | 4 | U | 3.6 | U | 3.7 | U | 3.7 | U | 3.6 | U |
| Nonafluoro-3,6-Dioxahexanoic Acid (NFDHA) | 151772-58-6 | -- | ng/L | -- | | 5 | U | 4.5 | U | 4.6 | U | 4.6 | U | 4.5 | U |
| Perfluoro (2-Ethoxyethane) Sulfonic Acid (PFEEESA) | 113507-82-7 | -- | ng/L | -- | | 2 | U | 1.8 | U | 1.8 | U | 1.8 | U | 1.8 | U |
| Perfluoro-3-methoxypropanoic acid (PFMPA) | 377-73-1 | -- | ng/L | -- | | 2 | U | 1.8 | U | 1.8 | U | 1.8 | U | 1.8 | U |
| Perfluoro-4-Methoxybutanoic acid (PFMBA) | 863090-89-5 | -- | ng/L | -- | | 2 | U | 1.8 | U | 1.8 | U | 1.8 | U | 1.8 | U |
| Perfluorobutane sulfonic acid (PFBS) | 375-73-5 | 601 | ng/L | 18 | | 23 | | 32 | | 18 | | 1.8 | U | 1.8 | U |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | -- | ng/L | 7.5 | | 11 | | 9.4 | | 8.4 | | 1.8 | U | 1.8 | U |
| Perfluorodecane sulfonic acid (PFDS) | 335-77-3 | -- | ng/L | 1.6 | U | 4 | U | 3.6 | U | 3.7 | U | 3.7 | U | 3.6 | U |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | -- | ng/L | 1.6 | U | 2 | U | 1.8 | U | 1.8 | U | 1.8 | U | 1.8 | U |
| Perfluorododecane sulfonic acid (PFDOS) | 79780-39-5 | -- | ng/L | -- | | 5 | U | 4.5 | U | 4.6 | U | 4.6 | U | 4.5 | U |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | -- | ng/L | 1.6 | U | 2 | U | 1.8 | U | 1.8 | U | 1.8 | U | 1.8 | U |
| Perfluoroheptane sulfonic acid (PFHpS) | 375-92-8 | -- | ng/L | 1.7 | | 4.3 | J | 15 | | 2.9 | J | 3.7 | U | 3.6 | U |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | -- | ng/L | 5.6 | | 5.2 | | 6.6 | | 16 | | 1.8 | U | 1.8 | U |
| Perfluorohexane sulfonic acid (PFHxS) | 355-46-4 | 39 | ng/L | 75 | | 140 | | 250 | | 120 | | 1.8 | U | 1.8 | U |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | -- | ng/L | 27 | | 33 | | 37 | | 44 | | 1.8 | U | 1.8 | U |
| Perfluorononane sulfonic acid (PFNS) | 68259-12-1 | -- | ng/L | 1.6 | U | 4 | U | 3.6 | U | 3.7 | U | 3.7 | U | 3.6 | U |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 6 | ng/L | 1.6 | U | 4 | U | 3.6 | U | 3.7 | U | 3.7 | U | 3.6 | U |
| Perfluorooctane sulfonamide (PFOSA) | 754-91-6 | -- | ng/L | 1.6 | U | 2 | U | 1.8 | U | 1.8 | U | 1.8 | U | 1.8 | U |
| Perfluorooctane sulfonic acid (PFOS) | 1763-23-1 | 4 | ng/L | 30 | | 69 | | 270 | | 64 | | 3.7 | U | 3.6 | U |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 6 | ng/L | 6.0 | | 8.3 | | 14 | | 20 | | 1.8 | U | 1.8 | U |
| Perfluoropentane sulfonic acid (PFPeSA) | 2706-91-4 | -- | ng/L | 17 | | 24 | | 37 | | 17 | | 1.8 | U | 1.8 | U |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | -- | ng/L | 12 | | 18 | | 20 | | 25 | | 1.8 | U | 1.8 | U |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | -- | ng/L | 1.6 | U | 4 | U | 3.6 | U | 3.7 | U | 3.7 | U | 3.6 | U |
| Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 | -- | ng/L | 1.6 | U | 2 | U | 1.8 | U | 1.8 | U | 1.8 | U | 1.8 | U |
| Perfluoroundecanoic acid (PFUdA) | 2058-94-8 | -- | ng/L | 1.6 | U | 4 | U | 3.6 | U | 3.7 | U | 3.7 | U | 3.6 | U |

Table 10-3 - Groundwater PFAS SI, Baseline, and Boundary Investigation Analytical Results
 PFAS RI QAPP
 Yakima Training Center
 Yakima, Washington



| AOPI Location Sample/Duplicate ID Sample Date Sample Type Matrix | | | | YTC-MW-03-GW | | | | | | | | YTC-MW-04-GW | | | | | | | | | | | |
|---|-------------|-----|------|---------------------------------|-----|--------------|-------|---------------------------------|------|--------|------|---------------------------------|------|--------|------|--|------|--------|------|---------------------------------|--|--|--|
| | | | | YTC-MW-03-GW-040623 | | | | YTC-MW-03-GW-052523 | | | | YTC-MW-04-GW-041323 | | | | YTC-MW-04-GW-041323 / YTC-FD-01-GW-041323 | | | | YTC-MW-04-GW-052423 | | | |
| | | | | 04/06/2023 N Ground Water | | | | 05/25/2023 N Ground Water | | | | 04/13/2023 N Ground Water | | | | 04/13/2023 FD Ground Water | | | | 05/24/2023 N Ground Water | | | |
| | | | | Analyte | CAS | OSD Tapwater | Units | Result | Qual | Result | Qual | Result | Qual | Result | Qual | Result | Qual | Result | Qual | | | | |
| PFAS | | | | | | | | | | | | | | | | | | | | | | | |
| 11-chloroeicosfluoro-3-oxaundecane-1-sulfonic acid (F-53B Minor) | 763051-92-9 | -- | ng/L | 2.1 | U | 1.8 | U | 1.9 | U | 2 | U | 1.8 | U | | | | | | | | | | |
| 2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA) | 13252-13-6 | 6 | ng/L | 2.1 | U | 1.8 | U | 1.9 | U | 2 | U | 1.8 | U | | | | | | | | | | |
| 2H, 2H, 3H, 3H-perfluorodecanoic acid (7:3 FTCA) | 812-70-4 | -- | ng/L | 100 | U | 90 | U | 93 | U | 99 | U | 92 | U | | | | | | | | | | |
| 2H, 2H, 3H, 3H-perfluorohexanoic acid | 356-02-5 | -- | ng/L | 21 | U | 18 | U | 19 | U | 20 | U | 18 | U | | | | | | | | | | |
| 2H, 2H, 3H, 3H-perfluorooctanoic acid (5:3 FTCA) | 914637-49-3 | -- | ng/L | 100 | U | 90 | U | 93 | U | 99 | U | 92 | U | | | | | | | | | | |
| 4,8-Dioxa-3H-perfluorononanoic acid (DONA) | 919005-14-4 | -- | ng/L | 2.1 | U | 1.8 | U | 1.3 | J | 2 | U | 1.8 | U | | | | | | | | | | |
| 4:2 Fluorotelomer sulfonate (4:2 FTS) | 757124-72-4 | -- | ng/L | 4.1 | U | 3.6 | U | 3.7 | U | 3.9 | U | 3.7 | U | | | | | | | | | | |
| 6:2 Fluorotelomer sulfonic acid (6:2 FTSA) | 27619-97-2 | -- | ng/L | 4.1 | U | 180 | | 3.7 | U | 3.9 | U | 11 | | | | | | | | | | | |
| 8:2 Fluorotelomer sulfonic acid (8:2 FTSA) | 39108-34-4 | -- | ng/L | 5.1 | U | 20 | | 4.6 | U | 4.9 | U | 4.6 | U | | | | | | | | | | |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9CL-PF3ONS) | 756426-58-1 | -- | ng/L | 4.1 | U | 3.6 | U | 3.7 | U | 3.9 | U | 3.7 | U | | | | | | | | | | |
| N-Ethyl perfluorooctane sulfonamide (N-EtFOSA) | 4151-50-2 | -- | ng/L | 5.1 | U | 4.5 | U | 4.6 | U | 4.9 | U | 4.6 | U | | | | | | | | | | |
| N-Ethyl perfluorooctane sulfonamide ethanol (N-EtFOSE) | 1691-99-2 | -- | ng/L | 51 | U | 45 | U | 46 | U | 49 | U | 46 | U | | | | | | | | | | |
| N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA) | 2991-50-6 | -- | ng/L | 4.1 | U | 3.6 | U | 3.7 | U | 3.9 | U | 3.7 | U | | | | | | | | | | |
| N-Methyl perfluorooctane sulfonamide (N-MeFOSA) | 31506-32-8 | -- | ng/L | 5.1 | U | 4.5 | U | 4.6 | U | 4.9 | U | 4.6 | U | | | | | | | | | | |
| N-Methyl perfluorooctane sulfonamidoethanol (N-MeFOSE) | 24448-09-7 | -- | ng/L | 51 | U | 45 | U | 46 | U | 49 | U | 46 | U | | | | | | | | | | |
| N-Methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) | 2355-31-9 | -- | ng/L | 4.1 | U | 3.6 | U | 3.7 | U | 3.9 | U | 3.7 | U | | | | | | | | | | |
| Nonafluoro-3,6-Dioxahexanoic Acid (NFDHA) | 151772-58-6 | -- | ng/L | 5.1 | U | 4.5 | U | 4.6 | U | 4.9 | U | 4.6 | U | | | | | | | | | | |
| Perfluoro (2-Ethoxyethane) Sulfonic Acid (PFEEESA) | 113507-82-7 | -- | ng/L | 2.1 | U | 1.8 | U | 1.9 | U | 2 | U | 1.8 | U | | | | | | | | | | |
| Perfluoro-3-methoxypropanoic acid (PFMPA) | 377-73-1 | -- | ng/L | 2.1 | U | 1.8 | U | 1.9 | U | 2 | U | 1.8 | U | | | | | | | | | | |
| Perfluoro-4-Methoxybutanoic acid (PFMBA) | 863090-89-5 | -- | ng/L | 2.1 | U | 1.8 | U | 1.9 | U | 2 | U | 1.8 | U | | | | | | | | | | |
| Perfluorobutane sulfonic acid (PFBS) | 375-73-5 | 601 | ng/L | 50 | | 130 | | 29 | | 30 | | 7 | | | | | | | | | | | |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | -- | ng/L | 22 | | 89 | | 7.9 | | 8.8 | | 4.3 | | | | | | | | | | | |
| Perfluorodecane sulfonic acid (PFDS) | 335-77-3 | -- | ng/L | 4.1 | U | 3.6 | U | 3.7 | U | 3.9 | U | 3.7 | U | | | | | | | | | | |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | -- | ng/L | 2.1 | U | 1.8 | U | 1.9 | U | 2 | U | 1.8 | U | | | | | | | | | | |
| Perfluorododecane sulfonic acid (PFDOS) | 79780-39-5 | -- | ng/L | 5.1 | U | 4.5 | U | 4.6 | U | 4.9 | U | 4.6 | U | | | | | | | | | | |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | -- | ng/L | 2.1 | U | 1.8 | U | 1.9 | U | 2 | U | 1.8 | U | | | | | | | | | | |
| Perfluoroheptane sulfonic acid (PFHpS) | 375-92-8 | -- | ng/L | 4 | J | 61 | | 3.7 | U | 3.9 | U | 2.4 | J | | | | | | | | | | |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | -- | ng/L | 20 | | 120 | | 9.8 | | 9.8 | | 5 | | | | | | | | | | | |
| Perfluorohexane sulfonic acid (PFHxS) | 355-46-4 | 39 | ng/L | 200 | | 1000 | | 140 | | 130 | | 64 | | | | | | | | | | | |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | -- | ng/L | 73 | | 340 | | 45 | | 44 | | 18 | | | | | | | | | | | |
| Perfluorononane sulfonic acid (PFNS) | 68259-12-1 | -- | ng/L | 4.1 | U | 3.6 | U | 3.7 | U | 3.9 | U | 3.7 | U | | | | | | | | | | |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 6 | ng/L | 4.1 | U | 18 | | 3.7 | U | 3.9 | U | 3.7 | U | | | | | | | | | | |
| Perfluorooctane sulfonamide (PFOSA) | 754-91-6 | -- | ng/L | 2.1 | U | 1.8 | U | 1.9 | U | 2 | U | 1.8 | U | | | | | | | | | | |
| Perfluorooctane sulfonic acid (PFOS) | 1763-23-1 | 4 | ng/L | 110 | | 1500 | | 27 | | 26 | | 73 | | | | | | | | | | | |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 6 | ng/L | 26 | | 140 | | 12 | | 12 | | 8.3 | | | | | | | | | | | |
| Perfluoropentane sulfonic acid (PFPeSA) | 2706-91-4 | -- | ng/L | 38 | | 140 | | 25 | | 27 | | 7.2 | | | | | | | | | | | |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | -- | ng/L | 61 | | 270 | | 25 | | 24 | | 15 | | | | | | | | | | | |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | -- | ng/L | 4.1 | U | 3.6 | U | 3.7 | U | 3.9 | U | 3.7 | U | | | | | | | | | | |
| Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 | -- | ng/L | 2.1 | U | 1.8 | U | 1.9 | U | 2 | U | 1.8 | U | | | | | | | | | | |
| Perfluoroundecanoic acid (PFUdA) | 2058-94-8 | -- | ng/L | 4.1 | U | 3.6 | U | 3.7 | U | 3.9 | U | 3.7 | U | | | | | | | | | | |

Table 10-3 - Groundwater PFAS SI, Baseline, and Boundary Investigation Analytical Results
 PFAS RI QAPP
 Yakima Training Center
 Yakima, Washington



| AOPI Location Sample/Duplicate ID Sample Date Sample Type Matrix | | | | YTC-MW-05-GW | | | | | | | | | | | |
|---|-------------|--------------|-------|---------------------------------|------|---------------------------------|------|---------------------------------|------|---------------------------------|------|---------------------------------|------|--|--|
| | | | | YTC-MW-05-GW | | | | YTC-MW-06-GW | | | | YTC-MW-07-GW | | | |
| | | | | YTC-MW-05-GW-041123 | | YTC-MW-05-GW-052323 | | YTC-MW-06-GW-041923 | | YTC-MW-06-GW-052423 | | YTC-MW-07-GW-052423 | | | |
| | | | | 04/11/2023 N Ground Water | | 05/23/2023 N Ground Water | | 04/19/2023 N Ground Water | | 05/24/2023 N Ground Water | | 05/24/2023 N Ground Water | | | |
| Analyte | CAS | OSD Tapwater | Units | Result | Qual | | |
| PFAS | | | | | | | | | | | | | | | |
| 11-chloroeicosfluoro-3-oxaundecane-1-sulfonic acid (F-53B Minor) | 763051-92-9 | -- | ng/L | 1.9 | U | 1.8 | U | 2.1 | U | 1.7 | U | 1.8 | U | | |
| 2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA) | 13252-13-6 | 6 | ng/L | 1.9 | U | 1.8 | U | 2.1 | U | 1.7 | U | 1.8 | U | | |
| 2H, 2H, 3H, 3H-perfluorodecanoic acid (7:3 FTCA) | 812-70-4 | -- | ng/L | 97 | U | 92 | U | 100 | U | 86 | U | 90 | U | | |
| 2H, 2H, 3H, 3H-perfluorohexanoic acid | 356-02-5 | -- | ng/L | 19 | U | 18 | U | 21 | U | 17 | U | 18 | U | | |
| 2H, 2H, 3H, 3H-perfluorooctanoic acid (5:3 FTCA) | 914637-49-3 | -- | ng/L | 97 | U | 92 | U | 100 | U | 86 | U | 90 | U | | |
| 4,8-Dioxa-3H-perfluorononanoic acid (DONA) | 919005-14-4 | -- | ng/L | 1.9 | U | 1.8 | U | 2.1 | U | 1.7 | U | 1.8 | U | | |
| 4:2 Fluorotelomer sulfonate (4:2 FTS) | 757124-72-4 | -- | ng/L | 3.9 | U | 3.7 | U | 4.2 | U | 3.5 | U | 3.6 | U | | |
| 6:2 Fluorotelomer sulfonic acid (6:2 FTSA) | 27619-97-2 | -- | ng/L | 3.9 | U | 3.7 | U | 36 | | 240 | | 21 | | | |
| 8:2 Fluorotelomer sulfonic acid (8:2 FTSA) | 39108-34-4 | -- | ng/L | 4.8 | U | 4.6 | U | 5.2 | U | 7.7 | J | 4.5 | U | | |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9CL-PF3ONS) | 756426-58-1 | -- | ng/L | 3.9 | U | 3.7 | U | 4.2 | U | 3.5 | U | 3.6 | U | | |
| N-Ethyl perfluorooctane sulfonamide (N-EtFOSA) | 4151-50-2 | -- | ng/L | 4.8 | U | 4.6 | U | 5.2 | U | 4.3 | U | 4.5 | U | | |
| N-Ethyl perfluorooctane sulfonamide ethanol (N-EtFOSE) | 1691-99-2 | -- | ng/L | 48 | U | 46 | U | 52 | U | 43 | U | 45 | U | | |
| N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA) | 2991-50-6 | -- | ng/L | 3.9 | U | 3.7 | U | 4.2 | U | 3.5 | U | 3.6 | U | | |
| N-Methyl perfluorooctane sulfonamide (N-MeFOSA) | 31506-32-8 | -- | ng/L | 4.8 | U | 4.6 | U | 5.2 | U | 4.3 | U | 4.5 | U | | |
| N-Methyl perfluorooctane sulfonamidoethanol (N-MeFOSE) | 24448-09-7 | -- | ng/L | 48 | U | 46 | U | 52 | U | 43 | U | 45 | U | | |
| N-Methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) | 2355-31-9 | -- | ng/L | 3.9 | U | 3.7 | U | 4.2 | U | 3.5 | U | 3.6 | U | | |
| Nonafluoro-3,6-Dioxahexanoic Acid (NFDHA) | 151772-58-6 | -- | ng/L | 4.8 | U | 4.6 | U | 5.2 | U | 4.3 | U | 4.5 | U | | |
| Perfluoro (2-Ethoxyethane) Sulfonic Acid (PFEEESA) | 113507-82-7 | -- | ng/L | 1.9 | U | 1.8 | U | 2.1 | U | 1.7 | U | 1.8 | U | | |
| Perfluoro-3-methoxypropanoic acid (PFMPA) | 377-73-1 | -- | ng/L | 1.9 | U | 1.8 | U | 2.1 | U | 1.7 | U | 1.8 | U | | |
| Perfluoro-4-Methoxybutanoic acid (PFMBA) | 863090-89-5 | -- | ng/L | 1.9 | U | 1.8 | U | 2.1 | U | 1.7 | U | 1.8 | U | | |
| Perfluorobutane sulfonic acid (PFBS) | 375-73-5 | 601 | ng/L | 7.4 | | 1.8 | U | 13 | | 120 | | 38 | | | |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | -- | ng/L | 3.8 | J | 1.8 | U | 9 | | 74 | | 20 | | | |
| Perfluorodecane sulfonic acid (PFDS) | 335-77-3 | -- | ng/L | 3.9 | U | 3.7 | U | 4.2 | U | 3.5 | U | 3.6 | U | | |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | -- | ng/L | 1.9 | U | 1.8 | U | 2.1 | U | 1.7 | U | 1.8 | U | | |
| Perfluorododecane sulfonic acid (PFDOS) | 79780-39-5 | -- | ng/L | 4.8 | U | 4.6 | U | 5.2 | U | 4.3 | U | 4.5 | U | | |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | -- | ng/L | 1.9 | U | 1.8 | U | 2.1 | U | 1.7 | U | 1.8 | U | | |
| Perfluoroheptane sulfonic acid (PFHpS) | 375-92-8 | -- | ng/L | 3.9 | U | 3.7 | U | 5.7 | J | 44 | | 5.7 | J | | |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | -- | ng/L | 7.5 | | 1.8 | U | 11 | | 120 | | 25 | | | |
| Perfluorohexane sulfonic acid (PFHxS) | 355-46-4 | 39 | ng/L | 66 | | 1.8 | U | 110 | | 1100 | | 200 | | | |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | -- | ng/L | 26 | | 1.8 | U | 38 | | 290 | | 85 | | | |
| Perfluorononane sulfonic acid (PFNS) | 68259-12-1 | -- | ng/L | 3.9 | U | 3.7 | U | 4.2 | U | 3.5 | U | 3.6 | U | | |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 6 | ng/L | 3.9 | U | 3.7 | U | 4.2 | U | 8.5 | | 3.6 | U | | |
| Perfluorooctane sulfonamide (PFOSA) | 754-91-6 | -- | ng/L | 1.9 | U | 1.8 | U | 2.1 | U | 1.7 | U | 1.8 | U | | |
| Perfluorooctane sulfonic acid (PFOS) | 1763-23-1 | 4 | ng/L | 3.9 | U | 3.7 | U | 200 | | 1200 | | 110 | | | |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 6 | ng/L | 12 | | 1.8 | U | 16 | | 120 | | 38 | | | |
| Perfluoropentane sulfonic acid (PFPeSA) | 2706-91-4 | -- | ng/L | 6.9 | | 1.8 | U | 15 | | 140 | | 35 | | | |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | -- | ng/L | 21 | | 1.8 | U | 33 | | 260 | | 95 | | | |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | -- | ng/L | 3.9 | U | 3.7 | U | 4.2 | U | 3.5 | U | 3.6 | U | | |
| Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 | -- | ng/L | 1.9 | U | 1.8 | U | 2.1 | U | 1.7 | U | 1.8 | U | | |
| Perfluoroundecanoic acid (PFUdA) | 2058-94-8 | -- | ng/L | 3.9 | U | 3.7 | U | 4.2 | U | 3.5 | U | 3.6 | U | | |

Table 10-3 - Groundwater PFAS SI, Baseline, and Boundary Investigation Analytical Results
 PFAS RI QAPP
 Yakima Training Center
 Yakima, Washington



| AOPI Location Sample/Duplicate ID Sample Date Sample Type Matrix | | | | YTC-MW-08-GW | | YTC-SOURCE-1 | | YTC-TVR-2-GW | | YTC-TVR-3-GW | | YTC-TVR-6-GW | | | |
|---|-------------|-----|------|---------------------------------|-----|---------------------------------|-------|---------------------------------|------|---------------------------------|------|---------------------------------|------|---------------------------------|------|
| | | | | YTC-MW-08-GW-041423 | | YTC-MW-08-GW-052423 | | YTC-SOURCE-1-020723 | | YTC-TVR-2-GW-020823 | | YTC-TVR-3-GW-020723 | | YTC-TVR-6-GW-020623 | |
| | | | | 04/14/2023 N Ground Water | | 05/24/2023 N Ground Water | | 02/07/2023 N Ground Water | | 02/08/2023 N Ground Water | | 02/07/2023 N Ground Water | | 02/06/2023 N Ground Water | |
| | | | | Analyte | CAS | OSD Tapwater | Units | Result | Qual | Result | Qual | Result | Qual | Result | Qual |
| PFAS | | | | | | | | | | | | | | | |
| 11-chloroeicosfluoro-3-oxaundecane-1-sulfonic acid (F-53B Minor) | 763051-92-9 | -- | ng/L | 2.1 | U | 1.8 | U | -- | | -- | | -- | | -- | |
| 2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA) | 13252-13-6 | 6 | ng/L | 2.1 | U | 1.8 | U | -- | | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorodecanoic acid (7:3 FTCA) | 812-70-4 | -- | ng/L | 100 | U | 89 | U | -- | | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorohexanoic acid | 356-02-5 | -- | ng/L | 21 | U | 18 | U | -- | | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorooctanoic acid (5:3 FTCA) | 914637-49-3 | -- | ng/L | 100 | U | 89 | U | -- | | -- | | -- | | -- | |
| 4,8-Dioxa-3H-perfluorononanoic acid (DONA) | 919005-14-4 | -- | ng/L | 2.1 | U | 1.8 | U | -- | | -- | | -- | | -- | |
| 4:2 Fluorotelomer sulfonate (4:2 FTS) | 757124-72-4 | -- | ng/L | 4.2 | U | 3.6 | U | 1.7 | U | 1.6 | U | 1.6 | U | 1.7 | |
| 6:2 Fluorotelomer sulfonic acid (6:2 FTSA) | 27619-97-2 | -- | ng/L | 4.2 | U | 3.6 | U | 2.5 | U | 2.4 | U | 2.5 | U | 2.6 | |
| 8:2 Fluorotelomer sulfonic acid (8:2 FTSA) | 39108-34-4 | -- | ng/L | 5.2 | U | 4.4 | U | 2.5 | U | 2.4 | U | 2.5 | U | 2.5 | |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9CL-PF3ONS) | 756426-58-1 | -- | ng/L | 4.2 | U | 3.6 | U | -- | | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamide (N-EtFOSA) | 4151-50-2 | -- | ng/L | 5.2 | U | 4.4 | U | -- | | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamide ethanol (N-EtFOSE) | 1691-99-2 | -- | ng/L | 52 | U | 44 | U | -- | | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA) | 2991-50-6 | -- | ng/L | 4.2 | U | 3.6 | U | 2.5 | U | 2.4 | U | 2.5 | U | 2.5 | |
| N-Methyl perfluorooctane sulfonamide (N-MeFOSA) | 31506-32-8 | -- | ng/L | 5.2 | U | 4.4 | U | -- | | -- | | -- | | -- | |
| N-Methyl perfluorooctane sulfonamidoethanol (N-MeFOSE) | 24448-09-7 | -- | ng/L | 52 | U | 44 | U | -- | | -- | | -- | | -- | |
| N-Methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) | 2355-31-9 | -- | ng/L | 4.2 | U | 3.6 | U | 1.6 | U | 1.6 | U | 1.6 | U | 1.7 | |
| Nonafluoro-3,6-Dioxaheptonic Acid (NFDHA) | 151772-58-6 | -- | ng/L | 5.2 | U | 4.4 | U | -- | | -- | | -- | | -- | |
| Perfluoro (2-Ethoxyethane) Sulfonic Acid (PFEEESA) | 113507-82-7 | -- | ng/L | 2.1 | U | 1.8 | U | -- | | -- | | -- | | -- | |
| Perfluoro-3-methoxypropanoic aci (PFMPA) | 377-73-1 | -- | ng/L | 2.1 | U | 1.8 | U | -- | | -- | | -- | | -- | |
| Perfluoro-4-Methoxybutanic acid (PFMBA) | 863090-89-5 | -- | ng/L | 2.1 | U | 1.8 | U | -- | | -- | | -- | | -- | |
| Perfluorobutane sulfonic acid (PFBS) | 375-73-5 | 601 | ng/L | 2.1 | U | 1.8 | U | 1.6 | U | 5.9 | | 27 | | 4.1 | |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | -- | ng/L | 2.1 | U | 1.8 | U | 4.1 | U | 9.5 | | 11 | | 4.0 | |
| Perfluorodecane sulfonic acid (PFDS) | 335-77-3 | -- | ng/L | 4.2 | U | 3.6 | U | 1.6 | U | 1.6 | U | 1.1 | J | 1.7 | |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | -- | ng/L | 2.1 | U | 1.8 | U | 1.6 | U | 1.6 | U | 1.6 | U | 2.8 | |
| Perfluorododecane sulfonic acid (PFDOS) | 79780-39-5 | -- | ng/L | 5.2 | U | 4.4 | U | -- | | -- | | -- | | -- | |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | -- | ng/L | 2.1 | U | 1.8 | U | 1.6 | U | 1.6 | U | 1.6 | U | 1.7 | |
| Perfluoroheptane sulfonic acid (PFHpS) | 375-92-8 | -- | ng/L | 4.2 | U | 3.6 | U | 1.6 | U | 1.4 | J | 11 | J+ | 1.4 | |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | -- | ng/L | 2.1 | U | 1.8 | U | 1.6 | U | 4.9 | | 8.8 | | 3.2 | |
| Perfluorohexane sulfonic acid (PFHxS) | 355-46-4 | 39 | ng/L | 2.1 | U | 1.8 | U | 1.6 | U | 43 | | 310 | | 45 | |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | -- | ng/L | 2.1 | U | 1.8 | U | 1.6 | U | 12 | | 46 | | 8.5 | |
| Perfluorononane sulfonic acid (PFNS) | 68259-12-1 | -- | ng/L | 4.2 | U | 3.6 | U | 1.6 | U | 1.6 | U | 0.52 | J | 1.7 | |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 6 | ng/L | 4.2 | U | 3.6 | U | 1.6 | U | 0.76 | J | 0.73 | J | 1.8 | |
| Perfluorooctane sulfonamide (PFOSA) | 754-91-6 | -- | ng/L | 2.1 | U | 1.8 | U | 1.6 | U | 1.6 | U | 0.80 | J | 1.7 | |
| Perfluorooctane sulfonic acid (PFOS) | 1763-23-1 | 4 | ng/L | 4.2 | U | 3.6 | U | 1.7 | U | 65 | | 370 | | 100 | |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 6 | ng/L | 2.1 | U | 1.8 | U | 1.6 | U | 5.3 | | 13 | | 3.6 | |
| Perfluoropentane sulfonic acid (PFPeSA) | 2706-91-4 | -- | ng/L | 2.1 | U | 1.8 | U | 1.6 | U | 7.1 | | 49 | | 7.2 | |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | -- | ng/L | 2.1 | U | 1.8 | U | 1.6 | U | 10 | | 21 | | 7.0 | |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | -- | ng/L | 4.2 | U | 3.6 | U | 1.6 | U | 1.6 | U | 1.6 | U | 1.7 | |
| Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 | -- | ng/L | 2.1 | U | 1.8 | U | 1.6 | U | 1.6 | U | 1.6 | U | 1.7 | |
| Perfluoroundecanoic acid (PFUdA) | 2058-94-8 | -- | ng/L | 4.2 | U | 3.6 | U | 1.6 | U | 1.6 | U | 1.6 | U | 1.7 | |

Table 10-3 - Groundwater PFAS SI, Baseline, and Boundary Investigation Analytical Results
 PFAS RI QAPP
 Yakima Training Center
 Yakima, Washington



| AOPI Location Sample/Duplicate ID Sample Date Sample Type Matrix | | | | BIRD BATH WASH RACK | | | | | | BOUNDARY | | | |
|---|-------------|--------------|-------|---------------------------------|------|---------------------------------|------|---------------------------------|------|---------------------------------|------|---------------------------------|------|
| | | | | YTC-BBWR-1 | | YTC-BBWR-2 | | YTC-BBWR-3 | | YTC-815-2 | | YTC-MRC-2 | |
| | | | | YTC-BBWR-1-GW-092420 | | YTC-BBWR-2-GW-092320 | | YTC-BBWR-3-GW-092420 | | YTC-815-2-092520 | | YTC-MRC-2-092520 | |
| | | | | 09/24/2020 N Ground Water | | 09/23/2020 N Ground Water | | 09/24/2020 N Ground Water | | 09/25/2020 N Ground Water | | 09/25/2020 N Ground Water | |
| Analyte | CAS | OSD Tapwater | Units | Result | Qual |
| PFAS | | | | | | | | | | | | | |
| 11-chloroeicosfluoro-3-oxaundecane-1-sulfonic acid (F-53B Minor) | 763051-92-9 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| 2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA) | 13252-13-6 | 6 | ng/L | -- | | -- | | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorodecanoic acid (7:3 FTCA) | 812-70-4 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorohexanoic acid | 356-02-5 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorooctanoic acid (5:3 FTCA) | 914637-49-3 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| 4,8-Dioxa-3H-perfluorononanoic acid (DONA) | 919005-14-4 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| 4:2 Fluorotelomer sulfonate (4:2 FTS) | 757124-72-4 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| 6:2 Fluorotelomer sulfonic acid (6:2 FTSA) | 27619-97-2 | -- | ng/L | 7.4 | J | 4.1 | J | 6.9 | U | 8.4 | | 4.1 | J |
| 8:2 Fluorotelomer sulfonic acid (8:2 FTSA) | 39108-34-4 | -- | ng/L | 7.7 | U | 7.1 | U | 6.9 | U | 7.7 | U | 5.2 | J |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9CL-PF3ONS) | 756426-58-1 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamide (N-EtFOSA) | 4151-50-2 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamide ethanol (N-EtFOSE) | 1691-99-2 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA) | 2991-50-6 | -- | ng/L | 7.7 | U | 7.1 | U | 6.9 | U | 7.7 | U | 7.5 | U |
| N-Methyl perfluorooctane sulfonamide (N-MeFOSA) | 31506-32-8 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| N-Methyl perfluorooctane sulfonamidoethanol (N-MeFOSE) | 24448-09-7 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| N-Methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) | 2355-31-9 | -- | ng/L | 7.7 | U | 7.1 | U | 6.9 | U | 7.7 | U | 7.5 | U |
| Nonafluoro-3,6-Dioxaheptonic Acid (NFDHA) | 151772-58-6 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| Perfluoro (2-Ethoxyethane) Sulfonic Acid (PFEEESA) | 113507-82-7 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| Perfluoro-3-methoxypropanoic acid (PFMPA) | 377-73-1 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| Perfluoro-4-Methoxybutanoic acid (PFMBA) | 863090-89-5 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| Perfluorobutane sulfonic acid (PFBS) | 375-73-5 | 601 | ng/L | 2200 | J | 1400 | J | 1800 | J | 75 | | 130 | |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | -- | ng/L | 540 | | 250 | | 360 | | 37 | | 64 | |
| Perfluorodecane sulfonic acid (PFDS) | 335-77-3 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | -- | ng/L | 3.9 | U | 3.5 | U | 3.5 | U | 3.8 | U | 3.8 | U |
| Perfluorododecane sulfonic acid (PFDOS) | 79780-39-5 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | -- | ng/L | 3.9 | U | 3.5 | U | 3.5 | U | 3.8 | U | 3.8 | U |
| Perfluoroheptane sulfonic acid (PFHpS) | 375-92-8 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | -- | ng/L | 690 | J+ | 250 | | 400 | | 47 | | 34 | |
| Perfluorohexane sulfonic acid (PFHxS) | 355-46-4 | 39 | ng/L | 20000 | J | 8300 | J | 7600 | J | 590 | | 830 | J |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | -- | ng/L | 2800 | J | 1500 | J | 2300 | J | 120 | | 170 | |
| Perfluorononane sulfonic acid (PFNS) | 68259-12-1 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 6 | ng/L | 3.9 | U | 3.5 | U | 3.5 | U | 3.8 | U | 2.6 | J |
| Perfluorooctane sulfonamide (PFOSA) | 754-91-6 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| Perfluorooctane sulfonic acid (PFOS) | 1763-23-1 | 4 | ng/L | 6600 | J | 3100 | J | 2400 | J | 260 | | 1200 | J |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 6 | ng/L | 820 | | 380 | | 220 | | 51 | | 49 | |
| Perfluoropentane sulfonic acid (PFPeSA) | 2706-91-4 | -- | ng/L | -- | | -- | | -- | | -- | | -- | |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | -- | ng/L | 650 | | 310 | | 460 | | 69 | | 110 | |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | -- | ng/L | 3.9 | U | 3.5 | U | 3.5 | U | 3.8 | U | 3.8 | U |
| Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 | -- | ng/L | 3.9 | U | 3.5 | U | 3.5 | U | 3.8 | U | 3.8 | U |
| Perfluoroundecanoic acid (PFUdA) | 2058-94-8 | -- | ng/L | 3.9 | U | 3.5 | U | 3.5 | U | 3.8 | U | 3.8 | U |

Table 10-3 - Groundwater PFAS SI, Baseline, and Boundary Investigation Analytical Results
 PFAS RI QAPP
 Yakima Training Center
 Yakima, Washington



| AOPI Location Sample/Duplicate ID Sample Date Sample Type Matrix | | | | FIRE TRAINING PIT | | | | | | | | | | | |
|---|-------------|--------------|-------|---------------------------------|------|---------------------------------|------|--|------|---------------------------------|------|---------------------------------|------|---------------------------------|------|
| | | | | YTC-FTP-1 | | YTC-FTP-13 | | | | YTC-FTP-14 | | YTC-FTP-15 | | YTC-FTP-16 | |
| | | | | YTC-FTP-1-092320 | | YTC-FTP-13-092120 | | YTC-FTP-13-092120 / YTC-FD-1-GW-092120 | | YTC-FTP-14-092220 | | YTC-FTP-15-092220 | | YTC-FTP-16-092420 | |
| | | | | 09/23/2020 N Ground Water | | 09/21/2020 N Ground Water | | 09/21/2020 FD Ground Water | | 09/22/2020 N Ground Water | | 09/22/2020 N Ground Water | | 09/24/2020 N Ground Water | |
| Analyte | CAS | OSD Tapwater | Units | Result | Qual | Result | Qual | Result | Qual | Result | Qual | Result | Qual | Result | Qual |
| PFAS | | | | | | | | | | | | | | | |
| 11-chloroeicosfluoro-3-oxaundecane-1-sulfonic acid (F-53B Minor) | 763051-92-9 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| 2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA) | 13252-13-6 | 6 | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorodecanoic acid (7:3 FTCA) | 812-70-4 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorohexanoic acid | 356-02-5 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorooctanoic acid (5:3 FTCA) | 914637-49-3 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| 4,8-Dioxa-3H-perfluorononanoic acid (DONA) | 919005-14-4 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| 4:2 Fluorotelomer sulfonate (4:2 FTS) | 757124-72-4 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| 6:2 Fluorotelomer sulfonic acid (6:2 FTSA) | 27619-97-2 | -- | ng/L | 3200 | | 7.1 | UJ | 7.2 | U | 7.4 | UJ | 3.7 | J | 46 | |
| 8:2 Fluorotelomer sulfonic acid (8:2 FTSA) | 39108-34-4 | -- | ng/L | 230 | | 7.1 | U | 7.2 | U | 7.4 | U | 6.9 | U | 7.4 | U |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9CL-PF3ONS) | 756426-58-1 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamide (N-EtFOSA) | 4151-50-2 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamide ethanol (N-EtFOSE) | 1691-99-2 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA) | 2991-50-6 | -- | ng/L | 7.5 | U | 7.1 | U | 7.2 | U | 7.4 | U | 6.9 | U | 7.4 | U |
| N-Methyl perfluorooctane sulfonamide (N-MeFOSA) | 31506-32-8 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| N-Methyl perfluorooctane sulfonamidoethanol (N-MeFOSE) | 24448-09-7 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| N-Methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) | 2355-31-9 | -- | ng/L | 7.5 | U | 7.1 | U | 7.2 | U | 7.4 | U | 6.9 | U | 7.4 | U |
| Nonafluoro-3,6-Dioxaheptonic Acid (NFDHA) | 151772-58-6 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluoro (2-Ethoxyethane) Sulfonic Acid (PFEEESA) | 113507-82-7 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluoro-3-methoxypropanoic aci (PFMPA) | 377-73-1 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluoro-4-Methoxybutanic acid (PFMBA) | 863090-89-5 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluorobutane sulfonic acid (PFBS) | 375-73-5 | 601 | ng/L | 5900 | J | 1100 | J | 1100 | J | 570 | | 320 | | 5200 | J |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | -- | ng/L | 1500 | J | 220 | | 220 | | 200 | | 100 | | 1700 | J |
| Perfluorodecane sulfonic acid (PFDS) | 335-77-3 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | -- | ng/L | 3.8 | U | 3.5 | U | 3.6 | U | 3.7 | U | 3.4 | U | 3.7 | U |
| Perfluorododecane sulfonic acid (PFDOS) | 79780-39-5 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | -- | ng/L | 3.8 | U | 3.5 | U | 3.6 | U | 3.7 | U | 3.4 | U | 3.7 | U |
| Perfluoroheptane sulfonic acid (PFHpS) | 375-92-8 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | -- | ng/L | 2000 | J | 120 | | 120 | | 190 | | 81 | | 2000 | J |
| Perfluorohexane sulfonic acid (PFHxS) | 355-46-4 | 39 | ng/L | 23000 | J | 3500 | J | 3500 | J | 2000 | J | 860 | J | 18000 | J |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | -- | ng/L | 11000 | J | 1200 | J | 1300 | J | 1400 | J | 530 | | 11000 | J |
| Perfluorononane sulfonic acid (PFNS) | 68259-12-1 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 6 | ng/L | 75 | J+ | 3.5 | U | 3.6 | U | 4.2 | | 3.9 | | 38 | |
| Perfluorooctane sulfonamide (PFOSA) | 754-91-6 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluorooctane sulfonic acid (PFOS) | 1763-23-1 | 4 | ng/L | 45000 | J | 410 | | 440 | | 1900 | J | 1700 | J | 10000 | J |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 6 | ng/L | 5200 | J | 150 | | 160 | | 490 | | 180 | | 3900 | J |
| Perfluoropentane sulfonic acid (PFPeSA) | 2706-91-4 | -- | ng/L | -- | | -- | | -- | | -- | | -- | | -- | |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | -- | ng/L | 5300 | J | 280 | | 300 | | 570 | | 300 | | 6400 | J |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | -- | ng/L | 3.8 | UJ | 3.5 | U | 3.6 | U | 3.7 | U | 3.4 | U | 3.7 | U |
| Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 | -- | ng/L | 3.8 | U | 3.5 | U | 3.6 | U | 3.7 | U | 3.4 | U | 3.7 | U |
| Perfluoroundecanoic acid (PFUdA) | 2058-94-8 | -- | ng/L | 4.0 | J | 3.5 | U | 3.6 | U | 3.7 | U | 3.4 | U | 3.7 | U |

Table 10-3 - Groundwater PFAS SI, Baseline, and Boundary Investigation Analytical Results
 PFAS RI QAPP
 Yakima Training Center
 Yakima, Washington



| AOPI Location Sample/Duplicate ID Sample Date Sample Type Matrix | | | | REFRACTOMETER SOLUTIONS TESTING AREA | | | | | |
|---|-------------|--------------|-------|--------------------------------------|------|---------------------------------|------|---------------------------------|------|
| | | | | YTC-MMP-1 | | YTC-MMP-2 | | YTC-TRV-5 | |
| | | | | YTC-MMP-1-092420 | | YTC-MMP-2-092520 | | YTC-TRV-5-092520 | |
| | | | | 09/24/2020 N Ground Water | | 09/25/2020 N Ground Water | | 09/25/2020 N Ground Water | |
| Analyte | CAS | OSD Tapwater | Units | Result | Qual | Result | Qual | Result | Qual |
| PFAS | | | | | | | | | |
| 11-chloroeicosfluoro-3-oxaundecane-1-sulfonic acid (F-53B Minor) | 763051-92-9 | -- | ng/L | -- | | -- | | -- | |
| 2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA) | 13252-13-6 | 6 | ng/L | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorodecanoic acid (7:3 FTCA) | 812-70-4 | -- | ng/L | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorohexanoic acid | 356-02-5 | -- | ng/L | -- | | -- | | -- | |
| 2H, 2H, 3H, 3H-perfluorooctanoic acid (5:3 FTCA) | 914637-49-3 | -- | ng/L | -- | | -- | | -- | |
| 4,8-Dioxa-3H-perfluorononanoic acid (DONA) | 919005-14-4 | -- | ng/L | -- | | -- | | -- | |
| 4:2 Fluorotelomer sulfonate (4:2 FTS) | 757124-72-4 | -- | ng/L | -- | | -- | | -- | |
| 6:2 Fluorotelomer sulfonic acid (6:2 FTSA) | 27619-97-2 | -- | ng/L | 670 | | 670 | | 8.0 | U |
| 8:2 Fluorotelomer sulfonic acid (8:2 FTSA) | 39108-34-4 | -- | ng/L | 300 | | 140 | | 8.0 | U |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9CL-PF3ONS) | 756426-58-1 | -- | ng/L | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamide (N-EtFOSA) | 4151-50-2 | -- | ng/L | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamide ethanol (N-EtFOSE) | 1691-99-2 | -- | ng/L | -- | | -- | | -- | |
| N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA) | 2991-50-6 | -- | ng/L | 7.5 | U | 7.9 | U | 8.0 | U |
| N-Methyl perfluorooctane sulfonamide (N-MeFOSA) | 31506-32-8 | -- | ng/L | -- | | -- | | -- | |
| N-Methyl perfluorooctane sulfonamidoethanol (N-MeFOSE) | 24448-09-7 | -- | ng/L | -- | | -- | | -- | |
| N-Methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) | 2355-31-9 | -- | ng/L | 7.5 | U | 7.9 | U | 8.0 | U |
| Nonafluoro-3,6-Dioxaheptonic Acid (NFDHA) | 151772-58-6 | -- | ng/L | -- | | -- | | -- | |
| Perfluoro (2-Ethoxyethane) Sulfonic Acid (PFEEESA) | 113507-82-7 | -- | ng/L | -- | | -- | | -- | |
| Perfluoro-3-methoxypropanoic aci (PFMPA) | 377-73-1 | -- | ng/L | -- | | -- | | -- | |
| Perfluoro-4-Methoxybutanic acid (PFMBA) | 863090-89-5 | -- | ng/L | -- | | -- | | -- | |
| Perfluorobutane sulfonic acid (PFBS) | 375-73-5 | 601 | ng/L | 46 | | 100 | | 47 | |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | -- | ng/L | 53 | | 81 | | 36 | |
| Perfluorodecane sulfonic acid (PFDS) | 335-77-3 | -- | ng/L | -- | | -- | | -- | |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | -- | ng/L | 5.7 | | 2.2 | J | 4.0 | U |
| Perfluorododecane sulfonic acid (PFDOS) | 79780-39-5 | -- | ng/L | -- | | -- | | -- | |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | -- | ng/L | 3.8 | U | 4.0 | U | 4.0 | U |
| Perfluoroheptane sulfonic acid (PFHpS) | 375-92-8 | -- | ng/L | -- | | -- | | -- | |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | -- | ng/L | 130 | | 140 | | 23 | |
| Perfluorohexane sulfonic acid (PFHxS) | 355-46-4 | 39 | ng/L | 960 | J | 970 | J | 300 | |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | -- | ng/L | 190 | | 290 | | 100 | |
| Perfluorononane sulfonic acid (PFNS) | 68259-12-1 | -- | ng/L | -- | | -- | | -- | |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 6 | ng/L | 14 | | 7.8 | | 4.0 | U |
| Perfluorooctane sulfonamide (PFOSA) | 754-91-6 | -- | ng/L | -- | | -- | | -- | |
| Perfluorooctane sulfonic acid (PFOS) | 1763-23-1 | 4 | ng/L | 5300 | J | 2600 | J | 180 | |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 6 | ng/L | 150 | | 170 | | 18 | |
| Perfluoropentane sulfonic acid (PFPeSA) | 2706-91-4 | -- | ng/L | -- | | -- | | -- | |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | -- | ng/L | 180 | | 270 | | 100 | |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | -- | ng/L | 3.8 | U | 4.0 | U | 4.0 | U |
| Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 | -- | ng/L | 3.8 | U | 4.0 | U | 4.0 | U |
| Perfluoroundecanoic acid (PFUdA) | 2058-94-8 | -- | ng/L | 3.8 | U | 4.0 | U | 4.0 | U |

Notes:

1. **Bolded** values indicate the result was detected greater than the limit of detection.
2. Grey shaded values indicate the result was detected greater than the 2022 Office of the Secretary of Defense (OSD) risk screening levels, (OSD. 2022. Memorandum: Investigating Per- and Polyfluoroalkyl Substances within the Department of Defense Cleanup Program. July).

Acronyms/Abbreviations:

- = not applicable
- % = percent
- AOPI = Area of Potential Interest
- CAS = Chemical Abstracts Service number
- FD = field duplicate sample
- ID = identification
- N = primary sample
- ng/L = nanograms per liter (parts per trillion)
- PFAS = per- and polyfluoroalkyl substances

| Qualifier | Description |
|-----------|--|
| J | The analyte was positively identified; however the associated numerical value is an estimated concentration only. |
| J+ | The result is an estimated quantity; the result may be biased high. |
| U | The analyte was analyzed for but the result was not detected above the limit of quantitation (LOQ). |
| UJ | The analyte was analyzed for but was not detected. The reported limit of quantitation (LOQ) is approximate and may be inaccurate or imprecise. |

| AOPI Location Sample/Duplicate ID Sample Date Sample Type Matrix | | | | BUILDING 321 AFFF STORAGE AREA | | | | BUILDING 821 AFFF STORAGE AREA | | | | REFRACTOMETER SOLUTIONS TESTING AREA | | | |
|---|------------|--------------------------|-------|--------------------------------|------|-------------------------|------|--------------------------------|------|-------------------------|------|--------------------------------------|------|-------------------------|--|
| | | | | YTC-B321-1 | | YTC-B321-2 | | YTC-B821-1 | | YTC-B821-2 | | YTC-RSTA-2 | | YTC-RSTA-3 | |
| | | | | YTC-B321-1-SO-092320 | | YTC-B321-2-SO-092320 | | YTC-B821-1-SO-092320 | | YTC-B821-2-SO-092320 | | YTC-RSTA-2-SO-092520 | | YTC-RSTA-3-SO-092420 | |
| | | | | 09/23/2020 N Soil | | 09/23/2020 N Soil | | 09/23/2020 N Soil | | 09/23/2020 N Soil | | 09/25/2020 N Soil | | 09/24/2020 N Soil | |
| Analyte | CAS | OSD Risk Screening Level | Units | Result | Qual | Result | Qual | Result | Qual | Result | Qual | Result | Qual | | |
| PFAS | | | | | | | | | | | | | | | |
| 6:2 Fluorotelomer sulfonic acid (6:2 FTSA) | 27619-97-2 | -- | mg/kg | 0.0019 | U | 0.002 | U | 0.0017 | U | 0.0019 | U | 0.0021 | U | 0.0019 | |
| 8:2 Fluorotelomer sulfonic acid (8:2 FTSA) | 39108-34-4 | -- | mg/kg | 0.0019 | U | 0.002 | U | 0.0017 | U | 0.0019 | U | 0.0013 | J | 0.0015 | |
| N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA) | 2991-50-6 | -- | mg/kg | 0.0019 | U | 0.002 | U | 0.0017 | U | 0.0019 | U | 0.0021 | U | 0.0019 | |
| N-Methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) | 2355-31-9 | -- | mg/kg | 0.0019 | U | 0.002 | U | 0.0017 | U | 0.0019 | U | 0.0021 | U | 0.0019 | |
| Perfluorobutane sulfonic acid (PFBS) | 375-73-5 | 1.9 (R) 25 (I/C) | mg/kg | 0.00093 | U | 0.00065 | J | 0.00085 | U | 0.00093 | U | 0.0011 | U | 0.0025 | |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | -- | mg/kg | 0.00093 | U | 0.0011 | | 0.00085 | U | 0.00093 | U | 0.0011 | U | 0.00093 | |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | -- | mg/kg | 0.00093 | U | 0.0011 | | 0.00085 | U | 0.00093 | U | 0.0022 | | 0.00093 | |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | -- | mg/kg | 0.00093 | U | 0.001 | U | 0.00085 | U | 0.00093 | U | 0.0012 | | 0.00093 | |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | -- | mg/kg | 0.00093 | U | 0.0007 | J | 0.00085 | U | 0.00093 | U | 0.0011 | U | 0.00084 | |
| Perfluorohexane sulfonic acid (PFHxS) | 355-46-4 | 0.13 (R) 1.6 (I/C) | mg/kg | 0.00093 | U | 0.0065 | | 0.00046 | J | 0.00093 | U | 0.0011 | U | 0.082 | |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | -- | mg/kg | 0.00093 | U | 0.0043 | | 0.00085 | U | 0.00093 | U | 0.0011 | U | 0.003 | |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 0.019 (R) 0.25 (I/C) | mg/kg | 0.00093 | U | 0.001 | U | 0.00085 | U | 0.00093 | U | 0.0011 | U | 0.00093 | |
| Perfluorooctane sulfonic acid (PFOS) | 1763-23-1 | 0.013 (R) 0.16 (I/C) | mg/kg | 0.0031 | | 0.015 | | 0.0027 | | 0.0017 | | 0.0035 | | 0.76 | |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 0.019 (R) 0.25 (I/C) | mg/kg | 0.00093 | U | 0.0009 | J | 0.00085 | U | 0.00093 | U | 0.0011 | U | 0.0027 | |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | -- | mg/kg | 0.00093 | U | 0.002 | | 0.00085 | U | 0.00093 | U | 0.0011 | U | 0.00098 | |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | -- | mg/kg | 0.00093 | U | 0.001 | U | 0.00085 | U | 0.00093 | U | 0.0011 | U | 0.00093 | |
| Perfluorotridecanoic acid (PFTTrDA) | 72629-94-8 | -- | mg/kg | 0.00093 | U | 0.001 | U | 0.00085 | U | 0.00093 | U | 0.0011 | U | 0.00093 | |
| Perfluoroundecanoic acid (PFUdA) | 2058-94-8 | -- | mg/kg | 0.00093 | U | 0.001 | U | 0.00085 | U | 0.00093 | U | 0.0016 | J | 0.00093 | |

Table 10-4 - Soil PFAS SI and Baseline Analytical Results
 PFAS RI QAPP
 Yakima Training Center
 Yakima, Washington



| AOPI Location Sample/Duplicate ID Sample Date Sample Type Matrix | | | | SELAH AIRSTRIP | | | | | | | | | |
|---|------------|--------------------------|-------|-------------------------|------|-------------------------|------|-------------------------|------|-------------------------|------|-------------------------|------|
| | | | | YTC-SELAH-1-SO | | YTC-SELAH-2 | | YTC-SELAH-3 | | YTC-SELAH-4 | | YTC-SELAH-5 | |
| | | | | YTC-SELAH-1-SO-092220 | | YTC-SELAH-2-SO-092220 | | YTC-SELAH-3-SO-092220 | | YTC-SELAH-4-SO-092220 | | YTC-SELAH-5-SO-092220 | |
| | | | | 09/22/2020 N Soil | | 09/22/2020 N Soil | | 09/22/2020 N Soil | | 09/22/2020 N Soil | | 09/22/2020 N Soil | |
| Analyte | CAS | OSD Risk Screening Level | Units | Result | Qual |
| PFAS | | | | | | | | | | | | | |
| 6:2 Fluorotelomer sulfonic acid (6:2 FTSA) | 27619-97-2 | -- | mg/kg | 0.0021 | U | 0.0019 | U | 0.0017 | U | 0.002 | U | 0.0019 | U |
| 8:2 Fluorotelomer sulfonic acid (8:2 FTSA) | 39108-34-4 | -- | mg/kg | 0.0021 | U | 0.0019 | U | 0.0017 | U | 0.002 | U | 0.0019 | U |
| N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA) | 2991-50-6 | -- | mg/kg | 0.0021 | U | 0.0019 | U | 0.0017 | U | 0.002 | U | 0.0019 | U |
| N-Methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) | 2355-31-9 | -- | mg/kg | 0.0021 | U | 0.0019 | U | 0.0017 | U | 0.002 | U | 0.0019 | U |
| Perfluorobutane sulfonic acid (PFBS) | 375-73-5 | 1.9 (R) 25 (I/C) | mg/kg | 0.0011 | U | 0.00097 | U | 0.00087 | U | 0.001 | U | 0.00095 | U |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | -- | mg/kg | 0.0011 | U | 0.00097 | U | 0.00087 | U | 0.001 | U | 0.00095 | U |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | -- | mg/kg | 0.0011 | U | 0.00097 | U | 0.00087 | U | 0.001 | U | 0.00095 | U |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | -- | mg/kg | 0.0011 | U | 0.00097 | U | 0.00087 | U | 0.001 | U | 0.00095 | U |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | -- | mg/kg | 0.0011 | U | 0.00097 | U | 0.00087 | U | 0.001 | U | 0.00095 | U |
| Perfluorohexane sulfonic acid (PFHxS) | 355-46-4 | 0.13 (R) 1.6 (I/C) | mg/kg | 0.011 | | 0.00072 | J | 0.00087 | U | 0.001 | U | 0.00095 | U |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | -- | mg/kg | 0.002 | | 0.00097 | U | 0.00087 | U | 0.001 | U | 0.00095 | U |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 0.019 (R) 0.25 (I/C) | mg/kg | 0.0011 | U | 0.00097 | U | 0.00087 | U | 0.001 | U | 0.00095 | U |
| Perfluorooctane sulfonic acid (PFOS) | 1763-23-1 | 0.013 (R) 0.16 (I/C) | mg/kg | 0.12 | | 0.071 | | 0.0038 | | 0.001 | U | 0.00095 | U |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 0.019 (R) 0.25 (I/C) | mg/kg | 0.002 | | 0.00097 | U | 0.00087 | U | 0.001 | U | 0.00095 | U |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | -- | mg/kg | 0.0011 | U | 0.00097 | U | 0.00087 | U | 0.001 | U | 0.00095 | U |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | -- | mg/kg | 0.0011 | U | 0.00097 | U | 0.00087 | U | 0.001 | U | 0.00095 | U |
| Perfluorotridecanoic acid (PFTTrDA) | 72629-94-8 | -- | mg/kg | 0.0011 | U | 0.00097 | U | 0.00087 | U | 0.001 | U | 0.00095 | U |
| Perfluoroundecanoic acid (PFUdA) | 2058-94-8 | -- | mg/kg | 0.0011 | U | 0.00097 | U | 0.00087 | U | 0.001 | U | 0.00095 | U |

Table 10-4 - Soil PFAS SI and Baseline Analytical Results
 PFAS RI QAPP
 Yakima Training Center
 Yakima, Washington



| AOPI Location Sample/Duplicate ID Sample Date Sample Type Matrix | | | | VEHICLE MAINTENANCE SHOP AFFF STORAGE AREA | | | | | |
|---|------------|--------------------------|-------|--|------|-------------------------|------|--------------------------|------|
| | | | | YTC-VMS-1 | | YTC-VMS-2 | | | |
| | | | | YTC-VMS-1-SO-092320 | | YTC-VMS-2-SO-092320 | | YTC-FD-1-SO-092320 | |
| | | | | 09/23/2020 N Soil | | 09/23/2020 N Soil | | 09/23/2020 FD Soil | |
| Analyte | CAS | OSD Risk Screening Level | Units | Result | Qual | Result | Qual | Result | Qual |
| PFAS | | | | | | | | | |
| 6:2 Fluorotelomer sulfonic acid (6:2 FTSA) | 27619-97-2 | -- | mg/kg | 0.0021 | U | 0.0018 | UJ | 0.0022 | U |
| 8:2 Fluorotelomer sulfonic acid (8:2 FTSA) | 39108-34-4 | -- | mg/kg | 0.0021 | U | 0.0018 | U | 0.0022 | U |
| N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA) | 2991-50-6 | -- | mg/kg | 0.0021 | U | 0.0018 | U | 0.0022 | U |
| N-Methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) | 2355-31-9 | -- | mg/kg | 0.0021 | U | 0.0018 | U | 0.0022 | U |
| Perfluorobutane sulfonic acid (PFBS) | 375-73-5 | 1.9 (R) 25 (I/C) | mg/kg | 0.0011 | U | 0.0009 | U | 0.0011 | U |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | -- | mg/kg | 0.0011 | U | 0.0009 | U | 0.0011 | U |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | -- | mg/kg | 0.0011 | U | 0.0009 | U | 0.0011 | U |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | -- | mg/kg | 0.0011 | U | 0.0009 | U | 0.0011 | U |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | -- | mg/kg | 0.0011 | U | 0.0009 | U | 0.0011 | U |
| Perfluorohexane sulfonic acid (PFHxS) | 355-46-4 | 0.13 (R) 1.6 (I/C) | mg/kg | 0.0011 | U | 0.0009 | U | 0.0011 | U |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | -- | mg/kg | 0.0011 | U | 0.0009 | U | 0.0011 | U |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 0.019 (R) 0.25 (I/C) | mg/kg | 0.0011 | U | 0.0009 | U | 0.0011 | U |
| Perfluorooctane sulfonic acid (PFOS) | 1763-23-1 | 0.013 (R) 0.16 (I/C) | mg/kg | 0.0011 | U | 0.0018 | | 0.0018 | |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 0.019 (R) 0.25 (I/C) | mg/kg | 0.0011 | U | 0.0009 | U | 0.0011 | U |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | -- | mg/kg | 0.0011 | U | 0.0009 | U | 0.0011 | U |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | -- | mg/kg | 0.0011 | U | 0.0009 | U | 0.0011 | U |
| Perfluorotridecanoic acid (PFTTrDA) | 72629-94-8 | -- | mg/kg | 0.0011 | U | 0.0009 | U | 0.0011 | U |
| Perfluoroundecanoic acid (PFUdA) | 2058-94-8 | -- | mg/kg | 0.0011 | U | 0.0009 | U | 0.0011 | U |

Notes:

1. **Bolded** values indicate the result was detected greater than the limit of detection.
2. All laboratory reported results in nanograms per gram (ng/g) were converted to milligrams per kilogram (mg/kg).
3. Data are compared to the 2022 Office of the Secretary of Defense (OSD) risk screening levels, (OSD. 2022. Memorandum: Investigating Per- and Polyfluoroalkyl Substances within the Department of Defense Cleanup Program. July).
4. Grey shaded values indicate the result was detected greater than or equal to the OSD risk screening level for the residential scenario. Italicized values indicate the result was detected greater than the OSD risk screening level for the industrial/commercial and residential scenario.

Acronyms/Abbreviations:

- = not applicable/not analyzed
- % = percent
- AOP1 = Area of Potential Interest
- CAS = Chemical Abstracts Service number
- FD = field duplicate sample
- I/C = industrial/commercial receptor scenario
- ID = identification
- mg/kg = milligrams per kilogram (parts per million)
- N = primary sample
- PFAS = per- and polyfluoroalkyl substances
- R = residential receptor scenario

| Qualifier | Description |
|-----------|--|
| J | The analyte was positively identified; however the associated numerical value is an estimated concentration only. |
| U | The analyte was analyzed for but the result was not detected above the limit of quantitation (LOQ). |
| UJ | The analyte was analyzed for but was not detected. The reported limit of quantitation (LOQ) is approximate and may be inaccurate or imprecise. |

Table 10-5 - Surface Water PFAS SI and Baseline Analytical Results
 PFAS RI QAPP
 Yakima Training Center
 Yakima, Washington



| Analyte | AOPI | | | YTC-SW-04 | | | | YTC-SW-08 | | SELAH CREEK | | SELAH SPRINGS | | | | | |
|--|-------------|--------------|------------|---------------------|--|------------------|-----------------------|-----------------------|--|---------------|---------------|---------------|---------------|---------------|---|-------------|--|
| | CAS | OSD Tapwater | Units | YTC-SW-04 | | YTC-SW-08 | | YTC-SELAHCR-1 | | YTC-SELAHSP-1 | | | | | | | |
| | | | | Sample/Duplicate ID | | Sample Date | | Sample Type | | Sample Date | | Sample Type | | Sample Date | | Sample Type | |
| | | | | YTC-SW-04-020723 | YTC-SW-04-020723 / YTC-FD-01-SW-020723 | YTC-SW-08-020723 | YTC-SELAHCR-SW-092220 | YTC-SELAHSP-SW-092220 | YTC-SELAHSP-SW-092220 / YTC-FD-1-SW-092220 | | | | | | | | |
| Matrix | 02/07/2023 | 02/07/2023 | 02/07/2023 | 09/22/2020 | 09/22/2020 | 09/22/2020 | Surface Water | Surface Water | Surface Water | Surface Water | Surface Water | Surface Water | Surface Water | Surface Water | | | |
| Result | Qual | Result | Qual | Result | Qual | Result | Qual | Result | Qual | Result | Qual | Result | Qual | | | | |
| PFAS | | | | | | | | | | | | | | | | | |
| 4:2 Fluorotelomer sulfonate (4:2 FTS) | 757124-72-4 | -- | ng/L | 0.60 | J | 0.61 | J | 1.8 | U | -- | | -- | | -- | | | |
| 6:2 Fluorotelomer sulfonic acid (6:2 FTSA) | 27619-97-2 | -- | ng/L | 180 | | 200 | | 2.7 | U | 7.8 | U | 6.8 | U | 6.7 | U | | |
| 8:2 Fluorotelomer sulfonic acid (8:2 FTSA) | 39108-34-4 | -- | ng/L | 81 | | 80 | | 2.7 | U | 7.8 | U | 6.8 | U | 6.7 | U | | |
| N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA) | 2991-50-6 | -- | ng/L | 2.7 | U | 2.6 | U | 2.7 | U | 7.8 | U | 6.8 | U | 6.7 | U | | |
| N-Methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) | 2355-31-9 | -- | ng/L | 1.8 | U | 1.7 | U | 1.8 | U | 7.8 | U | 6.8 | U | 6.7 | U | | |
| Perfluorobutane sulfonic acid (PFBS) | 375-73-5 | 601 | ng/L | 26 | | 29 | | 1.8 | U | 3.9 | U | 3.4 | U | 3.4 | U | | |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | -- | ng/L | 22 | | 21 | | 4.5 | U | 3.9 | U | 3.4 | U | 3.4 | U | | |
| Perfluorodecane sulfonic acid (PFDS) | 335-77-3 | -- | ng/L | 2.5 | | 2.4 | | 1.8 | U | -- | | -- | | -- | | | |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | -- | ng/L | 11 | | 11 | | 1.8 | U | 3.9 | U | 3.4 | U | 3.4 | U | | |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | -- | ng/L | 1.9 | J | 1.9 | | 1.8 | UX | 3.9 | U | 3.4 | U | 3.4 | U | | |
| Perfluoroheptane sulfonic acid (PFHpS) | 375-92-8 | -- | ng/L | 41 | | 43 | | 1.8 | U | -- | | -- | | -- | | | |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | -- | ng/L | 59 | | 59 | | 1.8 | U | 3.9 | U | 3.4 | U | 3.4 | U | | |
| Perfluorohexane sulfonic acid (PFHxS) | 355-46-4 | 39 | ng/L | 360 | | 360 | | 1.8 | U | 3.9 | U | 3.4 | U | 3.4 | U | | |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | -- | ng/L | 160 | | 170 | | 1.8 | U | 3.9 | U | 3.4 | U | 3.4 | U | | |
| Perfluorononane sulfonic acid (PFNS) | 68259-12-1 | -- | ng/L | 3.8 | | 3.4 | | 1.8 | U | -- | | -- | | -- | | | |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 6 | ng/L | 12 | | 12 | | 1.8 | U | 3.9 | U | 3.4 | U | 3.4 | U | | |
| Perfluorooctane sulfonamide (PFOSA) | 754-91-6 | -- | ng/L | 11 | J+ | 12 | J+ | 1.8 | U | -- | | -- | | -- | | | |
| Perfluorooctane sulfonic acid (PFOS) | 1763-23-1 | 4 | ng/L | 2900 | | 2900 | | 1.9 | U | 3.9 | U | 3.4 | U | 3.4 | U | | |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 6 | ng/L | 78 | | 83 | | 1.8 | U | 3.9 | U | 3.4 | U | 3.4 | U | | |
| Perfluoropentane sulfonic acid (PFPeSA) | 2706-91-4 | -- | ng/L | 39 | | 41 | | 1.8 | U | -- | | -- | | -- | | | |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | -- | ng/L | 79 | | 78 | | 1.8 | U | 3.9 | U | 3.4 | U | 3.4 | U | | |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | -- | ng/L | 1.8 | U | 1.7 | U | 1.8 | UX | 3.9 | U | 3.4 | U | 3.4 | U | | |
| Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 | -- | ng/L | 1.8 | U | 1.7 | U | 1.8 | UX | 3.9 | U | 3.4 | U | 3.4 | U | | |
| Perfluoroundecanoic acid (PFUdA) | 2058-94-8 | -- | ng/L | 2.3 | | 2.4 | | 1.8 | U | 3.9 | U | 3.4 | U | 3.4 | U | | |

Notes:

1. **Bolded** values indicate the result was detected greater than the limit of detection.
2. Grey shaded values indicate the result was detected greater than the 2022 Office of the Secretary of Defense (OSD) risk screening levels, (OSD. 2022. Memorandum: Investigating Per- and Polyfluoroalkyl Substances within the Department of Defense Cleanup Program. July).

Acronyms/Abbreviations:

- = not applicable
- % = percent
- AOPI = Area of Potential Interest
- CAS = Chemical Abstracts Service number
- FD = field duplicate sample
- ID = identification
- N = primary sample
- ng/L = nanograms per liter (parts per trillion)
- PFAS = per- and polyfluoroalkyl substances

| Qualifier | Description |
|-----------|---|
| J | The analyte was positively identified; however the associated numerical value is an estimated concentration only. |
| J+ | The result is an estimated quantity; the result may be biased high. |
| U | The analyte was analyzed for but the result was not detected above the limit of quantitation (LOQ). |

| AOPI Location | | | | YTC-SED-01 | | | | | | | | | | | | YTC-SED-02 | | YTC-SED-03 | | YTC-SED-04 | | | | YTC-SED-05 | |
|--|-------------|--------------------------|-------|-----------------------------|------|---------|------|-----------------------------|------|----------------|------|-----------------------------|------|----------------|------|------------------------------|------|--|--|-------------------|--|--|--|------------|--|
| | | | | YTC-SED-01-020723 | | | | YTC-SED-02-020723 | | | | YTC-SED-03-020723 | | | | YTC-SED-04-020723 | | YTC-SED-04-020723 / YTC-FD-01-SED-020723 | | YTC-SED-05-020723 | | | | | |
| Sample/Duplicate ID | | | | 02/07/2023 N Sediment | | | | 02/07/2023 N Sediment | | | | 02/07/2023 N Sediment | | | | 02/07/2023 FD Sediment | | 02/07/2023 N Sediment | | | | | | | |
| Sample Date | | | | | | | | | | | | | | | | | | | | | | | | | |
| Sample Type | | | | | | | | | | | | | | | | | | | | | | | | | |
| Matrix | | | | | | | | | | | | | | | | | | | | | | | | | |
| Analyte | CAS | OSD Risk Screening Level | Units | Result | Qual | Result | Qual | Result | Qual | Result | Qual | Result | Qual | Result | Qual | Result | Qual | | | | | | | | |
| PFAS | | | | | | | | | | | | | | | | | | | | | | | | | |
| 4:2 Fluorotelomer sulfonate (4:2 FTS) | 757124-72-4 | -- | mg/kg | 0.0024 | U | 0.0029 | U | 0.0023 | U | 0.0026 | U | 0.0022 | U | 0.0022 | U | 0.0022 | U | | | | | | | | |
| 6:2 Fluorotelomer sulfonic acid (6:2 FTSA) | 27619-97-2 | -- | mg/kg | 0.0024 | U | 0.0029 | U | 0.0023 | U | 0.0026 | U | 0.0022 | U | 0.0022 | U | 0.0022 | U | | | | | | | | |
| 8:2 Fluorotelomer sulfonic acid (8:2 FTSA) | 39108-34-4 | -- | mg/kg | 0.0036 | U | 0.0044 | U | 0.0034 | U | 0.0038 | U | 0.0033 | U | 0.0033 | U | 0.0033 | U | | | | | | | | |
| N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA) | 2991-50-6 | -- | mg/kg | 0.0024 | U | 0.0029 | U | 0.0023 | U | 0.0026 | U | 0.0022 | U | 0.0022 | U | 0.0022 | U | | | | | | | | |
| N-Methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) | 2355-31-9 | -- | mg/kg | 0.0024 | U | 0.0029 | U | 0.0023 | U | 0.0026 | U | 0.0022 | U | 0.0022 | U | 0.0022 | U | | | | | | | | |
| Perfluorobutane sulfonic acid (PFBS) | 375-73-5 | 1.9 (R) 25 (I/C) | mg/kg | 0.0024 | U | 0.0029 | U | 0.0023 | U | 0.0026 | U | 0.0022 | U | 0.0022 | U | 0.0022 | U | | | | | | | | |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | -- | mg/kg | 0.0024 | U | 0.0029 | U | 0.0023 | U | 0.0026 | U | 0.0022 | U | 0.0022 | U | 0.0022 | U | | | | | | | | |
| Perfluorodecane sulfonic acid (PFDS) | 335-77-3 | -- | mg/kg | 0.00073 | U | 0.00087 | U | 0.00069 | U | 0.00077 | U | 0.00066 | U | 0.00067 | U | 0.00067 | U | | | | | | | | |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | -- | mg/kg | 0.00073 | U | 0.00087 | U | 0.00024 | J | 0.00077 | U | 0.00025 | J | 0.00067 | U | 0.00067 | U | | | | | | | | |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | -- | mg/kg | 0.00073 | U | 0.00087 | U | 0.00069 | U | 0.00077 | U | 0.00039 | J | 0.0003 | J | 0.00067 | U | | | | | | | | |
| Perfluoroheptane sulfonic acid (PFHpS) | 375-92-8 | -- | mg/kg | 0.00073 | U | 0.00087 | U | 0.00069 | U | 0.00077 | U | 0.00066 | U | 0.00067 | U | 0.00067 | U | | | | | | | | |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | -- | mg/kg | 0.00073 | U | 0.00087 | U | 0.00069 | U | 0.00077 | U | 0.00066 | U | 0.00067 | U | 0.00067 | U | | | | | | | | |
| Perfluorohexane sulfonic acid (PFHxS) | 355-46-4 | 0.13 (R) 1.6 (I/C) | mg/kg | 0.00073 | U | 0.00087 | U | 0.00069 | U | 0.00037 | J | 0.00068 | J | 0.00029 | J | 0.00067 | U | | | | | | | | |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | -- | mg/kg | 0.00073 | U | 0.00087 | U | 0.00069 | U | 0.00026 | J | 0.00047 | J | 0.00067 | U | 0.00067 | U | | | | | | | | |
| Perfluorononane sulfonic acid (PFNS) | 68259-12-1 | -- | mg/kg | 0.00073 | U | 0.00087 | U | 0.00069 | U | 0.00077 | U | 0.00066 | U | 0.00067 | U | 0.00067 | U | | | | | | | | |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 0.019 (R) 0.25 (I/C) | mg/kg | 0.00073 | U | 0.00087 | U | 0.00069 | U | 0.00077 | U | 0.00066 | U | 0.00067 | U | 0.00067 | U | | | | | | | | |
| Perfluorooctane sulfonamide (PFOSA) | 754-91-6 | -- | mg/kg | 0.00073 | U | 0.00087 | U | 0.00069 | U | 0.00077 | U | 0.00066 | U | 0.00067 | U | 0.00067 | U | | | | | | | | |
| Perfluorooctane sulfonic acid (PFOS) | 1763-23-1 | 0.013 (R) 0.16 (I/C) | mg/kg | 0.00073 | U | 0.00087 | U | 0.001 | J | 0.0052 | J | 0.0094 | J | 0.0014 | J | 0.00067 | U | | | | | | | | |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 0.019 (R) 0.25 (I/C) | mg/kg | 0.00073 | U | 0.00087 | U | 0.00069 | U | 0.00077 | U | 0.00025 | J | 0.00067 | U | 0.00067 | U | | | | | | | | |
| Perfluoropentane sulfonic acid (PFPeSA) | 2706-91-4 | -- | mg/kg | 0.0036 | U | 0.0044 | U | 0.0034 | U | 0.0038 | U | 0.0033 | U | 0.0033 | U | 0.0033 | U | | | | | | | | |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | -- | mg/kg | 0.00073 | U | 0.00087 | U | 0.00069 | U | 0.00059 | J | 0.0011 | J | 0.00067 | U | 0.00067 | U | | | | | | | | |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | -- | mg/kg | 0.00073 | U | 0.00087 | U | 0.00069 | U | 0.00077 | U | 0.00066 | U | 0.00067 | U | 0.00067 | U | | | | | | | | |
| Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 | -- | mg/kg | 0.00073 | U | 0.00087 | U | 0.00069 | U | 0.00077 | U | 0.00066 | U | 0.00067 | U | 0.00067 | U | | | | | | | | |
| Perfluoroundecanoic acid (PFUdA) | 2058-94-8 | -- | mg/kg | 0.00073 | U | 0.00087 | U | 0.00069 | U | 0.00077 | U | 0.00066 | U | 0.00067 | U | 0.00067 | U | | | | | | | | |

Table 10-6 - Sediment PFAS SI and Baseline Analytical Results
 PFAS RI QAPP
 Yakima Training Center
 Yakima, Washington



| AOPI Location Sample/Duplicate ID Sample Date Sample Type Matrix | | | | YTC-SED-06 | | YTC-SED-07 | | YTC-SED-08 | |
|---|-------------|----------------------|-------|-----------------------------|-----|-----------------------------|-------|-----------------------------|------|
| | | | | YTC-SED-06-020723 | | YTC-SED-07-020723 | | YTC-SED-08-020723 | |
| | | | | 02/07/2023 N Sediment | | 02/07/2023 N Sediment | | 02/07/2023 N Sediment | |
| | | | | Analyte | CAS | OSD Risk Screening Level | Units | Result | Qual |
| PFAS | | | | | | | | | |
| 4:2 Fluorotelomer sulfonate (4:2 FTS) | 757124-72-4 | -- | mg/kg | 0.0023 | U | 0.0028 | U | 0.0032 | U |
| 6:2 Fluorotelomer sulfonic acid (6:2 FTSA) | 27619-97-2 | -- | mg/kg | 0.0023 | U | 0.0028 | U | 0.0032 | U |
| 8:2 Fluorotelomer sulfonic acid (8:2 FTSA) | 39108-34-4 | -- | mg/kg | 0.0034 | U | 0.0041 | U | 0.0047 | U |
| N-Ethyl perfluorooctane sulfonamidoacetic acid (EtFOSAA) | 2991-50-6 | -- | mg/kg | 0.0023 | U | 0.0028 | U | 0.0032 | U |
| N-Methylperfluorooctane sulfonamidoacetic acid (MeFOSAA) | 2355-31-9 | -- | mg/kg | 0.0023 | U | 0.0028 | U | 0.0032 | U |
| Perfluorobutane sulfonic acid (PFBS) | 375-73-5 | 1.9 (R) 25 (I/C) | mg/kg | 0.0023 | U | 0.0028 | U | 0.0032 | U |
| Perfluorobutanoic acid (PFBA) | 375-22-4 | -- | mg/kg | 0.0023 | U | 0.0028 | U | 0.0032 | U |
| Perfluorodecane sulfonic acid (PFDS) | 335-77-3 | -- | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |
| Perfluorodecanoic acid (PFDA) | 335-76-2 | -- | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |
| Perfluorododecanoic acid (PFDoA) | 307-55-1 | -- | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |
| Perfluoroheptane sulfonic acid (PFHpS) | 375-92-8 | -- | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |
| Perfluoroheptanoic acid (PFHpA) | 375-85-9 | -- | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |
| Perfluorohexane sulfonic acid (PFHxS) | 355-46-4 | 0.13 (R) 1.6 (I/C) | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |
| Perfluorohexanoic acid (PFHxA) | 307-24-4 | -- | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |
| Perfluorononane sulfonic acid (PFNS) | 68259-12-1 | -- | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |
| Perfluorononanoic acid (PFNA) | 375-95-1 | 0.019 (R) 0.25 (I/C) | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |
| Perfluorooctane sulfonamide (PFOSA) | 754-91-6 | -- | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |
| Perfluorooctane sulfonic acid (PFOS) | 1763-23-1 | 0.013 (R) 0.16 (I/C) | mg/kg | 0.00027 | J | 0.004 | | 0.00095 | U |
| Perfluorooctanoic acid (PFOA) | 335-67-1 | 0.019 (R) 0.25 (I/C) | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |
| Perfluoropentane sulfonic acid (PFPeSA) | 2706-91-4 | -- | mg/kg | 0.0034 | U | 0.0041 | U | 0.0047 | U |
| Perfluoropentanoic acid (PFPeA) | 2706-90-3 | -- | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |
| Perfluorotetradecanoic acid (PFTeDA) | 376-06-7 | -- | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |
| Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 | -- | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |
| Perfluoroundecanoic acid (PFUdA) | 2058-94-8 | -- | mg/kg | 0.00068 | U | 0.00083 | U | 0.00095 | U |

Table 10-6 - Sediment PFAS SI and Baseline Analytical Results

PFAS RI QAPP
Yakima Training Center
Yakima, Washington



Notes:

1. **Bolded** values indicate the result was detected greater than the limit of detection.
2. All laboratory reported results in nanograms per gram (ng/g) were converted to milligrams per kilogram (mg/kg).
3. Data are compared to the 2022 Office of the Secretary of Defense (OSD) risk screening levels, (OSD. 2022. Memorandum: Investigating Per- and Polyfluoroalkyl Substances within the Department of Defense Cleanup Program. July).
4. Grey shaded values indicate the result was detected greater than or equal to the OSD risk screening level for the residential scenario. Italicized values indicate the result was detected greater than the OSD risk screening level for the industrial/commercial and residential scenario.

Acronyms/Abbreviations:

- = not applicable/not analyzed
- % = percent
- AOPI = Area of Potential Interest
- CAS = Chemical Abstracts Service number
- FD = field duplicate sample
- I/C = industrial/commercial receptor scenario
- ID = identification
- mg/kg = milligrams per kilogram (parts per million)
- N = primary sample
- PFAS = per- and polyfluoroalkyl substances
- R = residential receptor scenario

| Qualifier | Description |
|-----------|---|
| J | The analyte was positively identified; however the associated numerical value is an estimated concentration only. |
| U | The analyte was analyzed for but the result was not detected above the limit of quantitation (LOQ). |

APPENDICES

Appendix A
Primary Laboratory (AEL) Certifications and Standard Operating
Procedures



PERRY JOHNSON LABORATORY ACCREDITATION, INC.

Certificate of Accreditation

Perry Johnson Laboratory Accreditation, Inc.
has assessed the Organization of:

***Advanced Environmental Laboratories, Inc.
6681 Southpoint Parkway, Jacksonville, FL 32216***

(Hereinafter called the Organization) and hereby declares that Organization has met the requirements of ISO/IEC 17025:2017 General Requirements for the competence of Testing and Calibration Laboratories and the United States Department of Defense Environmental Laboratory Accreditation Program (DoD-ELAP) requirements identified within the DoD/DOE Quality Systems Manual (DoD/DOE QSM) Version 5.4 October 2021 and is accredited in accordance with the:

United States Department of Defense Environmental Laboratory Accreditation Program (DoD-ELAP)

This accreditation demonstrates the technical competence for the defined scope and the operation of a laboratory quality management system
(as outlined by the joint ISO-ILAC-IAF Communiqué dated April 2017):

Environmental Testing (As detailed in the supplement)

Accreditation claims for such activities shall only be made from the addresses referenced within this certificate. This Accreditation is granted subject to the system rules governing the Accreditation referred to above, and the Organization hereby covenants with the Accreditation Body's duty to observe and comply with the said rules.

For PJLA

Tracy Szorszen
President

Initial Accreditation Date:

March 07, 2019

Issue Date:

August 04, 2021

Expiration Date

August 04, 2023

Revision Date:

November 21, 2022

Accreditation No:

104509

Certificate No:

L21-470-R2

Perry Johnson Laboratory
Accreditation, Inc. (PJLA)
755 W. Big Beaver, Suite 1325
Troy, Michigan 48084

The validity of this certificate is maintained through ongoing assessments based on a continuous accreditation cycle. The validity of this certificate should be confirmed through the PJLA website: www.pjllabs.com



Certificate of Accreditation: Supplement

Advanced Environmental Laboratories, Inc.

6681 Southpoint Parkway, Jacksonville, FL 32216

Contact Name: Todd Romero Phone 352-538-4939, Heather Quilal-lan Phone: 904-363-9350

Accreditation is granted to the facility to perform the following testing:

Code

Bacteriological

| | |
|--|----------|
| SM 9223B (2016) by Presence/Absence (Quanti-Tray) | 20213610 |
| Aqueous | |
| Coliform (Total) | 2500 |
| Drinking Water | |
| Coliform (Total) | 2500 |

General Chemistry

| | |
|---|----------|
| EPA 1020C by Setaflash Closed-Cup Apparatus | 10117154 |
| Aqueous | |
| Ignitability (Flashpoint) | 1780 |
| Solid | |
| Ignitability (Flashpoint) | 1780 |
| EPA 1030 by Manual Ignition (Burn Mold/ Burn Rate) | 10117212 |
| Solid | |
| Ignitability (Flashpoint) | 1780 |
| EPA 150.1 by Ion Selective Electrode (ISE) | 10008205 |
| Aqueous | |
| pH | 1900 |
| EPA 160.1 by Gravimetry | 10009004 |
| Aqueous | |
| Solids (Total Dissolved, TDS, Residue, Filterable) | 1955 |
| EPA 160.2 by Gravimetry | 10009402 |
| Aqueous | |
| Solids (Total Suspended, TSS, Non-Filterable Residue) | 1960 |
| EPA 160.3 by Gravimetry | 10009800 |
| Aqueous | |
| Solids (Total, TS) | 1950 |
| Solid | |
| Solids (Total, TS) | 1950 |
| EPA 1650C by Coulometry | 10125005 |
| Aqueous | |
| Absorbable Organic Halides (AOX) | 2045 |
| EPA 1664B by Gravimetry | 10260628 |
| Aqueous | |
| n-Hexane Extractable Material (HEM) Oil & Grease (O&G) | 1803 |
| Total Petroleum Hydrocarbons (TPH) | 2050 |
| EPA 180.1 by Nephelometry | 10011800 |
| Aqueous | |
| Turbidity | 2055 |



Certificate of Accreditation: Supplement

Advanced Environmental Laboratories, Inc.

6681 Southpoint Parkway, Jacksonville, FL 32216

Contact Name: Todd Romero Phone 352-538-4939, Heather Quilal-lan Phone: 904-363-9350

Accreditation is granted to the facility to perform the following testing:

| | Code |
|--|----------|
| General Chemistry | |
| EPA 310.1 by Titrimetry | 10054601 |
| Aqueous | |
| Alkalinity (as CaCO ₃) | 1505 |
| EPA 410.4 by Spectrophotometry | 10077404 |
| Aqueous | |
| Chemical Oxygen Demand (COD) | 1565 |
| EPA 415.1 by TOC Analyzer | 10078407 |
| Aqueous | |
| Total Organic Carbon (TOC) | 2040 |
| Drinking Water | |
| Dissolved Organic Carbon (DOC) | 1710 |
| Total Organic Carbon (TOC) | 2040 |
| EPA 9040 by Ion Selective Electrode (ISE) | 10244403 |
| Aqueous | |
| pH | 1900 |
| Solid | |
| pH | 1900 |
| EPA 9040C by Electrometry | 10244403 |
| Aqueous | |
| pH (Corrosivity) | 1625 |
| EPA 9045D by Ion Selective Electrode (ISE) | 10198455 |
| Solid | |
| pH (Corrosivity) | 1625 |
| EPA 9060A by TOC Analyzer | 10244823 |
| Solid | |
| Total Organic Carbon (TOC) | 2040 |
| EPA 9095B by Manual Filtration | 10204009 |
| Solid | |
| Paint Filter Liquids Test | 1434 |
| SM 2320B (2011) by Titrimetry | 20045414 |
| Aqueous | |
| Alkalinity (as CaCO ₃) | 1505 |
| SM 2540B (2015) by Gravimetry | 20049212 |
| Aqueous | |
| Solids (Total, TS) | 1950 |
| SM 2540C (2015) by Gravimetry | 20050424 |
| Aqueous | |
| Solids (Total Dissolved, TDS, Residue, Filterable) | 1955 |



Certificate of Accreditation: Supplement

Advanced Environmental Laboratories, Inc.

6681 Southpoint Parkway, Jacksonville, FL 32216

Contact Name: Todd Romero Phone 352-538-4939, Heather Quilal-Ian Phone: 904-363-9350

Accreditation is granted to the facility to perform the following testing:

| | Code |
|--|----------|
| General Chemistry | |
| SM 2540D (2015) by Gravimetry | 20051018 |
| Aqueous | |
| Solids (Total Suspended, TSS, Non-Filterable Residue) | 1960 |
| SM 2540G (2015) by Gravimetry | 20005270 |
| Solid | |
| Solids (Total, TS) | 1950 |
| SM 4500 S D/UV VIS (2011) by Spectrophotometry | 20125615 |
| Aqueous | |
| Sulfide | 2005 |
| SM 5210B (2016) by 5-Day BOD Test | 20135017 |
| Aqueous | |
| Biochemical Oxygen Demand (BOD) | 1530 |
| SM 5310C (2014) by TOC Analyzer (Chemical Oxidation/IR) | 20138834 |
| Aqueous | |
| Dissolved Organic Carbon (DOC) | 1710 |
| Total Inorganic Carbon (TIC) | 1813 |
| Total Organic Carbon (TOC) | 2040 |
| Drinking Water | |
| Dissolved Organic Carbon (DOC) | 1710 |
| Total Organic Carbon (TOC) | 2040 |
| Inorganic | |
| EPA 1631E by Cold Vapor Atomic Fluorescence Spectrophotometry (CVAFS) | 10237204 |
| Aqueous | |
| Mercury (Low Level) | 1095 |
| EPA 200.7 by Inductively Coupled Plasma Optical Emission Spectrometry (ICP/OES) | 10013806 |
| Aqueous | |
| Aluminum | 1000 |
| Antimony | 1005 |
| Arsenic | 1010 |
| Barium | 1015 |
| Beryllium | 1020 |
| Boron | 1025 |
| Cadmium | 1030 |
| Calcium | 1035 |
| Chromium | 1040 |
| Cobalt | 1050 |
| Copper | 1055 |
| Iron | 1070 |



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Inorganic

EPA 200.7 by Inductively Coupled Plasma Optical Emission Spectrometry (ICP/OES) 10013806

Aqueous

| | |
|-------------------------------|------|
| Lead | 1075 |
| Lithium | 1080 |
| Magnesium | 1085 |
| Manganese | 1090 |
| Molybdenum | 1100 |
| Nickel | 1105 |
| Potassium | 1125 |
| Selenium | 1140 |
| Silica (as SiO ₂) | 1990 |
| Silver | 1150 |
| Sodium | 1155 |
| Strontium | 1160 |
| Thallium | 1165 |
| Tin | 1175 |
| Titanium | 1180 |
| Vanadium | 1185 |
| Zinc | 1190 |

EPA 200.8 by Inductively Coupled Plasma Mass Spectrometry (ICP/MS) 10014605

Aqueous

| | |
|------------|------|
| Aluminum | 1000 |
| Antimony | 1005 |
| Arsenic | 1010 |
| Barium | 1015 |
| Beryllium | 1020 |
| Boron | 1025 |
| Cadmium | 1030 |
| Chromium | 1040 |
| Cobalt | 1050 |
| Copper | 1055 |
| Iron | 1070 |
| Lead | 1075 |
| Manganese | 1090 |
| Molybdenum | 1100 |
| Nickel | 1105 |
| Selenium | 1140 |
| Silver | 1150 |



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Inorganic

| | |
|--|----------|
| EPA 200.8 by Inductively Coupled Plasma Mass Spectrometry (ICP/MS) | 10014605 |
| Aqueous | |
| Strontium | 1160 |
| Thallium | 1165 |
| Tin | 1175 |
| Titanium | 1180 |
| Uranium | 1184 |
| Vanadium | 1185 |
| Zinc | 1190 |
| EPA 245.1 by Cold Vapor Atomic Absorption Spectrophotometry (CVAAS) | 10036609 |
| Aqueous | |
| Mercury | 1095 |
| EPA 300.0 by Ion Chromatography (IC) | 10053200 |
| Aqueous | |
| Bromide | 1540 |
| Chloride | 1575 |
| Flouride | 1730 |
| Nitrate (as N) | 1810 |
| Nitrate + Nitrite (as N) (NOX) | 1820 |
| Nitrite (as N) | 1840 |
| Orthophosphate (as P) | 1870 |
| Sulfate | 2000 |
| EPA 6010 by Inductively Coupled Plasma Optical Emission Spectrometry (ICP/OES) | 10155609 |
| Aqueous | |
| Ferric Iron (Fe III, by Calculation) | 1074 |
| EPA 6010C by Inductively Coupled Plasma Optical Emission Spectrometry (ICP/OES) | 10155905 |
| Aqueous | |
| Aluminum | 1000 |
| Antimony | 1005 |
| Arsenic | 1010 |
| Barium | 1015 |
| Beryllium | 1020 |
| Boron | 1025 |
| Cadmium | 1030 |
| Calcium | 1035 |
| Calcium hardness as CaCO ₃ | 1550 |
| Chromium | 1040 |
| Cobalt | 1050 |



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Inorganic

EPA 6010C by Inductively Coupled Plasma Optical Emission Spectrometry (ICP/OES) 10155905

Aqueous

Copper 1055

Ferric Iron (Fe III, by Calculation) 1074

Hardness (Total, as CaCO₃, By Calculation) 1755

Iron 1070

Lead 1075

Lithium 1080

Magnesium 1085

Manganese 1090

Molybdenum 1100

Nickel 1105

Potassium 1125

Selenium 1140

Silica (as SiO₂) 1990

Silver 1150

Sodium 1155

Strontium 1160

Thallium 1165

Tin 1175

Titanium 1180

Vanadium 1185

Zinc 1190

Solid

Aluminum 1000

Antimony 1005

Arsenic 1010

Barium 1015

Beryllium 1020

Boron 1025

Cadmium 1030

Calcium 1035

Chromium 1040

Cobalt 1050

Copper 1055

Iron 1070

Lead 1075

Magnesium 1085



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Inorganic

| | |
|--|----------|
| EPA 6010C by Inductively Coupled Plasma Optical Emission Spectrometry (ICP/OES) | 10155905 |
| Solid | |
| Manganese | 1090 |
| Molybdenum | 1100 |
| Nickel | 1105 |
| Potassium | 1125 |
| Selenium | 1140 |
| Silver | 1150 |
| Sodium | 1155 |
| Strontium | 1160 |
| Thallium | 1165 |
| Tin | 1175 |
| Titanium | 1180 |
| Vanadium | 1185 |
| Zinc | 1190 |
| EPA 6010D by Inductively Coupled Plasma Optical Emission Spectrometry (ICP/OES) | 10155949 |
| Aqueous | |
| Aluminum | 1000 |
| Antimony | 1005 |
| Arsenic | 1010 |
| Barium | 1015 |
| Beryllium | 1020 |
| Boron | 1025 |
| Cadmium | 1030 |
| Calcium | 1035 |
| Calcium hardness as CaCO ₃ | 1550 |
| Chromium | 1040 |
| Cobalt | 1050 |
| Copper | 1055 |
| Ferric Iron (Fe III, by Calculation) | 1074 |
| Hardness (Total, as CaCO ₃ , By Calculation) | 1755 |
| Iron | 1070 |
| Lead | 1075 |
| Lithium | 1080 |
| Magnesium | 1085 |
| Manganese | 1090 |
| Molybdenum | 1100 |
| Nickel | 1105 |



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Inorganic

EPA 6010D by Inductively Coupled Plasma Optical Emission Spectrometry (ICP/OES)

10155949

Aqueous

| | |
|-------------------------------|------|
| Potassium | 1125 |
| Selenium | 1140 |
| Silica (as SiO ₂) | 1990 |
| Silver | 1150 |
| Sodium | 1155 |
| Strontium | 1160 |
| Thallium | 1165 |
| Tin | 1175 |
| Titanium | 1180 |
| Vanadium | 1185 |
| Zinc | 1190 |

Solid

| | |
|------------|------|
| Aluminum | 1000 |
| Antimony | 1005 |
| Arsenic | 1010 |
| Barium | 1015 |
| Beryllium | 1020 |
| Boron | 1025 |
| Cadmium | 1030 |
| Calcium | 1035 |
| Chromium | 1040 |
| Cobalt | 1050 |
| Copper | 1055 |
| Iron | 1070 |
| Lead | 1075 |
| Magnesium | 1085 |
| Manganese | 1090 |
| Molybdenum | 1100 |
| Nickel | 1105 |
| Potassium | 1125 |
| Selenium | 1140 |
| Silver | 1150 |
| Sodium | 1155 |
| Strontium | 1160 |
| Thallium | 1165 |
| Tin | 1175 |



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Inorganic

| | |
|--|----------|
| EPA 6010D by Inductively Coupled Plasma Optical Emission Spectrometry (ICP/OES) | 10155949 |
| Solid | |
| Titanium | 1180 |
| Vanadium | 1185 |
| Zinc | 1190 |
| EPA 6020A by Inductively Coupled Plasma Mass Spectrometry (ICP/MS) | 10156408 |
| Aqueous | |
| Aluminum | 1000 |
| Antimony | 1005 |
| Arsenic | 1010 |
| Barium | 1015 |
| Beryllium | 1020 |
| Cadmium | 1030 |
| Chromium | 1040 |
| Cobalt | 1050 |
| Copper | 1055 |
| Iron | 1070 |
| Lead | 1075 |
| Manganese | 1090 |
| Molybdenum | 1100 |
| Nickel | 1105 |
| Selenium | 1140 |
| Silver | 1150 |
| Strontium | 1160 |
| Thallium | 1165 |
| Tin | 1175 |
| Titanium | 1180 |
| Uranium | 1184 |
| Vanadium | 1185 |
| Zinc | 1190 |
| Solid | |
| Antimony | 1005 |
| Arsenic | 1010 |
| Barium | 1015 |
| Beryllium | 1020 |
| Cadmium | 1030 |
| Chromium | 1040 |
| Cobalt | 1050 |



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Code

Inorganic

EPA 6020A by Inductively Coupled Plasma Mass Spectrometry (ICP/MS) 10156408

| Solid | |
|--------------|------|
| Copper | 1055 |
| Lead | 1075 |
| Manganese | 1090 |
| Molybdenum | 1100 |
| Nickel | 1105 |
| Selenium | 1140 |
| Silver | 1150 |
| Strontium | 1160 |
| Thallium | 1165 |
| Tin | 1175 |
| Titanium | 1180 |
| Vanadium | 1185 |
| Zinc | 1190 |

EPA 6020B by Inductively Coupled Plasma Mass Spectrometry (ICP/MS) 10156408

| Aqueous | |
|----------------|------|
| Aluminum | 1000 |
| Antimony | 1005 |
| Arsenic | 1010 |
| Barium | 1015 |
| Beryllium | 1020 |
| Cadmium | 1030 |
| Chromium | 1040 |
| Cobalt | 1050 |
| Copper | 1055 |
| Iron | 1070 |
| Lead | 1075 |
| Manganese | 1090 |
| Molybdenum | 1100 |
| Nickel | 1105 |
| Selenium | 1140 |
| Silver | 1150 |
| Strontium | 1160 |
| Thallium | 1165 |
| Tin | 1175 |
| Titanium | 1180 |
| Uranium | 1184 |



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Code

Inorganic

| | |
|--|----------|
| EPA 6020B by Inductively Coupled Plasma Mass Spectrometry (ICP/MS) | 10156408 |
| Aqueous | |
| Vanadium | 1185 |
| Zinc | 1190 |
| Solid | |
| Antimony | 1005 |
| Arsenic | 1010 |
| Barium | 1015 |
| Beryllium | 1020 |
| Cadmium | 1030 |
| Chromium | 1040 |
| Cobalt | 1050 |
| Copper | 1055 |
| Lead | 1075 |
| Manganese | 1090 |
| Molybdenum | 1100 |
| Nickel | 1105 |
| Selenium | 1140 |
| Silver | 1150 |
| Strontium | 1160 |
| Thallium | 1165 |
| Tin | 1175 |
| Titanium | 1180 |
| Vanadium | 1185 |
| Zinc | 1190 |
| EPA 7196 by Spectrophotometry | 10162400 |
| Aqueous | |
| Hexavalent Chromium (Cr VI) | 1045 |
| EPA 7470A by Cold Vapor Atomic Absorption Spectrophotometry (CVAAS) | 10165807 |
| Aqueous | |
| Mercury | 1095 |
| EPA 7471A by Cold Vapor Atomic Absorption Spectrophotometry (CVAAS) | 10166208 |
| Solid | |
| Mercury | 1095 |
| EPA 7471B by Cold Vapor Atomic Absorption Spectrophotometry (CVAAS) | 10166457 |
| Solid | |
| Mercury | 1095 |



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| | Code |
|--|----------|
| Inorganic | |
| EPA 9020B by Coulometry | 10194408 |
| Aqueous | |
| Total Organic Halides (TOX) | 2045 |
| EPA 9023 by Coulometry | 10195003 |
| Solid | |
| Extractable Organic Halides (EOX) | 1720 |
| EPA 9056A by Ion Chromatography (IC) | 10199607 |
| Solid | |
| Bromide | 1540 |
| Chloride | 1575 |
| Flouride | 1730 |
| Nitrate (as N) | 1810 |
| Nitrate + Nitrite (as N) (NOX) | 1820 |
| Nitrite (as N) | 1840 |
| Orthophosphate (as P) | 1870 |
| Sulfate | 2000 |
| SM 2340B (2011) by Inductively Coupled Plasma Optical Emission Spectrometry (ICP/OES) | 20046417 |
| Aqueous | |
| Calcium hardness as CaCO ₃ | 1550 |
| Hardness (Total, as CaCO ₃ , By Calculation) | 1755 |
| SM 3500 Cr D (1995) by Spectrophotometry | 20067009 |
| Aqueous | |
| Hexavalent Chromium (Cr VI) | 1045 |
| SM 3500 Fe B (1990) by Spectrometry | 20068819 |
| Aqueous | |
| Ferric Iron (Fe III, by Calculation) | 1074 |
| SM 3500 Fe D (1990) by Spectrophotometry | 20069209 |
| Aqueous | |
| Ferrous Iron (Fe II) | 1073 |
| Organic | |
| Draft EPA Method 1633 by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) | 10123429 |
| Aqueous | |
| 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS) | 9490 |
| 2H,2H,3H,3H-Perfluorodecanoic Acid (7:3 FTCA, 3-Perfluoroheptyl Propanoic Acid) | 9340 |
| 2H,2H,3H,3H-Perfluorooctanoic Acid (5:3 FTCA) | 9338 |
| 4,4,5,5,6,6,6-Heptafluorohexanoi Acid (3:3 FTCA, 3-Perfluoropropyl Propanoic Acid) | 9353 |
| 4,8-dioxa-3H-perfluorononanoic acid (ADONA) | 6951 |
| 4:2 Fluorotelomersulfonic acid (4:2FTS) | 6946 |



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Organic

Draft EPA Method 1633 by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) 10123429

Aqueous

6:2 Fluorotelomersulfonic acid (6:2FTS) 6947

8:2 Fluorotelomersulfonic acid (8:2FTS) 6948

9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS) 6952

Hexafluoropropylene oxide dimer acid (HFPO-DA) 9460

N-ethylperfluorooctanesulfonamide (EtFOSA) 9395

N-ethylperfluorooctanesulfonamidoacetic acid (EtFOSAA) 4847

N-ethylperfluorooctanesulfonamidoethanol (EtFOSE) 9431

N-methylperfluorooctanesulfonamide (MeFOSA) 9433

N-methylperfluorooctanesulfonamidoacetic acid (MeFOSAA) 4846

N-methylperfluorooctanesulfonamidoethanol (MeFOSE) 6949

Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) 6956

Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA) 6957

Perfluoro-3-methoxypropanoic acid (PFMPA) 6965

Perfluoro-4-methoxybutanoic acid (PFMBA) 6966

Perfluorobutanesulfonic acid (PFBS) 6918

Perfluorobutanoic acid (PFBA) 6915

Perfluorodecanesulfonic acid (PFDS) 6920

Perfluorodecanoic acid (PFDA) 6905

Perfluorododecanesulfonic acid (PFDoS) 6923

Perfluorododecanoic acid (PFDoA) 6903

Perfluoroheptanesulfonic acid (PFHpS) 9470

Perfluoroheptanoic acid (PFHpA) 6908

Perfluorohexanesulfonic acid (PFHxS) 6927

Perfluorohexanoic acid (PFHxA) 6913

Perfluorononanesulfonic acid (PFNS) 6929

Perfluorononanoic acid (PFNA) 6906

Perfluorooctanesulfonamide (PFOSA) 6917

Perfluorooctanesulfonic acid (PFOS) 6931

Perfluorooctanoic acid (PFOA) 6912

Perfluoropentanesulfonic acid (PFPeS) 6934

Perfluoropentanoic acid (PFPeA) 6914

Perfluorotetradecanoic acid (PFTeDA) 6902

Perfluorotridecanoic acid (PFTrDA) 9563

Perfluoroundecanoic acid (PFUnA) 6904

Solid

11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS) 9490



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Organic

| | |
|---|----------|
| Draft EPA Method 1633 by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) | 10123429 |
| Solid | |
| 2H,2H,3H,3H-Perfluorodecanoic Acid (7:3 FTCA, 3-Perfluoroheptyl Propanoic Acid) | 9340 |
| 2H,2H,3H,3H-Perfluorooctanoic Acid (5:3 FTCA) | 9338 |
| 4,4,5,5,6,6,6-Heptafluorohexanoic Acid (3:3 FTCA, 3-Perfluoropropyl Propanoic Acid) | 9353 |
| 4,8-dioxa-3H-perfluorononanoic acid (ADONA) | 6951 |
| 4:2 Fluorotelomersulfonic acid (4:2FTS) | 6946 |
| 6:2 Fluorotelomersulfonic acid (6:2FTS) | 6947 |
| 8:2 Fluorotelomersulfonic acid (8:2FTS) | 6948 |
| 9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS) | 6952 |
| Hexafluoropropylene oxide dimer acid (HFPO-DA) | 9460 |
| N-ethylperfluorooctanesulfonamide (EtFOSA) | 9395 |
| N-ethylperfluorooctanesulfonamidoacetic acid (EtFOSAA) | 4847 |
| N-ethylperfluorooctanesulfonamidoethanol (EtFOSE) | 9431 |
| N-methylperfluorooctanesulfonamide (MeFOSA) | 9433 |
| N-methylperfluorooctanesulfonamidoacetic acid (MeFOSAA) | 4846 |
| N-methylperfluorooctanesulfonamidoethanol (MeFOSE) | 6949 |
| Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) | 6956 |
| Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA) | 6957 |
| Perfluoro-3-methoxypropanoic acid (PFMPA) | 6965 |
| Perfluoro-4-methoxybutanoic acid (PFMBA) | 6966 |
| Perfluorobutanesulfonic acid (PFBS) | 6918 |
| Perfluorobutanoic acid (PFBA) | 6915 |
| Perfluorodecanesulfonic acid (PFDS) | 6920 |
| Perfluorodecanoic acid (PFDA) | 6905 |
| Perfluorododecanesulfonic acid (PFDoS) | 6923 |
| Perfluorododecanoic acid (PFDoA) | 6903 |
| Perfluoroheptanesulfonic acid (PFHpS) | 9470 |
| Perfluoroheptanoic acid (PFHpA) | 6908 |
| Perfluorohexanesulfonic acid (PFHxS) | 6927 |
| Perfluorohexanoic acid (PFHxA) | 6913 |
| Perfluorononanesulfonic acid (PFNS) | 6929 |
| Perfluorononanoic acid (PFNA) | 6906 |
| Perfluorooctanesulfonamide (PFOSA) | 6917 |
| Perfluorooctanesulfonic acid (PFOS) | 6931 |
| Perfluorooctanoic acid (PFOA) | 6912 |
| Perfluoropentanesulfonic acid (PFPeS) | 6934 |
| Perfluoropentanoic acid (PFPeA) | 6914 |



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| | |
|--|----------|
| Draft EPA Method 1633 by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) | 10123429 |
| Solid | |
| Perfluorotetradecanoic acid (PFTeDA) | 6902 |
| Perfluorotridecanoic acid (PFTrDA) | 9563 |
| Perfluoroundecanoic acid (PFUnA) | 6904 |
| EPA 533 by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) | 10091619 |
| Drinking Water | |
| 11-Chloroeicosafluoro-3-Oxaundecane-1-Sulfonic Acid (11Cl-PF3OUdS) | 9490 |
| 1H, 1H, 2H, 2H-Perfluorodecane Sulfonic Acid (8:2 FTS) | 6948 |
| 1H, 1H, 2H, 2H-Perfluorohexane Sulfonic Acid (4:2 FTS) | 6946 |
| 1H, 1H, 2H, 2H-Perfluorooctane Sulfonic Acid (6:2 FTS) | 6947 |
| 4,8-Dioxa-3H-Perfluorononanoic Acid (ADONA) | 6951 |
| 9-Chlorohexadecafluoro-3-Oxanonane-1-Sulfonic Acid (9-Cl-PF3ONS) | 6952 |
| Hexafluoropropylene Oxide Dimer Acid (HFPO-DA) – GenX | 9460 |
| Nonafluoro-3,6-Dioxaheptanoic Acid (NFDHA) | 6956 |
| Perfluoro(2-Ethoxyethane)Sulfonic Acid (PFEEESA) | 6957 |
| Perfluoro-3-Methoxypropanoic Acid (PFMPA) | 6965 |
| Perfluorobutane Sulfonic Acid (PFBS) | 6918 |
| Perfluorobutanoic Acid (PFBA) | 6915 |
| Perfluorodecanoic Acid (PFDA) | 6905 |
| Perfluorododecanoic Acid (PFDoA) | 6903 |
| Perfluoroheptane Sulfonic Acid (PFHpS) | 9470 |
| Perfluoroheptanoic Acid (PFHpA) | 6908 |
| Perfluorohexane Sulfonic Acid (PFHxS) | 6927 |
| Perfluorohexanoic Acid (PFHxA) | 6913 |
| Perfluorononanoic Acid (PFNA) | 6906 |
| Perfluorooctane Sulfonic Acid (PFOS) | 6931 |
| Perfluorooctanoic Acid (PFOA) | 6912 |
| Perfluoropentane Sulfonic Acid (PFPeS) | 6934 |
| Perfluoropentanoic Acid (PFPeA) | 6914 |
| Perfluoroundecanoic Acid (PFUnDA) | 6904 |
| Sum Perfluorooctanoic Acid (PFOA) + Perfluorooctane Sulfonic Acid (PFOS) Total (Calculation) | 6894 |
| EPA 608.3 by Gas Chromatography Electron Capture Detector (GC/ECD) | 10296614 |
| Aqueous | |
| 4,4'-DDD | 7355 |
| 4,4'-DDE | 7360 |
| 4,4'-DDT | 7365 |
| Aldrin | 7025 |



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Organic

EPA 608.3 by Gas Chromatography Electron Capture Detector (GC/ECD) 10296614

Aqueous

alpha-BHC (a-BHC, alpha-Hexachlorocyclohexane) 7110

alpha-Chlordane (cis-Chlordane) 7240

Aroclor 1016 8880

Aroclor 1221 8885

Aroclor 1232 8890

Aroclor 1242 8895

Aroclor 1248 8900

Aroclor 1254 8905

Aroclor 1260 8910

beta-BHC (b-BHC, beta-Hexachlorocyclohexane) 7115

Chlordane (Technical) 7250

delta-BHC (d-BHC) 7105

Endosulfan I 7510

Endosulfan II 7515

Endosulfan Sulfate 7520

Endrin 7540

Endrin Aldehyde 7530

gamma-BHC (γ -BHC, Lindane, gamma-Hexachlorocyclohexane) 7120

gamma-Chlordane 7245

Heptachlor 7685

Heptachlor Epoxide (beta) 7690

Methoxychlor 7810

Toxaphene (Chlorinated Camphene) 8250

EPA 624.1 by Gas Chromatography Mass Spectrometry (GC/MS) 10298121

Aqueous

1,1,1-Trichloroethane 5160

1,1,2,2-Tetrachloroethane 5110

1,1,2-Trichloroethane 5165

1,1-Dichloroethane 4630

1,1-Dichloroethylene 4640

1,2-Dichlorobenzene 4610

1,2-Dichloroethane (Ethylene Dichloride, EDC) 4635

1,2-Dichloropropane 4655

1,3-Dichlorobenzene 4615

1,4-Dichlorobenzene 4620

2-Chloroethyl Vinyl Ether (2-CEVE) 4500



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Code

Organic

EPA 624.1 by Gas Chromatography Mass Spectrometry (GC/MS) 10298121

Aqueous

Acrolein (Propenal) 4325

Acrylonitrile 4340

Benzene 4375

Bromodichloromethane 4395

Bromoform 4400

Carbon Tetrachloride 4455

Chlorobenzene 4475

Chlorodibromomethane (Dibromochloromethane) 4575

Chloroethane (Ethyl Chloride) 4485

Chloroform 4505

cis-1,2-Dichloroethylene 4645

cis-1,3-Dichloropropene 4680

Dichlorodifluoromethane (Freon 12) 4625

Ethylbenzene 4765

m,p-Xylene 5240

Methyl Bromide (Bromomethane) 4950

Methyl Chloride (Chloromethane) 4960

Methyl tert-Butyl Ether (MTBE) 5000

Methylene Chloride (Dichloromethane) 4975

o-Xylene (1,2-Xylene) 5250

Tetrachloroethylene (Perchloroethylene, PCE) 5115

Toluene 5140

trans-1,2-Dichloroethylene 4700

trans-1,3-Dichloropropylene 4685

Trichloroethene (TCE, Trichloroethylene) 5170

Trichlorofluoromethane (Fluorotrichloromethane, Freon 11) 5175

Vinyl Chloride (Chloroethene) 5235

Xylenes (Total) 5260

EPA 625.1 by Gas Chromatography Mass Spectrometry (GC/MS) 10300024

Aqueous

1,2,4-Trichlorobenzene 5155

1,2-Dichlorobenzene 4610

1,3-Dichlorobenzene 4615

1,4-Dichlorobenzene 4620

1-Methylnaphthalene 6380

2,2'-Oxybis(1-Chloropropane) (was bis(2-Chloroisopropyl)ether) 4659



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Organic

EPA 625.1 by Gas Chromatography Mass Spectrometry (GC/MS)

10300024

Aqueous

| | |
|---|------|
| 2,3-Dichloroaniline | 9363 |
| 2,4,6-Trichlorophenol | 6840 |
| 2,4-Dichlorophenol | 6000 |
| 2,4-Dimethylphenol | 6130 |
| 2,4-Dinitrophenol | 6175 |
| 2,4-Dinitrotoluene (2,4-DNT) | 6185 |
| 2,6-Dichlorophenol | 6005 |
| 2,6-Dinitrotoluene (2,6-DNT) | 6190 |
| 2-Chloronaphthalene | 5795 |
| 2-Chlorophenol | 5800 |
| 2-Methyl-4,6-Dinitrophenol (4,6-Dinitro-2-Methylphenol) | 6360 |
| 2-Methylnaphthalene | 6385 |
| 2-Methylphenol (o-Cresol) | 6400 |
| 2-Nitrophenol | 6490 |
| 3,3'-Dichlorobenzidine | 5945 |
| 3+4-Methylphenol (m+p Cresol) | 6412 |
| 4-Bromophenyl Phenyl Ether (BDE-3) | 5660 |
| 4-Chloro-3-Methylphenol | 5700 |
| 4-Chlorophenyl Phenylether | 5825 |
| 4-Nitrophenol | 6500 |
| Acenaphthene | 5500 |
| Acenaphthylene | 5505 |
| Acetophenone | 5510 |
| alpha-Terpineol | 6700 |
| Aniline | 5545 |
| Anthracene | 5555 |
| Benzidine | 5595 |
| Benzo(a)Anthracene | 5575 |
| Benzo(a)Pyrene | 5580 |
| Benzo(b)Fluoranthene | 5585 |
| Benzo(g,h,i)Perylene | 5590 |
| Benzo(k)Fluoranthene | 5600 |
| Benzoic Acid | 5610 |
| bis(2-Chloroethoxy)Methane | 5760 |
| bis(2-Chloroethyl)Ether | 5765 |
| Butyl Benzyl Phthalate | 5670 |



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Organic

| | |
|--|----------|
| EPA 625.1 by Gas Chromatography Mass Spectrometry (GC/MS) | 10300024 |
| Aqueous | |
| Carbazole | 5680 |
| Chrysene | 5855 |
| di(2-ethylhexyl)Phthalate | 6065 |
| Dibenz(a,h)Anthracene | 5895 |
| Dibenzofuran | 5905 |
| Diethyl Phthalate | 6070 |
| Dimethyl Phthalate | 6135 |
| di-n-Butyl Phthalate | 5925 |
| di-n-Octyl Phthalate | 6200 |
| Fluoranthene | 6265 |
| Fluorene | 6270 |
| Hexachlorobenzene | 6275 |
| Hexachlorobutadiene | 4835 |
| Hexachlorocyclopentadiene | 6285 |
| Hexachloroethane | 4840 |
| Indeno(1,2,3,cd)Pyrene | 6315 |
| Isophorone | 6320 |
| Naphthalene | 5005 |
| n-Decane | 5875 |
| Nitrobenzene | 5015 |
| n-Nitrosodimethylamine | 6530 |
| n-Nitrosodi-n-Propylamine | 6545 |
| n-Nitrosodiphenylamine | 6535 |
| n-Octadecane | 6580 |
| Pentachlorophenol (PCP) | 6605 |
| Phenanthrene | 6615 |
| Phenol | 6625 |
| Pyrene | 6665 |
| Pyridine | 5095 |
| EPA 8011 by Gas Chromatography Electron Capture Detector (GC/ECD) | 10173009 |
| Aqueous | |
| 1,2-Dibromo-3-Chloropropane (DBCP) | 4570 |
| 1,2-Dibromoethane (EDB, Ethylene Dibromide) | 4585 |
| EPA 8015C by Gas Chromatography Flame Ionization Detection (GC/FID) | 10173816 |
| Aqueous | |
| 2-Ethoxyethanol (Cellosolve) | 5866 |



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Code

Organic

| | |
|--|----------|
| EPA 8015C by Gas Chromatography Flame Ionization Detection (GC/FID) | 10173816 |
| Aqueous | |
| Ethanol | 4750 |
| Ethylene Glycol | 4785 |
| Isobutyl Alcohol (2-Methyl-1-Propanol, Isobutanol) | 4875 |
| Isopropyl Alcohol (IPA, 2-Propanol, Isopropanol) | 4895 |
| Methanol | 4930 |
| n-Butyl Alcohol (1-Butanol, n-Butanol) | 4425 |
| n-Propanol (1-Propanol) | 5055 |
| Oil Range Organics (ORO) (C20-C35) | 6748 |
| Propylene Glycol | 6657 |
| Residual Range Organics (RRO) (C25-C36) | 6751 |
| Total Petroleum Hydrocarbons Diesel Range Organics (TPH DRO) (C10 - C28) | 9369 |
| Total Petroleum Hydrocarbons Gasoline Range Organics (TPH GRO) (C6 - C10) | 9408 |
| Solid | |
| 2-Ethoxyethanol (Cellosolve) | 5866 |
| Ethanol | 4750 |
| Ethylene Glycol | 4785 |
| Isobutyl Alcohol (2-Methyl-1-Propanol, Isobutanol) | 4875 |
| Isopropyl Alcohol (IPA, 2-Propanol, Isopropanol) | 4895 |
| Methanol | 4930 |
| n-Butyl Alcohol (1-Butanol, n-Butanol) | 4425 |
| n-Propanol (1-Propanol) | 5055 |
| Oil Range Organics (ORO) (C20-C35) | 6748 |
| Propylene Glycol | 6657 |
| Residual Range Organics (RRO) (C25-C36) | 6751 |
| Total Petroleum Hydrocarbons Diesel Range Organics (TPH DRO) (C10 - C28) | 9369 |
| Total Petroleum Hydrocarbons Gasoline Range Organics (TPH GRO) (C6 - C10) | 9408 |
| EPA 8081B by Gas Chromatography Electron Capture Detector (GC/ECD) | 10178811 |
| Aqueous | |
| 4,4'-DDD | 7355 |
| 4,4'-DDE | 7360 |
| 4,4'-DDT | 7365 |
| Aldrin | 7025 |
| alpha-BHC (a-BHC, alpha-Hexachlorocyclohexane) | 7110 |
| alpha-Chlordane (cis-Chlordane) | 7240 |
| beta-BHC (b-BHC, beta-Hexachlorocyclohexane) | 7115 |
| Chlordane (Technical) | 7250 |



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Organic

| | |
|---|----------|
| EPA 8081B by Gas Chromatography Electron Capture Detector (GC/ECD) | 10178811 |
| Aqueous | |
| delta-BHC (d-BHC) | 7105 |
| Dieldrin | 7470 |
| Endosulfan I | 7510 |
| Endosulfan II | 7515 |
| Endosulfan Sulfate | 7520 |
| Endrin | 7540 |
| Endrin Aldehyde | 7530 |
| Endrin Ketone | 7535 |
| gamma-BHC (γ -BHC, Lindane, gamma-Hexachlorocyclohexane) | 7120 |
| gamma-Chlordane | 7245 |
| Heptachlor | 7685 |
| Heptachlor Epoxide (beta) | 7690 |
| Hexachlorobenzene | 6275 |
| Methoxychlor | 7810 |
| Toxaphene (Chlorinated Camphene) | 8250 |
| Solid | |
| 4,4'-DDD | 7355 |
| 4,4'-DDE | 7360 |
| 4,4'-DDT | 7365 |
| Aldrin | 7025 |
| alpha-BHC (α -BHC, alpha-Hexachlorocyclohexane) | 7110 |
| alpha-Chlordane (cis-Chlordane) | 7240 |
| beta-BHC (β -BHC, beta-Hexachlorocyclohexane) | 7115 |
| Chlordane (Technical) | 7250 |
| delta-BHC (d-BHC) | 7105 |
| Dieldrin | 7470 |
| Endosulfan I | 7510 |
| Endosulfan II | 7515 |
| Endosulfan Sulfate | 7520 |
| Endrin | 7540 |
| Endrin Aldehyde | 7530 |
| Endrin Ketone | 7535 |
| gamma-BHC (γ -BHC, Lindane, gamma-Hexachlorocyclohexane) | 7120 |
| gamma-Chlordane | 7245 |
| Heptachlor | 7685 |
| Heptachlor Epoxide (beta) | 7690 |



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Organic

EPA 8081B by Gas Chromatography Electron Capture Detector (GC/ECD) 10178811

| | |
|----------------------------------|------|
| Solid | |
| Methoxychlor | 7810 |
| Toxaphene (Chlorinated Camphene) | 8250 |

EPA 8082A by Gas Chromatography Electron Capture Detector (GC/ECD) 10179358

| | |
|----------------|------|
| Aqueous | |
| Aroclor 1016 | 8880 |
| Aroclor 1221 | 8885 |
| Aroclor 1232 | 8890 |
| Aroclor 1242 | 8895 |
| Aroclor 1248 | 8900 |
| Aroclor 1254 | 8905 |
| Aroclor 1260 | 8910 |
| Aroclor 1262 | 8912 |
| Aroclor 1268 | 8913 |
| Solid | |
| Aroclor 1016 | 8880 |
| Aroclor 1221 | 8885 |
| Aroclor 1232 | 8890 |
| Aroclor 1242 | 8895 |
| Aroclor 1248 | 8900 |
| Aroclor 1254 | 8905 |
| Aroclor 1260 | 8910 |
| Aroclor 1262 | 8912 |
| Aroclor 1268 | 8913 |

EPA 8141B by Gas Chromatography Nitrogen-Phosphorus Detector (GC/NPD) 10182204

| | |
|---------------------------|------|
| Aqueous | |
| Atrazine | 7065 |
| Azinphos Methyl (Guthion) | 7075 |
| Chlorpyrifos (Dursban) | 7300 |
| Chlorpyrifos Methyl | 7305 |
| Demeton (o+s, Total) | 7390 |
| Demeton-o | 7395 |
| Demeton-s | 7385 |
| Diazinon | 7410 |
| Dimethoate | 7475 |
| Disulfoton | 8625 |
| Ethion | 7565 |



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Code

Organic

EPA 8141B by Gas Chromatography Nitrogen-Phosphorus Detector (GC/NPD) 10182204

Aqueous

| | |
|---|------|
| Ethoprop (Ethoprophos) | 7570 |
| Ethyl Parathion | 7955 |
| Famphur | 7580 |
| Fensulfothion | 7600 |
| Fonophos | 7640 |
| Malathion | 7770 |
| Merphos (a+b) | 7785 |
| Methyl Parathion | 7825 |
| Mevinphos | 7850 |
| Phorate | 7985 |
| Phosmet (Imidan) | 8000 |
| Ronnel (Fenchlorphos) | 8110 |
| Simazine | 8125 |
| Sulfotep (Tetraethyl Dithiopyrophospahte) | 8155 |

Solid

| | |
|---------------------------|------|
| Atrazine | 7065 |
| Azinphos Methyl (Guthion) | 7075 |
| Chlorpyrifos (Dursban) | 7300 |
| Chlorpyrifos Methyl | 7305 |
| Demeton (o+s, Total) | 7390 |
| Demeton-o | 7395 |
| Demeton-s | 7385 |
| Diazinon | 7410 |
| Dimethoate | 7475 |
| Disulfoton | 8625 |
| Ethion | 7565 |
| Ethoprop (Ethoprophos) | 7570 |
| Ethyl Parathion | 7955 |
| Famphur | 7580 |
| Fensulfothion | 7600 |
| Fonophos | 7640 |
| Malathion | 7770 |
| Merphos (a+b) | 7785 |
| Methyl Parathion | 7825 |
| Mevinphos | 7850 |
| Phorate | 7985 |



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Code

Organic

| | |
|--|----------|
| EPA 8141B by Gas Chromatography Nitrogen-Phosphorus Detector (GC/NPD) | 10182204 |
| Solid | |
| Phosmet (Imidan) | 8000 |
| Ronnel (Fenchlorphos) | 8110 |
| Simazine | 8125 |
| Sulfotep (Tetraethyl Dithiopyrophospahte) | 8155 |
| EPA 8151A by Gas Chromatography Electron Capture Detector (GC/ECD) | 10183207 |
| Aqueous | |
| 2,4,5-T | 8655 |
| 2,4-DB | 8560 |
| 2,4-Dichlorophenoxyacetic Acid (2,4-D) | 8545 |
| Dalapon | 8555 |
| Dicamba | 8595 |
| Dichloroprop (2,4-DP) | 8605 |
| Dinoseb (2-sec-Butyl-4,6-Dinitrophenol, DNBP) | 8620 |
| Pentachlorophenol (PCP) | 6605 |
| Silvex (2,4,5-TP) | 8650 |
| Solid | |
| 2,4,5-T | 8655 |
| 2,4-DB | 8560 |
| 2,4-Dichlorophenoxyacetic Acid (2,4-D) | 8545 |
| Dalapon | 8555 |
| Dicamba | 8595 |
| Dichloroprop (2,4-DP) | 8605 |
| Dinoseb (2-sec-Butyl-4,6-Dinitrophenol, DNBP) | 8620 |
| Pentachlorophenol (PCP) | 6605 |
| Silvex (2,4,5-TP) | 8650 |
| EPA 8260C by Gas Chromatography Mass Spectrometry (GC/MS) | 10307003 |
| Aqueous | |
| 1,1,1,2-Tetrachloroethane | 5105 |
| 1,1,1-Trichloroethane | 5160 |
| 1,1,2,2-Tetrachloroethane | 5110 |
| 1,1,2-Trichloro-1,2,2-Trifluoroethane (Trichlorotrifluoroethane, Freon 113) | 5185 |
| 1,1,2-Trichloroethane | 5165 |
| 1,1-Dichloroethane | 4630 |
| 1,1-Dichloroethylene | 4640 |
| 1,1-Dichloropropene | 4670 |
| 1,2,3-Trichlorobenzene | 5150 |



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EPA 8260C by Gas Chromatography Mass Spectrometry (GC/MS)

10307003

Aqueous

| | |
|--|------|
| 1,2,3-Trichloropropane (TCP) | 5180 |
| 1,2,4-Trichlorobenzene | 5155 |
| 1,2,4-Trimethylbenzene | 5210 |
| 1,2-Dibromo-3-Chloropropane (DBCP) | 4570 |
| 1,2-Dibromoethane (EDB, Ethylene Dibromide) | 4585 |
| 1,2-Dichlorobenzene | 4610 |
| 1,2-Dichloroethane (Ethylene Dichloride, EDC) | 4635 |
| 1,2-Dichloropropane | 4655 |
| 1,3,5-Trimethylbenzene | 5215 |
| 1,3-Dichlorobenzene | 4615 |
| 1,3-Dichloropropane | 4660 |
| 1,4-Dichlorobenzene | 4620 |
| 1,4-Dioxane (1,4-Diethyleneoxide, p-Dioxane) | 4735 |
| 2,2-Dichloropropane | 4665 |
| 2-Butanone (Methyl Ethyl Ketone, MEK) | 4410 |
| 2-Chloroethyl Vinyl Ether (2-CEVE) | 4500 |
| 2-Chlorotoluene | 4535 |
| 2-Hexanone (Methyl Butyl Ketone, MBK) | 4860 |
| 2-Nitropropane | 5020 |
| 4-Chlorotoluene | 4540 |
| 4-Isopropyltoluene (p-Isopropyltoluene, p-Cymene) | 4910 |
| 4-Methyl-2-Pentanone (Methyl Isobutyl Ketone (MIBK), Hexone) | 4995 |
| Acetone | 4315 |
| Acetonitrile | 4320 |
| Acrolein (Propenal) | 4325 |
| Acrylonitrile | 4340 |
| Allyl Chloride (3-Chloropropene) | 4355 |
| Benzene | 4375 |
| Bromobenzene | 4385 |
| Bromochloromethane | 4390 |
| Bromodichloromethane | 4395 |
| Bromoform | 4400 |
| Carbon Disulfide | 4450 |
| Carbon Tetrachloride | 4455 |
| Chlorobenzene | 4475 |
| Chlorodibromomethane (Dibromochloromethane) | 4575 |



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EPA 8260C by Gas Chromatography Mass Spectrometry (GC/MS)

10307003

Aqueous

Chloroethane (Ethyl Chloride)

4485

Chloroform

4505

Chloroprene (2-Chloro-1,3-Butadiene)

4525

cis-1,2-Dichloroethylene

4645

cis-1,3-Dichloropropene

4680

cis-1,4-Dichloro-2-Butene

4600

Cyclohexane

4555

Cyclohexanone

4560

Dibromomethane (Methylene Bromide)

4595

Dichlorodifluoromethane (Freon 12)

4625

Diethyl Ether

4725

Di-Isopropylether (DIPE, Propane)

9375

Ethanol

4750

Ethyl Acetate

4755

Ethyl Methacrylate

4810

Ethylbenzene

4765

Ethyl-tert-Butylether (ETBE, 2-Ethoxy-2-Methylpropane)

4770

Hexachlorobutadiene

4835

Iodomethane (Methyl Iodide)

4870

Isobutyl Alcohol (2-Methyl-1-Propanol, Isobutanol)

4875

Isopropyl Alcohol (IPA, 2-Propanol, Isopropanol)

4895

Isopropylbenzene (Cumene)

4900

m,p-Xylene

5240

Methacrylonitrile

4925

Methyl Acetate

4940

Methyl Bromide (Bromomethane)

4950

Methyl Chloride (Chloromethane)

4960

Methyl Methacrylate

4990

Methyl tert-Butyl Ether (MTBE)

5000

Methylcyclohexane

4965

Methylene Chloride (Dichloromethane)

4975

Naphthalene

5005

n-Butylbenzene

4435

n-Propylbenzene (1-phenylpropane)

5090

o-Xylene (1,2-Xylene)

5250

Pentachloroethane

5035



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Organic

EPA 8260C by Gas Chromatography Mass Spectrometry (GC/MS) 10307003

Aqueous

| | |
|---|------|
| Propionitrile (Ethyl Cyanide) | 5080 |
| sec-Butylbenzene | 4440 |
| Styrene | 5100 |
| tert-Amyl Methyl Ether (TAME) | 4370 |
| tert-Butyl Alcohol (TBA, 2-Methyl-2-Propanol, t-Butanol) | 4420 |
| tert-Butylbenzene | 4445 |
| Tetrachloroethylene (Perchloroethylene, PCE) | 5115 |
| Tetrahydrofuran (THF) | 5120 |
| Toluene | 5140 |
| trans-1,2-Dichloroethylene | 4700 |
| trans-1,3-Dichloropropylene | 4685 |
| trans-1,4-Dichloro-2-Butene | 4605 |
| Trichloroethene (TCE, Trichloroethylene) | 5170 |
| Trichlorofluoromethane (Fluorotrichloromethane, Freon 11) | 5175 |
| Vinyl Acetate | 5225 |
| Vinyl Chloride (Chloroethene) | 5235 |
| Xylenes (Total) | 5260 |

Solid

| | |
|---|------|
| 1,1,1,2-Tetrachloroethane | 5105 |
| 1,1,1-Trichloroethane | 5160 |
| 1,1,2,2-Tetrachloroethane | 5110 |
| 1,1,2-Trichloro-1,2,2-Trifluoroethane (Trichlorotrifluoroethane, Freon 113) | 5185 |
| 1,1,2-Trichloroethane | 5165 |
| 1,1-Dichloroethane | 4630 |
| 1,1-Dichloroethylene | 4640 |
| 1,1-Dichloropropene | 4670 |
| 1,2,3-Trichlorobenzene | 5150 |
| 1,2,3-Trichloropropane (TCP) | 5180 |
| 1,2,4-Trichlorobenzene | 5155 |
| 1,2,4-Trimethylbenzene | 5210 |
| 1,2-Dibromo-3-Chloropropane (DBCP) | 4570 |
| 1,2-Dibromoethane (EDB, Ethylene Dibromide) | 4585 |
| 1,2-Dichlorobenzene | 4610 |
| 1,2-Dichloroethane (Ethylene Dichloride, EDC) | 4635 |
| 1,2-Dichloropropane | 4655 |
| 1,3,5-Trimethylbenzene | 5215 |



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Organic

EPA 8260C by Gas Chromatography Mass Spectrometry (GC/MS)

10307003

Solid

| | |
|--|------|
| 1,3-Dichlorobenzene | 4615 |
| 1,3-Dichloropropane | 4660 |
| 1,4-Dichlorobenzene | 4620 |
| 1,4-Dioxane (1,4-Diethyleneoxide, p-Dioxane) | 4735 |
| 2,2-Dichloropropane | 4665 |
| 2-Butanone (Methyl Ethyl Ketone, MEK) | 4410 |
| 2-Chloroethyl Vinyl Ether (2-CEVE) | 4500 |
| 2-Chlorotoluene | 4535 |
| 2-Hexanone (Methyl Butyl Ketone, MBK) | 4860 |
| 2-Nitropropane | 5020 |
| 4-Chlorotoluene | 4540 |
| 4-Isopropyltoluene (p-Isopropyltoluene, p-Cymene) | 4910 |
| 4-Methyl-2-Pentanone (Methyl Isobutyl Ketone (MIBK), Hexone) | 4995 |
| Acetone | 4315 |
| Acetonitrile | 4320 |
| Acrolein (Propenal) | 4325 |
| Acrylonitrile | 4340 |
| Allyl Chloride (3-Chloropropene) | 4355 |
| Benzene | 4375 |
| Bromobenzene | 4385 |
| Bromochloromethane | 4390 |
| Bromodichloromethane | 4395 |
| Bromoform | 4400 |
| Carbon Disulfide | 4450 |
| Carbon Tetrachloride | 4455 |
| Chlorobenzene | 4475 |
| Chlorodibromomethane (Dibromochloromethane) | 4575 |
| Chloroethane (Ethyl Chloride) | 4485 |
| Chloroform | 4505 |
| Chloroprene (2-Chloro-1,3-Butadiene) | 4525 |
| cis-1,2-Dichloroethylene | 4645 |
| cis-1,3-Dichloropropene | 4680 |
| cis-1,4-Dichloro-2-Butene | 4600 |
| Cyclohexane | 4555 |
| Cyclohexanone | 4560 |
| Dibromomethane (Methylene Bromide) | 4595 |



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Advanced Environmental Laboratories, Inc.

6681 Southpoint Parkway, Jacksonville, FL 32216

Contact Name: Todd Romero Phone 352-538-4939, Heather Quilal-Ian Phone: 904-363-9350

Accreditation is granted to the facility to perform the following testing:

Code

Organic

EPA 8260C by Gas Chromatography Mass Spectrometry (GC/MS)

10307003

Solid

| | |
|--|------|
| Dichlorodifluoromethane (Freon 12) | 4625 |
| Diethyl Ether | 4725 |
| Di-Isopropylether (DIPE, Propane) | 9375 |
| Ethanol | 4750 |
| Ethyl Acetate | 4755 |
| Ethyl Methacrylate | 4810 |
| Ethylbenzene | 4765 |
| Ethyl-tert-Butylether (ETBE, 2-Ethoxy-2-Methylpropane) | 4770 |
| Hexachlorobutadiene | 4835 |
| Iodomethane (Methyl Iodide) | 4870 |
| Isobutyl Alcohol (2-Methyl-1-Propanol, Isobutanol) | 4875 |
| Isopropyl Alcohol (IPA, 2-Propanol, Isopropanol) | 4895 |
| Isopropylbenzene (Cumene) | 4900 |
| m,p-Xylene | 5240 |
| Methacrylonitrile | 4925 |
| Methyl Acetate | 4940 |
| Methyl Bromide (Bromomethane) | 4950 |
| Methyl Chloride (Chloromethane) | 4960 |
| Methyl Methacrylate | 4990 |
| Methyl tert-Butyl Ether (MTBE) | 5000 |
| Methylcyclohexane | 4965 |
| Methylene Chloride (Dichloromethane) | 4975 |
| Naphthalene | 5005 |
| n-Butylbenzene | 4435 |
| n-Propylbenzene (1-phenylpropane) | 5090 |
| o-Xylene (1,2-Xylene) | 5250 |
| Pentachloroethane | 5035 |
| Propionitrile (Ethyl Cyanide) | 5080 |
| sec-Butylbenzene | 4440 |
| Styrene | 5100 |
| tert-Amyl Methyl Ether (TAME) | 4370 |
| tert-Butyl Alcohol (TBA, 2-Methyl-2-Propanol, t-Butanol) | 4420 |
| tert-Butylbenzene | 4445 |
| Tetrachloroethylene (Perchloroethylene, PCE) | 5115 |
| Tetrahydrofuran (THF) | 5120 |
| Toluene | 5140 |



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Code

Organic

| | |
|---|----------|
| EPA 8260C by Gas Chromatography Mass Spectrometry (GC/MS) | 10307003 |
| Solid | |
| trans-1,2-Dichloroethylene | 4700 |
| trans-1,3-Dichloropropylene | 4685 |
| trans-1,4-Dichloro-2-Butene | 4605 |
| Trichloroethene (TCE, Trichloroethylene) | 5170 |
| Trichlorofluoromethane (Fluorotrichloromethane, Freon 11) | 5175 |
| Vinyl Acetate | 5225 |
| Vinyl Chloride (Chloroethene) | 5235 |
| Xylenes (Total) | 5260 |
| EPA 8260C SIM by Gas Chromatography Mass Spectrometry (GC/MS) Selective Ion Monitoring (SIM) | 10307105 |
| Aqueous | |
| 1,2-Dibromo-3-Chloropropane (DBCP) | 4570 |
| 1,2-Dibromoethane (EDB, Ethylene Dibromide) | 4585 |
| 1,4-Dioxane (1,4-Diethyleneoxide, p-Dioxane) | 4735 |
| EPA 8260D by Gas Chromatography Mass Spectrometry (GC/MS) | 10307127 |
| Aqueous | |
| 1,1,1,2-Tetrachloroethane | 5105 |
| 1,1,1-Trichloroethane | 5160 |
| 1,1,2,2-Tetrachloroethane | 5110 |
| 1,1,2-Trichloro-1,2,2-Trifluoroethane (Trichlorotrifluoroethane, Freon 113) | 5185 |
| 1,1,2-Trichloroethane | 5165 |
| 1,1-Dichloroethane | 4630 |
| 1,1-Dichloroethylene | 4640 |
| 1,1-Dichloropropene | 4670 |
| 1,2,3-Trichlorobenzene | 5150 |
| 1,2,3-Trichloropropane (TCP) | 5180 |
| 1,2,4-Trichlorobenzene | 5155 |
| 1,2,4-Trimethylbenzene | 5210 |
| 1,2-Dibromo-3-Chloropropane (DBCP) | 4570 |
| 1,2-Dibromoethane (EDB, Ethylene Dibromide) | 4585 |
| 1,2-Dichlorobenzene | 4610 |
| 1,2-Dichloroethane (Ethylene Dichloride, EDC) | 4635 |
| 1,2-Dichloropropane | 4655 |
| 1,3,5-Trimethylbenzene | 5215 |
| 1,3-Dichlorobenzene | 4615 |
| 1,3-Dichloropropane | 4660 |
| 1,4-Dichlorobenzene | 4620 |



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Code

Organic

EPA 8260D by Gas Chromatography Mass Spectrometry (GC/MS)

10307127

Aqueous

1,4-Dioxane (1,4-Diethyleneoxide, p-Dioxane)

4735

2,2-Dichloropropane

4665

2-Butanone (Methyl Ethyl Ketone, MEK)

4410

2-Chloroethyl Vinyl Ether (2-CEVE)

4500

2-Chlorotoluene

4535

2-Hexanone (Methyl Butyl Ketone, MBK)

4860

2-Nitropropane

5020

4-Chlorotoluene

4540

4-Isopropyltoluene (p-Isopropyltoluene, p-Cymene)

4910

4-Methyl-2-Pentanone (Methyl Isobutyl Ketone (MIBK), Hexone)

4995

Acetone

4315

Acetonitrile

4320

Acrolein (Propenal)

4325

Acrylonitrile

4340

Allyl Chloride (3-Chloropropene)

4355

Benzene

4375

Bromobenzene

4385

Bromochloromethane

4390

Bromodichloromethane

4395

Bromoform

4400

Carbon Disulfide

4450

Carbon Tetrachloride

4455

Chlorobenzene

4475

Chlorodibromomethane (Dibromochloromethane)

4575

Chloroethane (Ethyl Chloride)

4485

Chloroform

4505

Chloroprene (2-Chloro-1,3-Butadiene)

4525

cis-1,2-Dichloroethylene

4645

cis-1,3-Dichloropropene

4680

cis-1,4-Dichloro-2-Butene

4600

Cyclohexane

4555

Cyclohexanone

4560

Dibromomethane (Methylene Bromide)

4595

Dichlorodifluoromethane (Freon 12)

4625

Diethyl Ether

4725

Di-Isopropylether (DIPE)

9375



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Code

Organic

| | |
|--|----------|
| EPA 8260D by Gas Chromatography Mass Spectrometry (GC/MS) | 10307127 |
| Aqueous | |
| Ethanol | 4750 |
| Ethyl Acetate | 4755 |
| Ethyl Methacrylate | 4810 |
| Ethylbenzene | 4765 |
| Ethyl-tert-Butylether (ETBE, 2-Ethoxy-2-Methylpropane) | 4770 |
| Hexachlorobutadiene | 4835 |
| Iodomethane (Methyl Iodide) | 4870 |
| Isobutyl Alcohol (2-Methyl-1-Propanol, Isobutanol) | 4875 |
| Isopropyl Alcohol (IPA, 2-Propanol, Isopropanol) | 4895 |
| Isopropylbenzene (Cumene) | 4900 |
| m,p-Xylene | 5240 |
| Methacrylonitrile | 4925 |
| Methyl Acetate | 4940 |
| Methyl Bromide (Bromomethane) | 4950 |
| Methyl Chloride (Chloromethane) | 4960 |
| Methyl Methacrylate | 4990 |
| Methyl tert-Butyl Ether (MTBE) | 5000 |
| Methylcyclohexane | 4965 |
| Methylene Chloride (Dichloromethane) | 4975 |
| Naphthalene | 5005 |
| n-Butylbenzene | 4435 |
| n-Propylbenzene (1-phenylpropane) | 5090 |
| o-Xylene (1,2-Xylene) | 5250 |
| Pentachloroethane | 5035 |
| Propionitrile (Ethyl Cyanide) | 5080 |
| sec-Butylbenzene | 4440 |
| Styrene | 5100 |
| tert-Amyl Methyl Ether (TAME) | 4370 |
| tert-Butyl Alcohol (TBA, 2-Methyl-2-Propanol, t-Butanol) | 4420 |
| tert-Butylbenzene | 4445 |
| Tetrachloroethylene (Perchloroethylene, PCE) | 5115 |
| Tetrahydrofuran (THF) | 5120 |
| Toluene | 5140 |
| trans-1,2-Dichloroethylene | 4700 |
| trans-1,3-Dichloropropylene | 4685 |
| trans-1,4-Dichloro-2-Butene | 4605 |



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Code

Organic

EPA 8260D by Gas Chromatography Mass Spectrometry (GC/MS)

10307127

Aqueous

| | |
|---|------|
| Trichloroethene (TCE, Trichloroethylene) | 5170 |
| Trichlorofluoromethane (Fluorotrichloromethane, Freon 11) | 5175 |
| Vinyl Acetate | 5225 |
| Vinyl Chloride (Chloroethene) | 5235 |
| Xylenes (Total) | 5260 |

Solid

| | |
|---|------|
| 1,1,1,2-Tetrachloroethane | 5105 |
| 1,1,1-Trichloroethane | 5160 |
| 1,1,2,2-Tetrachloroethane | 5110 |
| 1,1,2-Trichloro-1,2,2-Trifluoroethane (Trichlorotrifluoroethane, Freon 113) | 5185 |
| 1,1,2-Trichloroethane | 5165 |
| 1,1-Dichloroethane | 4630 |
| 1,1-Dichloroethylene | 4640 |
| 1,1-Dichloropropene | 4670 |
| 1,2,3-Trichlorobenzene | 5150 |
| 1,2,3-Trichloropropane (TCP) | 5180 |
| 1,2,4-Trichlorobenzene | 5155 |
| 1,2,4-Trimethylbenzene | 5210 |
| 1,2-Dibromo-3-Chloropropane (DBCP) | 4570 |
| 1,2-Dibromoethane (EDB, Ethylene Dibromide) | 4585 |
| 1,2-Dichlorobenzene | 4610 |
| 1,2-Dichloroethane (Ethylene Dichloride, EDC) | 4635 |
| 1,2-Dichloropropane | 4655 |
| 1,3,5-Trimethylbenzene | 5215 |
| 1,3-Dichlorobenzene | 4615 |
| 1,3-Dichloropropane | 4660 |
| 1,4-Dichlorobenzene | 4620 |
| 1,4-Dioxane (1,4-Diethyleneoxide, p-Dioxane) | 4735 |
| 2,2-Dichloropropane | 4665 |
| 2-Butanone (Methyl Ethyl Ketone, MEK) | 4410 |
| 2-Chloroethyl Vinyl Ether (2-CEVE) | 4500 |
| 2-Chlorotoluene | 4535 |
| 2-Hexanone (Methyl Butyl Ketone, MBK) | 4860 |
| 2-Nitropropane | 5020 |
| 4-Chlorotoluene | 4540 |
| 4-Isopropyltoluene (p-Isopropyltoluene, p-Cymene) | 4910 |



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EPA 8260D by Gas Chromatography Mass Spectrometry (GC/MS)

10307127

Solid

| | |
|--|------|
| 4-Methyl-2-Pentanone (Methyl Isobutyl Ketone (MIBK), Hexone) | 4995 |
| Acetone | 4315 |
| Acetonitrile | 4320 |
| Acrolein (Propenal) | 4325 |
| Acrylonitrile | 4340 |
| Allyl Chloride (3-Chloropropene) | 4355 |
| Benzene | 4375 |
| Bromobenzene | 4385 |
| Bromochloromethane | 4390 |
| Bromodichloromethane | 4395 |
| Bromoform | 4400 |
| Carbon Disulfide | 4450 |
| Carbon Tetrachloride | 4455 |
| Chlorobenzene | 4475 |
| Chlorodibromomethane (Dibromochloromethane) | 4575 |
| Chloroethane (Ethyl Chloride) | 4485 |
| Chloroform | 4505 |
| Chloroprene (2-Chloro-1,3-Butadiene) | 4525 |
| cis-1,2-Dichloroethylene | 4645 |
| cis-1,3-Dichloropropene | 4680 |
| cis-1,4-Dichloro-2-Butene | 4600 |
| Cyclohexane | 4555 |
| Cyclohexanone | 4560 |
| Dibromomethane (Methylene Bromide) | 4595 |
| Dichlorodifluoromethane (Freon 12) | 4625 |
| Diethyl Ether | 4725 |
| Di-Isopropylether (DIPE) | 9375 |
| Ethanol | 4750 |
| Ethyl Acetate | 4755 |
| Ethyl Methacrylate | 4810 |
| Ethylbenzene | 4765 |
| Ethyl-tert-Butylether (ETBE, 2-Ethoxy-2-Methylpropane) | 4770 |
| Hexachlorobutadiene | 4835 |
| Iodomethane (Methyl Iodide) | 4870 |
| Isobutyl Alcohol (2-Methyl-1-Propanol, Isobutanol) | 4875 |
| Isopropyl Alcohol (IPA, 2-Propanol, Isopropanol) | 4895 |



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Organic

EPA 8260D by Gas Chromatography Mass Spectrometry (GC/MS)

10307127

Solid

| | |
|---|------|
| Isopropylbenzene (Cumene) | 4900 |
| m,p-Xylene | 5240 |
| Methacrylonitrile | 4925 |
| Methyl Acetate | 4940 |
| Methyl Bromide (Bromomethane) | 4950 |
| Methyl Chloride (Chloromethane) | 4960 |
| Methyl Methacrylate | 4990 |
| Methyl tert-Butyl Ether (MTBE) | 5000 |
| Methylcyclohexane | 4965 |
| Methylene Chloride (Dichloromethane) | 4975 |
| Naphthalene | 5005 |
| n-Butylbenzene | 4435 |
| n-Propylbenzene (1-phenylpropane) | 5090 |
| o-Xylene (1,2-Xylene) | 5250 |
| Pentachloroethane | 5035 |
| Propionitrile (Ethyl Cyanide) | 5080 |
| sec-Butylbenzene | 4440 |
| Styrene | 5100 |
| tert-Amyl Methyl Ether (TAME) | 4370 |
| tert-Butyl Alcohol (TBA, 2-Methyl-2-Propanol, t-Butanol) | 4420 |
| tert-Butylbenzene | 4445 |
| Tetrachloroethylene (Perchloroethylene, PCE) | 5115 |
| Tetrahydrofuran (THF) | 5120 |
| Toluene | 5140 |
| trans-1,2-Dichloroethylene | 4700 |
| trans-1,3-Dichloropropylene | 4685 |
| trans-1,4-Dichloro-2-Butene | 4605 |
| Trichloroethene (TCE, Trichloroethylene) | 5170 |
| Trichlorofluoromethane (Fluorotrichloromethane, Freon 11) | 5175 |
| Vinyl Acetate | 5225 |
| Vinyl Chloride (Chloroethene) | 5235 |
| Xylenes (Total) | 5260 |

EPA 8260D SIM by Gas Chromatography Mass Spectrometry (GC/MS) Selective Ion Monitoring (SIM)

10307138

Aqueous

| | |
|---|------|
| 1,2-Dibromo-3-Chloropropane (DBCP) | 4570 |
| 1,2-Dibromoethane (EDB, Ethylene Dibromide) | 4585 |



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Organic

EPA 8260D SIM by Gas Chromatography Mass Spectrometry (GC/MS) Selective Ion Monitoring (SIM) 10307138

Aqueous

1,4-Dioxane (1,4-Diethyleneoxide, p-Dioxane) 4735

EPA 8270D by Gas Chromatography Mass Spectrometry (GC/MS) 10186035

Aqueous

1,2,4,5-Tetrachlorobenzene 6715

1,2,4-Trichlorobenzene 5155

1,2-Dichlorobenzene 4610

1,2-Diphenylhydrazine (as Azobenzene) 6220

1,3-Dichlorobenzene 4615

1,3-Dinitrobenzene (1,3-DNB) 6160

1,4-Dichlorobenzene 4620

1-Chloronaphthalene 5790

1-Methylnaphthalene 6380

1-Naphthylamine 6425

2,2'-Oxybis(1-Chloropropane) (was bis(2-Chloroisopropyl)ether) 4659

2,3,4,6-Tetrachlorophenol 6735

2,4,5-Trichlorophenol 6835

2,4,6-Trichlorophenol 6840

2,4-Dichlorophenol 6000

2,4-Dimethylphenol 6130

2,4-Dinitrophenol 6175

2,4-Dinitrotoluene (2,4-DNT) 6185

2,6-Dichlorophenol 6005

2,6-Dinitrotoluene (2,6-DNT) 6190

2-Acetylaminofluorene 5515

2-Chloronaphthalene 5795

2-Chlorophenol 5800

2-Methyl-4,6-Dinitrophenol (4,6-Dinitro-2-Methylphenol) 6360

2-Methylaniline (o-Toluidine) 5145

2-Methylnaphthalene 6385

2-Methylphenol (o-Cresol) 6400

2-Naphthylamine 6430

2-Nitroaniline 6460

2-Nitrophenol 6490

2-Picoline (2-Methylpyridine) 5050

3,3'-Dichlorobenzidine 5945

3,3'-Dimethoxybenzidine 6100



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Accreditation is granted to the facility to perform the following testing:

Code

Organic

| | |
|--|----------|
| EPA 8270D by Gas Chromatography Mass Spectrometry (GC/MS) | 10186035 |
| Aqueous | |
| 3,3'-Dimethylbenzidine | 6120 |
| 3+4-Methylphenol (m+p Cresol) | 6412 |
| 3-Methylcholanthrene | 5781 |
| 3-Nitroaniline | 6465 |
| 4-Aminobiphenyl | 5540 |
| 4-Bromophenyl Phenyl Ether (BDE-3) | 5660 |
| 4-Chloro-3-Methylphenol | 5700 |
| 4-Chloroaniline | 5745 |
| 4-Chlorophenyl Phenylether | 5825 |
| 4-Dimethyl Aminoazobenzene (p-Dimethylamino Azobenzene) | 6105 |
| 4-Nitroaniline | 6470 |
| 4-Nitrophenol | 6500 |
| 5-Nitro-o-Toluidine | 6570 |
| 7,12-Dimethylbenz(a)Anthracene | 6115 |
| a-a-Dimethylphenethylamine | 6125 |
| Acenaphthene | 5500 |
| Acenaphthylene | 5505 |
| Acetophenone | 5510 |
| Alachlor | 7005 |
| Aniline | 5545 |
| Anthracene | 5555 |
| Aramite | 5560 |
| Atrazine | 7065 |
| Benzaldehyde | 5570 |
| Benzidine | 5595 |
| Benzo(a)Anthracene | 5575 |
| Benzo(a)Pyrene | 5580 |
| Benzo(b)Fluoranthene | 5585 |
| Benzo(g,h,i)Perylene | 5590 |
| Benzo(k)Fluoranthene | 5600 |
| Benzoic Acid | 5610 |
| Benzyl Alcohol | 5630 |
| Biphenyl | 5640 |
| bis(2-Chloroethoxy)Methane | 5760 |
| bis(2-Chloroethyl)Ether | 5765 |
| bis(2-Ethylhexyl)Adipate | 6062 |



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Code

Organic

EPA 8270D by Gas Chromatography Mass Spectrometry (GC/MS)

10186035

Aqueous

Butyl Benzyl Phthalate 5670

Caprolactam 7180

Carbazole 5680

Chlorobenzilate 7260

Chrysene 5855

di(2-ethylhexyl)Phthalate 6065

Diallate (cis/trans) 7405

Dibenz(a,h)Anthracene 5895

Dibenz(a,j)Acridine 5900

Dibenzofuran 5905

Diethyl Phthalate 6070

Dimethoate 7475

Dimethyl Phthalate 6135

di-n-Butyl Phthalate 5925

di-n-Octyl Phthalate 6200

Dinoseb (2-sec-Butyl-4,6-Dinitrophenol, DNBP) 8620

Diphenylamine 6205

Disulfoton 8625

Ethyl Methanesulfonate 6260

Fluoranthene 6265

Fluorene 6270

Hexachlorobenzene 6275

Hexachlorobutadiene 4835

Hexachlorocyclopentadiene 6285

Hexachloroethane 4840

Hexachloropropene 6295

Indeno(1,2,3,cd)Pyrene 6315

Isodrin 7725

Isophorone 6320

Isosafrole 6325

Kepone 7740

Methyl Methanesulfonate 6375

Methyl Parathion 7825

Naphthalene 5005

Nitrobenzene 5015

n-Nitrosodiethylamine 6525



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Code

Organic

EPA 8270D by Gas Chromatography Mass Spectrometry (GC/MS)

10186035

Aqueous

| | |
|---|------|
| n-Nitrosodimethylamine | 6530 |
| n-Nitroso-di-n-Butylamine | 5025 |
| n-Nitrosodi-n-Propylamine | 6545 |
| n-Nitrosodiphenylamine | 6535 |
| n-Nitrosomethylethylamine | 6550 |
| n-Nitrosomorpholine | 6555 |
| n-Nitrosopiperidine | 6560 |
| n-Nitrosopyrrolidine | 6565 |
| o,o,o-Triethyl Phosphorothioate | 8290 |
| Parathion (Ethyl) | 7955 |
| Pentachlorobenzene | 6590 |
| Pentachloroethane | 5035 |
| Pentachloronitrobenzene (PCNB) | 6600 |
| Pentachlorophenol (PCP) | 6605 |
| Phenacetin | 6610 |
| Phenanthrene | 6615 |
| Phenol | 6625 |
| Phorate | 7985 |
| Pronamide (Kerb) | 6650 |
| Pyrene | 6665 |
| Pyridine | 5095 |
| Safrole | 6685 |
| Simazine | 8125 |
| Sulfotep (Tetraethyl Dithiopyrophosphate) | 8155 |
| Thionazin | 8235 |

Solid

| | |
|---------------------------------------|------|
| 1,2,4,5-Tetrachlorobenzene | 6715 |
| 1,2,4-Trichlorobenzene | 5155 |
| 1,2-Dichlorobenzene | 4610 |
| 1,2-Diphenylhydrazine (as Azobenzene) | 6220 |
| 1,3-Dichlorobenzene | 4615 |
| 1,3-Dinitrobenzene (1,3-DNB) | 6160 |
| 1,4-Dichlorobenzene | 4620 |
| 1-Chloronaphthalene | 5790 |
| 1-Methylnaphthalene | 6380 |
| 1-Naphthylamine | 6425 |



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Code

Organic

EPA 8270D by Gas Chromatography Mass Spectrometry (GC/MS)

10186035

Solid

| | |
|--|------|
| 2,2'-Oxybis(1-Chloropropane) (was bis(2-Chloroisopropyl)ether) | 4659 |
| 2,3,4,6-Tetrachlorophenol | 6735 |
| 2,4,5-Trichlorophenol | 6835 |
| 2,4,6-Trichlorophenol | 6840 |
| 2,4-Dichlorophenol | 6000 |
| 2,4-Dimethylphenol | 6130 |
| 2,4-Dinitrophenol | 6175 |
| 2,4-Dinitrotoluene (2,4-DNT) | 6185 |
| 2,6-Dichlorophenol | 6005 |
| 2,6-Dinitrotoluene (2,6-DNT) | 6190 |
| 2-Acetylaminofluorene | 5515 |
| 2-Chloronaphthalene | 5795 |
| 2-Chlorophenol | 5800 |
| 2-Methyl-4,6-Dinitrophenol (4,6-Dinitro-2-Methylphenol) | 6360 |
| 2-Methylaniline (o-Toluidine) | 5145 |
| 2-Methylnaphthalene | 6385 |
| 2-Methylphenol (o-Cresol) | 6400 |
| 2-Naphthylamine | 6430 |
| 2-Nitroaniline | 6460 |
| 2-Nitrophenol | 6490 |
| 2-Picoline (2-Methylpyridine) | 5050 |
| 3,3'-Dichlorobenzidine | 5945 |
| 3,3'-Dimethoxybenzidine | 6100 |
| 3,3'-Dimethylbenzidine | 6120 |
| 3+4-Methylphenol (m+p Cresol) | 6412 |
| 3-Methylcholanthrene | 5781 |
| 3-Nitroaniline | 6465 |
| 4-Aminobiphenyl | 5540 |
| 4-Bromophenyl Phenyl Ether (BDE-3) | 5660 |
| 4-Chloro-3-Methylphenol | 5700 |
| 4-Chloroaniline | 5745 |
| 4-Chlorophenyl Phenylether | 5825 |
| 4-Dimethyl Aminoazobenzene (p-Dimethylamino Azobenzene) | 6105 |
| 4-Nitroaniline | 6470 |
| 4-Nitrophenol | 6500 |
| 5-Nitro-o-Toluidine | 6570 |



Certificate of Accreditation: Supplement

Advanced Environmental Laboratories, Inc.

6681 Southpoint Parkway, Jacksonville, FL 32216

Contact Name: Todd Romero Phone 352-538-4939, Heather Quilal-Ian Phone: 904-363-9350

Accreditation is granted to the facility to perform the following testing:

Code

Organic

EPA 8270D by Gas Chromatography Mass Spectrometry (GC/MS)

10186035

Solid

| | |
|--------------------------------|------|
| 7,12-Dimethylbenz(a)Anthracene | 6115 |
| a-a-Dimethylphenethylamine | 6125 |
| Acenaphthene | 5500 |
| Acenaphthylene | 5505 |
| Acetophenone | 5510 |
| Alachlor | 7005 |
| Aniline | 5545 |
| Anthracene | 5555 |
| Aramite | 5560 |
| Atrazine | 7065 |
| Benzaldehyde | 5570 |
| Benzidine | 5595 |
| Benzo(a)Anthracene | 5575 |
| Benzo(a)Pyrene | 5580 |
| Benzo(b)Fluoranthene | 5585 |
| Benzo(g,h,i)Perylene | 5590 |
| Benzo(k)Fluoranthene | 5600 |
| Benzoic Acid | 5610 |
| Benzyl Alcohol | 5630 |
| Biphenyl | 5640 |
| bis(2-Chloroethoxy)Methane | 5760 |
| bis(2-Chloroethyl)Ether | 5765 |
| Butyl Benzyl Phthalate | 5670 |
| Caprolactam | 7180 |
| Carbazole | 5680 |
| Chlorobenzilate | 7260 |
| di(2-ethylhexyl)Phthalate | 6065 |
| Diallate (cis/trans) | 7405 |
| Dibenz(a,h)Anthracene | 5895 |
| Dibenz(a,j)Acridine | 5900 |
| Dibenzofuran | 5905 |
| Diethyl Phthalate | 6070 |
| Dimethoate | 7475 |
| Dimethyl Phthalate | 6135 |
| di-n-Butyl Phthalate | 5925 |
| di-n-Octyl Phthalate | 6200 |



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Code

Organic

EPA 8270D by Gas Chromatography Mass Spectrometry (GC/MS)

10186035

Solid

| | |
|---------------------------------|------|
| Diphenylamine | 6205 |
| Disulfoton | 8625 |
| Ethyl Methanesulfonate | 6260 |
| Fluoranthene | 6265 |
| Fluorene | 6270 |
| Hexachlorobenzene | 6275 |
| Hexachlorobutadiene | 4835 |
| Hexachlorocyclopentadiene | 6285 |
| Hexachloroethane | 4840 |
| Hexachloropropene | 6295 |
| Indeno(1,2,3,cd)Pyrene | 6315 |
| Isodrin | 7725 |
| Isophorone | 6320 |
| Isosafrole | 6325 |
| Kepone | 7740 |
| Methyl Methanesulfonate | 6375 |
| Methyl Parathion | 7825 |
| Naphthalene | 5005 |
| Nitrobenzene | 5015 |
| n-Nitrosodiethylamine | 6525 |
| n-Nitrosodimethylamine | 6530 |
| n-Nitroso-di-n-Butylamine | 5025 |
| n-Nitrosodi-n-Propylamine | 6545 |
| n-Nitrosodiphenylamine | 6535 |
| n-Nitrosomethylethylamine | 6550 |
| n-Nitrosomorpholine | 6555 |
| n-Nitrosopiperidine | 6560 |
| n-Nitrosopyrrolidine | 6565 |
| o,o,o-Triethyl Phosphorothioate | 8290 |
| Parathion (Ethyl) | 7955 |
| Pentachlorobenzene | 6590 |
| Pentachloroethane | 5035 |
| Pentachloronitrobenzene (PCNB) | 6600 |
| Pentachlorophenol (PCP) | 6605 |
| Phenacetin | 6610 |
| Phenanthrene | 6615 |



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Code

Organic

| | |
|---|----------|
| EPA 8270D by Gas Chromatography Mass Spectrometry (GC/MS) | 10186035 |
| Solid | |
| Phenol | 6625 |
| Phorate | 7985 |
| Pronamide (Kerb) | 6650 |
| Pyrene | 6665 |
| Pyridine | 5095 |
| Safrole | 6685 |
| Simazine | 8125 |
| Sulfotep (Tetraethyl Dithiopyrophosphate) | 8155 |
| Thionazin | 8235 |
| EPA 8270D SIM by Gas Chromatography Mass Spectrometry (GC/MS) Selective Ion Monitoring (SIM) | 10242532 |
| Aqueous | |
| 1,4-Dioxane (1,4-Diethyleneoxide, p-Dioxane) | 4735 |
| 1-Methylnaphthalene | 6380 |
| 2,4-Dinitrophenol | 6175 |
| 2-Methyl-4,6-Dinitrophenol (4,6-Dinitro-2-Methylphenol) | 6360 |
| 2-Methylnaphthalene | 6385 |
| Acenaphthene | 5500 |
| Acenaphthylene | 5505 |
| Anthracene | 5555 |
| Benzo(a)Anthracene | 5575 |
| Benzo(a)Pyrene | 5580 |
| Benzo(b)Fluoranthene | 5585 |
| Benzo(g,h,i)Perylene | 5590 |
| Benzo(k)Fluoranthene | 5600 |
| Carbazole | 5680 |
| Chrysene | 5855 |
| Dibenz(a,h)Anthracene | 5895 |
| Dibenzofuran | 5905 |
| Fluoranthene | 6265 |
| Fluorene | 6270 |
| Indeno(1,2,3,cd)Pyrene | 6315 |
| Kepone | 7740 |
| Naphthalene | 5005 |
| Phenanthrene | 6615 |
| Pyrene | 6665 |



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Code

Organic

EPA 8270D SIM by Gas Chromatography Mass Spectrometry (GC/MS) Selective Ion Monitoring (SIM) 10242532

Solid

| | |
|---|------|
| 1-Methylnaphthalene | 6380 |
| 2,4-Dinitrophenol | 6175 |
| 2-Methyl-4,6-Dinitrophenol (4,6-Dinitro-2-Methylphenol) | 6360 |
| 2-Methylnaphthalene | 6385 |
| Acenaphthene | 5500 |
| Acenaphthylene | 5505 |
| Anthracene | 5555 |
| Benzo(a)Anthracene | 5575 |
| Benzo(a)Pyrene | 5580 |
| Benzo(b)Fluoranthene | 5585 |
| Benzo(g,h,i)Perylene | 5590 |
| Benzo(k)Fluoranthene | 5600 |
| Carbazole | 5680 |
| Chrysene | 5855 |
| Dibenz(a,h)Anthracene | 5895 |
| Dibenzofuran | 5905 |
| Fluoranthene | 6265 |
| Fluorene | 6270 |
| Indeno(1,2,3,cd)Pyrene | 6315 |
| Kepone | 7740 |
| Naphthalene | 5005 |
| Phenanthrene | 6615 |
| Pyrene | 6665 |

EPA 8270E by Gas Chromatography Mass Spectrometry (GC/MS) 10242543

Aqueous

| | |
|--|------|
| 1,2,4,5-Tetrachlorobenzene | 6715 |
| 1,2,4-Trichlorobenzene | 5155 |
| 1,2-Dichlorobenzene | 4610 |
| 1,2-Diphenylhydrazine (as Azobenzene) | 6220 |
| 1,3-Dichlorobenzene | 4615 |
| 1,3-Dinitrobenzene (1,3-DNB) | 6160 |
| 1,4-Dichlorobenzene | 4620 |
| 1-Chloronaphthalene | 5790 |
| 1-Methylnaphthalene | 6380 |
| 1-Naphthylamine | 6425 |
| 2,2'-Oxybis(1-Chloropropane) (was bis(2-Chloroisopropyl)ether) | 4659 |



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Code

Organic

EPA 8270E by Gas Chromatography Mass Spectrometry (GC/MS)

10242543

Aqueous

| | |
|---|------|
| 2,3,4,6-Tetrachlorophenol | 6735 |
| 2,4,5-Trichlorophenol | 6835 |
| 2,4,6-Trichlorophenol | 6840 |
| 2,4-Dichlorophenol | 6000 |
| 2,4-Dimethylphenol | 6130 |
| 2,4-Dinitrophenol | 6175 |
| 2,4-Dinitrotoluene (2,4-DNT) | 6185 |
| 2,6-Dichlorophenol | 6005 |
| 2,6-Dinitrotoluene (2,6-DNT) | 6190 |
| 2-Acetylaminofluorene | 5515 |
| 2-Chloronaphthalene | 5795 |
| 2-Chlorophenol | 5800 |
| 2-Methyl-4,6-Dinitrophenol (4,6-Dinitro-2-Methylphenol) | 6360 |
| 2-Methylaniline (o-Toluidine) | 5145 |
| 2-Methylnaphthalene | 6385 |
| 2-Methylphenol (o-Cresol) | 6400 |
| 2-Naphthylamine | 6430 |
| 2-Nitroaniline | 6460 |
| 2-Picoline (2-Methylpyridine) | 5050 |
| 3,3'-Dichlorobenzidine | 5945 |
| 3,3'-Dimethoxybenzidine | 6100 |
| 3,3'-Dimethylbenzidine | 6120 |
| 3+4-Methylphenol (m+p Cresol) | 6412 |
| 3-Methylcholanthrene | 5781 |
| 3-Nitroaniline | 6465 |
| 4-Aminobiphenyl | 5540 |
| 4-Bromophenyl Phenyl Ether (BDE-3) | 5660 |
| 4-Chloro-3-Methylphenol | 5700 |
| 4-Chloroaniline | 5745 |
| 4-Chlorophenyl Phenylether | 5825 |
| 4-Dimethyl Aminoazobenzene (p-Dimethylamino Azobenzene) | 6105 |
| 4-Nitroaniline | 6470 |
| 4-Nitrophenol | 6500 |
| 5-Nitro-o-Toluidine | 6570 |
| 7,12-Dimethylbenz(a)Anthracene | 6115 |
| a-a-Dimethylphenethylamine | 6125 |



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Code

Organic

| | |
|--|----------|
| EPA 8270E by Gas Chromatography Mass Spectrometry (GC/MS) | 10242543 |
| Aqueous | |
| Acenaphthene | 5500 |
| Acenaphthylene | 5505 |
| Acetophenone | 5510 |
| Alachlor | 7005 |
| Aniline | 5545 |
| Anthracene | 5555 |
| Aramite | 5560 |
| Atrazine | 7065 |
| Benzaldehyde | 5570 |
| Benzidine | 5595 |
| Benzo(a)Anthracene | 5575 |
| Benzo(a)Pyrene | 5580 |
| Benzo(b)Fluoranthene | 5585 |
| Benzo(g,h,i)Perylene | 5590 |
| Benzo(k)Fluoranthene | 5600 |
| Benzoic Acid | 5610 |
| Benzyl Alcohol | 5630 |
| Biphenyl | 5640 |
| bis(2-Chloroethoxy)Methane | 5760 |
| bis(2-Chloroethyl)Ether | 5765 |
| bis(2-Ethylhexyl)Adipate | 6062 |
| Butyl Benzyl Phthalate | 5670 |
| Caprolactam | 7180 |
| Carbazole | 5680 |
| Chlorobenzilate | 7260 |
| Chrysene | 5855 |
| di(2-ethylhexyl)Phthalate | 6065 |
| Diallate (cis/trans) | 7405 |
| Dibenz(a,h)Anthracene | 5895 |
| Dibenz(a,j)Acridine | 5900 |
| Dibenzofuran | 5905 |
| Diethyl Phthalate | 6070 |
| Dimethoate | 7475 |
| Dimethyl Phthalate | 6135 |
| di-n-Butyl Phthalate | 5925 |
| di-n-Octyl Phthalate | 6200 |



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Code

Organic

EPA 8270E by Gas Chromatography Mass Spectrometry (GC/MS)

10242543

Aqueous

Dinoseb (2-sec-Butyl-4,6-Dinitrophenol, DNBP)

8620

Diphenylamine

6205

Disulfoton

8625

Ethyl Methanesulfonate

6260

Fluoranthene

6265

Fluorene

6270

Hexachlorobenzene

6275

Hexachlorobutadiene

4835

Hexachlorocyclopentadiene

6285

Hexachloroethane

4840

Hexachloropropene

6295

Indeno(1,2,3,cd)Pyrene

6315

Isodrin

7725

Isophorone

6320

Isosafrole

6325

Kepone

7740

Methyl Methanesulfonate

6375

Methyl Parathion

7825

Naphthalene

5005

Nitrobenzene

5015

n-Nitrosodiethylamine

6525

n-Nitrosodimethylamine

6530

n-Nitroso-di-n-Butylamine

5025

n-Nitrosodi-n-Propylamine

6545

n-Nitrosodiphenylamine

6535

n-Nitrosomethylethylamine

6550

n-Nitrosomorpholine

6555

n-Nitrosopiperidine

6560

n-Nitrosopyrrolidine

6565

o,o,o-Triethyl Phosphorothioate

8290

Parathion (Ethyl)

7955

Pentachlorobenzene

6590

Pentachloroethane

5035

Pentachloronitrobenzene (PCNB)

6600

Pentachlorophenol (PCP)

6605

Phenacetin

6610



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Code

Organic

EPA 8270E by Gas Chromatography Mass Spectrometry (GC/MS)

10242543

Aqueous

| | |
|---|------|
| Phenanthrene | 6615 |
| Phenol | 6625 |
| Phorate | 7985 |
| Pronamide (Kerb) | 6650 |
| Pyrene | 6665 |
| Pyridine | 5095 |
| Safrole | 6685 |
| Simazine | 8125 |
| Sulfotep (Tetraethyl Dithiopyrophosphate) | 8155 |
| Thionazin | 8235 |

Solid

| | |
|--|------|
| 1,2,4,5-Tetrachlorobenzene | 6715 |
| 1,2,4-Trichlorobenzene | 5155 |
| 1,2-Dichlorobenzene | 4610 |
| 1,2-Diphenylhydrazine (as Azobenzene) | 6220 |
| 1,3-Dichlorobenzene | 4615 |
| 1,3-Dinitrobenzene (1,3-DNB) | 6160 |
| 1,4-Dichlorobenzene | 4620 |
| 1-Chloronaphthalene | 5790 |
| 1-Methylnaphthalene | 6380 |
| 1-Naphthylamine | 6425 |
| 2,2'-Oxybis(1-Chloropropane) (was bis(2-Chloroisopropyl)ether) | 4659 |
| 2,3,4,6-Tetrachlorophenol | 6735 |
| 2,4,5-Trichlorophenol | 6835 |
| 2,4,6-Trichlorophenol | 6840 |
| 2,4-Dichlorophenol | 6000 |
| 2,4-Dimethylphenol | 6130 |
| 2,4-Dinitrophenol | 6175 |
| 2,4-Dinitrotoluene (2,4-DNT) | 6185 |
| 2,6-Dichlorophenol | 6005 |
| 2,6-Dinitrotoluene (2,6-DNT) | 6190 |
| 2-Acetylaminofluorene | 5515 |
| 2-Chloronaphthalene | 5795 |
| 2-Chlorophenol | 5800 |
| 2-Methyl-4,6-Dinitrophenol (4,6-Dinitro-2-Methylphenol) | 6360 |
| 2-Methylaniline (o-Toluidine) | 5145 |



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Code

Organic

EPA 8270E by Gas Chromatography Mass Spectrometry (GC/MS)

10242543

Solid

| | |
|---|------|
| 2-Methylnaphthalene | 6385 |
| 2-Methylphenol (o-Cresol) | 6400 |
| 2-Naphthylamine | 6430 |
| 2-Nitroaniline | 6460 |
| 2-Nitrophenol | 6490 |
| 2-Picoline (2-Methylpyridine) | 5050 |
| 3,3'-Dichlorobenzidine | 5945 |
| 3,3'-Dimethoxybenzidine | 6100 |
| 3,3'-Dimethylbenzidine | 6120 |
| 3+4-Methylphenol (m+p Cresol) | 6412 |
| 3-Methylcholanthrene | 5781 |
| 3-Nitroaniline | 6465 |
| 4-Aminobiphenyl | 5540 |
| 4-Bromophenyl Phenyl Ether (BDE-3) | 5660 |
| 4-Chloro-3-Methylphenol | 5700 |
| 4-Chloroaniline | 5745 |
| 4-Chlorophenyl Phenylether | 5825 |
| 4-Dimethyl Aminoazobenzene (p-Dimethylamino Azobenzene) | 6105 |
| 4-Nitroaniline | 6470 |
| 4-Nitrophenol | 6500 |
| 5-Nitro-o-Toluidine | 6570 |
| 7,12-Dimethylbenz(a)Anthracene | 6115 |
| a-a-Dimethylphenethylamine | 6125 |
| Acenaphthene | 5500 |
| Acenaphthylene | 5505 |
| Acetophenone | 5510 |
| Alachlor | 7005 |
| Aniline | 5545 |
| Anthracene | 5555 |
| Aramite | 5560 |
| Atrazine | 7065 |
| Benzaldehyde | 5570 |
| Benzidine | 5595 |
| Benzo(a)Anthracene | 5575 |
| Benzo(a)Pyrene | 5580 |
| Benzo(b)Fluoranthene | 5585 |



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Code

Organic

EPA 8270E by Gas Chromatography Mass Spectrometry (GC/MS)

10242543

Solid

| | |
|----------------------------|------|
| Benzo(g,h,i)Perylene | 5590 |
| Benzo(k)Fluoranthene | 5600 |
| Benzoic Acid | 5610 |
| Benzyl Alcohol | 5630 |
| Biphenyl | 5640 |
| bis(2-Chloroethoxy)Methane | 5760 |
| bis(2-Chloroethyl)Ether | 5765 |
| Butyl Benzyl Phthalate | 5670 |
| Caprolactam | 7180 |
| Carbazole | 5680 |
| Chlorobenzilate | 7260 |
| Chrysene | 5855 |
| di(2-ethylhexyl)Phthalate | 6065 |
| Diallate (cis/trans) | 7405 |
| Dibenz(a,h)Anthracene | 5895 |
| Dibenz(a,j)Acridine | 5900 |
| Dibenzofuran | 5905 |
| Diethyl Phthalate | 6070 |
| Dimethoate | 7475 |
| Dimethyl Phthalate | 6135 |
| di-n-Butyl Phthalate | 5925 |
| di-n-Octyl Phthalate | 6200 |
| Diphenylamine | 6205 |
| Disulfoton | 8625 |
| Ethyl Methanesulfonate | 6260 |
| Fluoranthene | 6265 |
| Fluorene | 6270 |
| Hexachlorobenzene | 6275 |
| Hexachlorobutadiene | 4835 |
| Hexachlorocyclopentadiene | 6285 |
| Hexachloroethane | 4840 |
| Hexachloropropene | 6295 |
| Indeno(1,2,3,cd)Pyrene | 6315 |
| Isodrin | 7725 |
| Isophorone | 6320 |
| Isosafrole | 6325 |



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Code

Organic

| | |
|---|----------|
| EPA 8270E by Gas Chromatography Mass Spectrometry (GC/MS) | 10242543 |
| Solid | |
| Kepone | 7740 |
| Methyl Methanesulfonate | 6375 |
| Methyl Parathion | 7825 |
| Naphthalene | 5005 |
| Nitrobenzene | 5015 |
| n-Nitrosodiethylamine | 6525 |
| n-Nitrosodimethylamine | 6530 |
| n-Nitroso-di-n-Butylamine | 5025 |
| n-Nitrosodi-n-Propylamine | 6545 |
| n-Nitrosodiphenylamine | 6535 |
| n-Nitrosomethylethylamine | 6550 |
| n-Nitrosomorpholine | 6555 |
| n-Nitrosopiperidine | 6560 |
| n-Nitrosopyrrolidine | 6565 |
| o,o,o-Triethyl Phosphorothioate | 8290 |
| Parathion (Ethyl) | 7955 |
| Pentachlorobenzene | 6590 |
| Pentachloroethane | 5035 |
| Pentachloronitrobenzene (PCNB) | 6600 |
| Pentachlorophenol (PCP) | 6605 |
| Phenacetin | 6610 |
| Phenanthrene | 6615 |
| Phenol | 6625 |
| Phorate | 7985 |
| Pronamide (Kerb) | 6650 |
| Pyrene | 6665 |
| Pyridine | 5095 |
| Safrole | 6685 |
| Simazine | 8125 |
| Sulfotep (Tetraethyl Dithiopyrophosphate) | 8155 |
| Thionazin | 8235 |
| EPA 8270E SIM by Gas Chromatography Mass Spectrometry (GC/MS) Selective Ion Monitoring (SIM) | 10242565 |
| Aqueous | |
| 1,4-Dioxane (1,4-Diethyleneoxide, p-Dioxane) | 4735 |
| 1-Methylnaphthalene | 6380 |
| 2,4-Dinitrophenol | 6175 |



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Code

Organic

EPA 8270E SIM by Gas Chromatography Mass Spectrometry (GC/MS) Selective Ion Monitoring (SIM) 10242565

Aqueous

2-Methyl-4,6-Dinitrophenol (4,6-Dinitro-2-Methylphenol) 6360

2-Methylnaphthalene 6385

Acenaphthene 5500

Acenaphthylene 5505

Anthracene 5555

Benzo(a)Anthracene 5575

Benzo(a)Pyrene 5580

Benzo(b)Fluoranthene 5585

Benzo(g,h,i)Perylene 5590

Benzo(k)Fluoranthene 5600

Carbazole 5680

Chrysene 5855

Dibenz(a,h)Anthracene 5895

Dibenzofuran 5905

Fluoranthene 6265

Fluorene 6270

Indeno(1,2,3,cd)Pyrene 6315

Kepone 7740

Naphthalene 5005

Phenanthrene 6615

Pyrene 6665

Solid

1-Methylnaphthalene 6380

2,4-Dinitrophenol 6175

2-Methyl-4,6-Dinitrophenol (4,6-Dinitro-2-Methylphenol) 6360

2-Methylnaphthalene 6385

Acenaphthene 5500

Acenaphthylene 5505

Anthracene 5555

Benzo(a)Anthracene 5575

Benzo(a)Pyrene 5580

Benzo(b)Fluoranthene 5585

Benzo(g,h,i)Perylene 5590

Benzo(k)Fluoranthene 5600

Carbazole 5680

Chrysene 5855



Certificate of Accreditation: Supplement

Advanced Environmental Laboratories, Inc.

6681 Southpoint Parkway, Jacksonville, FL 32216

Contact Name: Todd Romero Phone 352-538-4939, Heather Quilal-Ian Phone: 904-363-9350

Accreditation is granted to the facility to perform the following testing:

Code

Organic

EPA 8270E SIM by Gas Chromatography Mass Spectrometry (GC/MS) Selective Ion Monitoring (SIM) 10242565

Solid

| | |
|------------------------|------|
| Dibenz(a,h)Anthracene | 5895 |
| Dibenzofuran | 5905 |
| Fluoranthene | 6265 |
| Fluorene | 6270 |
| Indeno(1,2,3,cd)Pyrene | 6315 |
| Kepone | 7740 |
| Naphthalene | 5005 |
| Phenanthrene | 6615 |
| Pyrene | 6665 |

EPA 8330 by High Performance Liquid Chromatography (HPLC) 10308006

Aqueous

| | |
|--|------|
| 1,3,5-Trinitrobenzene (1,3,5-TNB) | 6885 |
| 1,3-Dinitrobenzene (1,3-DNB) | 6160 |
| 2,4,6-Trinitrotoluene (2,4,6-TNT) | 9651 |
| 2,4-Dinitrotoluene (2,4-DNT) | 6185 |
| 2,6-Dinitrotoluene (2,6-DNT) | 6190 |
| 2-Amino-4,6-dinitrotoluene (2-am-DNT) | 9303 |
| 2-Nitrotoluene | 9507 |
| 3,5-Dinitroaniline | 6150 |
| 3-Nitrotoluene (3-NT) | 9510 |
| 4-Amino-2,6-dinitrotoluene (4-am-DNT) | 9306 |
| 4-Nitrotoluene (4-NT) | 9513 |
| Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) | 9432 |
| Methyl-2,4,6-Trinitrophenylnitramine (tetryl) | 6415 |
| Nitrobenzene | 5015 |
| Nitroglycerin | 6485 |
| Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) | 9522 |
| Pentaerythritoltetranitrate (PETN) | 9558 |

Solid

| | |
|---------------------------------------|------|
| 1,3,5-Trinitrobenzene (1,3,5-TNB) | 6885 |
| 1,3-Dinitrobenzene (1,3-DNB) | 6160 |
| 2,4,6-Trinitrotoluene (2,4,6-TNT) | 9651 |
| 2,4-Dinitrotoluene (2,4-DNT) | 6185 |
| 2,6-Dinitrotoluene (2,6-DNT) | 6190 |
| 2-Amino-4,6-dinitrotoluene (2-am-DNT) | 9303 |
| 2-Nitrotoluene | 9507 |



Certificate of Accreditation: Supplement

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6681 Southpoint Parkway, Jacksonville, FL 32216

Contact Name: Todd Romero Phone 352-538-4939, Heather Quilal-lan Phone: 904-363-9350

Accreditation is granted to the facility to perform the following testing:

Code

Organic

| | |
|--|----------|
| EPA 8330 by High Performance Liquid Chromatography (HPLC) | 10308006 |
| Solid | |
| 3,5-Dinitroaniline | 6150 |
| 3-Nitrotoluene (3-NT) | 9510 |
| 4-Amino-2,6-dinitrotoluene (4-am-DNT) | 9306 |
| 4-Nitrotoluene (4-NT) | 9513 |
| Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) | 9432 |
| Methyl-2,4,6-Trinitrophenylnitramine (tetryl) | 6415 |
| Nitrobenzene | 5015 |
| Nitroglycerin | 6485 |
| Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) | 9522 |
| Pentaerythritoltetranitrate (PETN) | 9558 |
| EPA RSK-175 by Gas Chromatography Flame Ionization Detection (GC/FID) | 10212905 |
| Aqueous | |
| Ethane | 4747 |
| Ethylene (as Ethylene Glycol) | 4785 |
| Methane | 4926 |
| FL-PRO by Gas Chromatography Flame Ionization Detection (GC/FID) | 90015808 |
| Aqueous | |
| Total Petroleum Hydrocarbons (TPH) | 2050 |
| Total Petroleum Hydrocarbons Diesel Range Organics (TPH DRO) (C10 - C28) | 9369 |
| Solid | |
| Total Petroleum Hydrocarbons (TPH) | 2050 |
| Total Petroleum Hydrocarbons Diesel Range Organics (TPH DRO) (C10 - C28) | 9369 |
| MADEP EPH by Gas Chromatography Flame Ionization Detection (GC/FID) | 90017202 |
| Aqueous | |
| Total Petroleum Hydrocarbons Diesel Range Organics (TPH DRO) (C10 - C28) | 9369 |
| Solid | |
| EPH Aromatic C11-C22 (Unadjusted) | 6234 |
| Extractable Petroleum Hydrocarbons (EPH) (Aliphatic >C19-C36) | 6218 |
| Extractable Petroleum Hydrocarbons (EPH) (Aliphatic >C9-C18) | 6222 |
| Total Petroleum Hydrocarbons Diesel Range Organics (TPH DRO) (C10 - C28) | 9369 |
| MADEP VPH by Gas Chromatography Flame Ionization Detection (GC/FID) | 90017451 |
| Aqueous | |
| Total Petroleum Hydrocarbons Gasoline Range Organics (TPH GRO) (C6 – C10) | 9408 |
| Solid | |
| Total Petroleum Hydrocarbons Gasoline Range Organics (TPH GRO) (C6 – C10) | 9408 |
| Volatile Petroleum Hydrocarbons (VPH) (Aliphatic C5-C8 Unadjusted) | 5305 |



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6681 Southpoint Parkway, Jacksonville, FL 32216

Contact Name: Todd Romero Phone 352-538-4939, Heather Quilal-Ian Phone: 904-363-9350

Accreditation is granted to the facility to perform the following testing:

Code

Organic

MADEP VPH by Gas Chromatography Flame Ionization Detection (GC/FID) 90017451

Solid

Volatile Petroleum Hydrocarbons (VPH) (Aliphatic C9-C12 Unadjusted) 5307

Volatile Petroleum Hydrocarbons (VPH) (Unadjusted C9-C10) 5311

PFAS by LC/MS/MS Compliant with Table B-15 of QSM 5.4 or Latest Version by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) 90000451

Aqueous

11-Chloroeicosafluoro-3-Oxaundecane-1-Sulfonic Acid (11Cl-PF3OUdS) 9490

1H, 1H, 2H, 2H-Perfluorodecane Sulfonic Acid (8:2 FTS) 6948

1H, 1H, 2H, 2H-Perfluorohexane Sulfonic Acid (4:2 FTS) 6946

1H, 1H, 2H, 2H-Perfluorooctane Sulfonic Acid (6:2 FTS) 6947

4,8-Dioxa-3H-Perfluorononanoic Acid (ADONA) 6951

9-Chlorohexadecafluoro-3-Oxanonane-1-Sulfonic Acid (9-Cl-PF3ONS) 6952

Hexafluoropropylene Oxide Dimer Acid (HFPO-DA) – GenX 9460

n-Ethylperfluorooctane Sulfonamido Acetic Acid (NEtFOSAA) 4846

n-Methylperfluorooctane Sulfonamido Acetic Acid (NMeFOSAA) 4847

Nonafluoro-3,6-Dioxaheptanoic Acid (NFDHA) 6956

Perfluoro(2-Ethoxyethane)Sulfonic Acid (PFEESA) 6957

Perfluoro-3-Methoxypropanoic Acid (PFMPA) 6965

Perfluoro-4-Methoxybutanoic Acid (PFMBA) 6966

Perfluorobutane Sulfonic Acid (PFBS) 6918

Perfluorobutanoic Acid (PFBA) 6915

Perfluorodecane Sulfonic Acid (PFDS) 6920

Perfluorodecanoic Acid (PFDA) 6905

Perfluorododecanoic Acid (PFDoA) 6903

Perfluoroheptane Sulfonic Acid (PFHpS) 9470

Perfluoroheptanoic Acid (PFHpA) 6908

Perfluorohexane Sulfonic Acid (PFHxS) 6927

Perfluorohexanoic Acid (PFHxA) 6913

Perfluorononane Sulfonic Acid (PFNS) 6929

Perfluorononanoic Acid (PFNA) 6906

Perfluorooctane Sulfonamide (PFOSA) 6917

Perfluorooctane Sulfonic Acid (PFOS) 6931

Perfluorooctanoic Acid (PFOA) 6912

Perfluoropentane Sulfonic Acid (PFPeS) 6934

Perfluoropentanoic Acid (PFPeA) 6914

Perfluorotetradecanoic Acid (PFTeDA) 6902

Perfluorotridecanoic Acid (PFTrDA) 9563



Certificate of Accreditation: Supplement

Advanced Environmental Laboratories, Inc.

6681 Southpoint Parkway, Jacksonville, FL 32216

Contact Name: Todd Romero Phone 352-538-4939, Heather Quilal-lan Phone: 904-363-9350

Accreditation is granted to the facility to perform the following testing:

Code

Organic

PFAS by LC/MS/MS Compliant with Table B-15 of QSM 5.4 or Latest Version by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) 90000451

Aqueous

| | |
|--|------|
| Perfluoroundecanoic Acid (PFUnDA) | 6904 |
| Sum Perfluorooctanoic Acid (PFOA) + Perfluorooctane Sulfonic Acid (PFOS) Total (Calculation) | 6894 |

Solid

| | |
|--|------|
| 11-Chloroeicosafluoro-3-Oxaundecane-1-Sulfonic Acid (11Cl-PF3OUdS) | 9490 |
| 1H, 1H, 2H, 2H-Perfluorodecane Sulfonic Acid (8:2 FTS) | 6948 |
| 1H, 1H, 2H, 2H-Perfluorohexane Sulfonic Acid (4:2 FTS) | 6946 |
| 1H, 1H, 2H, 2H-Perfluorooctane Sulfonic Acid (6:2 FTS) | 6947 |
| 4,8-Dioxa-3H-Perfluorononanoic Acid (ADONA) | 6951 |
| 9-Chlorohexadecafluoro-3-Oxanonane-1-Sulfonic Acid (9-Cl-PF3ONS) | 6952 |
| Hexafluoropropylene Oxide Dimer Acid (HFPO-DA) – GenX | 9460 |
| n-Ethylperfluorooctane Sulfonamido Acetic Acid (NEtFOSAA) | 4846 |
| n-Methylperfluorooctane Sulfonamido Acetic Acid (NMeFOSAA) | 4847 |
| Nonafluoro-3,6-Dioxaheptanoic Acid (NFDHA) | 6956 |
| Perfluoro(2-Ethoxyethane)Sulfonic Acid (PFEESA) | 6957 |
| Perfluoro-3-Methoxypropanoic Acid (PFMPA) | 6965 |
| Perfluoro-4-Methoxybutanoic Acid (PFMBA) | 6966 |
| Perfluorobutane Sulfonic Acid (PFBS) | 6918 |
| Perfluorobutanoic Acid (PFBA) | 6915 |
| Perfluorodecane Sulfonic Acid (PFDS) | 6920 |
| Perfluorodecanoic Acid (PFDA) | 6905 |
| Perfluorododecanoic Acid (PFDoA) | 6903 |
| Perfluoroheptane Sulfonic Acid (PFHpS) | 9470 |
| Perfluoroheptanoic Acid (PFHpA) | 6908 |
| Perfluorohexane Sulfonic Acid (PFHxS) | 6927 |
| Perfluorohexanoic Acid (PFHxA) | 6913 |
| Perfluorononane Sulfonic Acid (PFNS) | 6929 |
| Perfluorononanoic Acid (PFNA) | 6906 |
| Perfluorooctane Sulfonamide (PFOSA) | 6917 |
| Perfluorooctane Sulfonic Acid (PFOS) | 6931 |
| Perfluorooctanoic Acid (PFOA) | 6912 |
| Perfluoropentane Sulfonic Acid (PFPeS) | 6934 |
| Perfluoropentanoic Acid (PFPeA) | 6914 |
| Perfluorotetradecanoic Acid (PFTeDA) | 6902 |
| Perfluorotridecanoic Acid (PFTrDA) | 9563 |
| Perfluoroundecanoic Acid (PFUnDA) | 6904 |



Certificate of Accreditation: Supplement

Advanced Environmental Laboratories, Inc.

6681 Southpoint Parkway, Jacksonville, FL 32216

Contact Name: Todd Romero Phone 352-538-4939, Heather Quilal-Ian Phone: 904-363-9350

Accreditation is granted to the facility to perform the following testing:

Code

Organic

PFAS by LC/MS/MS Compliant with Table B-15 of QSM 5.4 or Latest Version by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) 90000451

Solid
Sum Perfluorooctanoic Acid (PFOA) + Perfluorooctane Sulfonic Acid (PFOS) Total (Calculation) 6894

Preparation

Aqueous

| | |
|-----------|---|
| EPA 1311 | Toxicity Characteristic Leaching Procedure (TCLP) |
| EPA 1312 | Synthetic Precipitation Leaching Procedure (SPLP) |
| EPA 3005A | Acid Digestion of Waters for Total Recoverable or Dissolved Metals |
| EPA 3010A | Acid Digestion for Total Metals (Hot Block) |
| EPA 3020A | Acid Digestion of Aqueous Samples and Extracts for Total Metals |
| EPA 3510C | Separatory Funnel Liquid-Liquid Extraction |
| EPA 3580A | Waste Dilution Extraction for Extractable Organics |
| EPA 3620B | Florisil Cleanup |
| EPA 3630C | Silica Gel Clean Up |
| EPA 3660B | Sulfur Cleanup |
| EPA 3665A | Sulfuric Acid Cleanup |
| EPA 5030B | Purge-and-Trap for Volatile Organics in Aqueous Samples |
| EPA 5035A | Purge-and-Trap and Extraction For Volatile Organics (Closed-System) |

Solid

| | |
|-----------|---|
| EPA 1311 | Toxicity Characteristic Leaching Procedure (TCLP) |
| EPA 1312 | Synthetic Precipitation Leaching Procedure (SPLP) |
| EPA 3010A | Acid Digestion for Total Metals (Hot Block) |
| EPA 3020A | Acid Digestion of Aqueous Samples and Extracts for Total Metals |
| EPA 3050B | Acid Digestion for Metals |
| EPA 3550C | Ultrasonic Extraction |
| EPA 3580A | Waste Dilution Extraction for Extractable Organics |
| EPA 3620B | Florisil Cleanup |
| EPA 3630C | Silica Gel Clean Up |
| EPA 3660B | Sulfur Cleanup |
| EPA 3665A | Sulfuric Acid Cleanup |
| EPA 5035A | Purge-and-Trap and Extraction For Volatile Organics (Closed-System) |

Footnotes:

> Method codes are typically based on The NELAC Institute (TNI) Laboratory Accreditation Management System (LAMS) and are used to compare to the laboratory reported Performance Test (PT) results. Although the method code may not represent the specific method version, it is the method code used to represent the method/technology used to report PTs. (NC = No Code)



STATE OF WASHINGTON

DEPARTMENT OF ECOLOGY

PO Box 488 • Manchester, WA 98353-0488 • (360) 871-8840

March 22, 2023

Heather Ann Joy Quilal-lan
Advanced Environmental Laboratories, Inc.- Jacksonville
6681 Southpoint Parkway
Jacksonville,FL 32216

Dear Heather Ann Joy Quilal-lan:

Thank you for submitting the required supporting documentation for adding third party recognition accreditation N-Ethylperfluorooctane sulfonamide by EPA 1633 2nd draft in non-potable water.

The labs supporting documentation submitted was:
Passing PT WP-031523S
Current Florida NELAP Scope

Remember two acceptable PT samples are required for the Labs March 2024 renewal.

As a reminder, continued participation in the Ecology Lab Accreditation Program requires the lab to:

- Submit a renewal application and fees annually.
- Report significant changes in facility, personnel, analytical methods, equipment, the lab's quality assurance (QA) manual or QA procedures as they occur.
- **Participate in proficiency testing studies semi-annually, with the following exception: For each parameter where all required PT samples were analyzed and all results were satisfactory, you are required to submit only one PT result over this next year, and in subsequent years, as long as the results are satisfactory.**
- Submit copies of current third-party Scopes of Accreditation when they are available.

Your Right To Appeal

You have a right to appeal Ecology's decision to the Pollution Control Hearing Board (PCHB) within 30 days of the date of receipt of this decision letter. The appeal process is governed by Chapter 43.21B RCW and Chapter 371-08 WAC. "Date of receipt" is defined in RCW 43.21B.001(2).

To appeal you must do the following within 30 days of the date of receipt of this decision:

- File your appeal and a copy of this decision with the PCHB (see addresses below). Filing means actual receipt by the PCHB during regular business hours.
- Serve a copy of your appeal and this decision on Ecology in paper form - by mail or in person. (See addresses below.) E-mail is not accepted.

You must also comply with other applicable requirements in Chapter 43.21B RCW and Chapter 371-08 WAC.

Address And Location Information

Street Addresses:

Department of Ecology

Attn: Appeals Processing Desk
300 Desmond Drive SE
Lacey, WA 98503

Pollution Control Hearings Board

1111 Israel RD SW
STE 301
Tumwater, WA 98501

Mailing Addresses:

Department of Ecology

Attn: Appeals Processing Desk
PO Box 47608
Olympia, WA 98504-7608

Pollution Control Hearings Board

PO Box 40903
Olympia, WA 98504-0903

E-Mail Address:

Department of Ecology

Not currently available (see WAC 371-08)

Pollution Control Hearings Board

Pchb-shbappeals@elaho.wa.gov

If you have any questions concerning the accreditation of your lab, please contact Kamilee Ginder by e-mail at kamilee.ginder@ecy.wa.gov.

Sincerely,

A handwritten signature in black ink, appearing to read "Rebecca Wood". The signature is fluid and cursive, with the first name being more prominent.

Rebecca Wood
Lab Accreditation Unit Supervisor

RW:KG:kg
Enclosures

WASHINGTON STATE DEPARTMENT OF ECOLOGY

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

SCOPE OF ACCREDITATION

Advanced Environmental Laboratories, Inc.- Jacksonville

Jacksonville, FL

is accredited for the analytes listed below using the methods indicated. Full accreditation is granted unless stated otherwise in a note. EPA is the U.S. Environmental Protection Agency. SM is "Standard Methods for the Examination of Water and Wastewater." SM refers to EPA approved method versions. ASTM is the American Society for Testing and Materials. USGS is the U.S. Geological Survey. AOAC is the Association of Official Analytical Chemists. Other references are described in notes.

| Matrix/Analyte | Method | Notes |
|--|---------|-------|
| Drinking Water | | |
| 11-Chloroeicosafuoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS) | EPA 533 | 1 |
| 1H,1H,2H,2H,-Perfluorodecanesulfonic acid (8:2 FTS) | EPA 533 | 1 |
| 1H,1H,2H,2H,-Perfluorooctanesulfonic acid (6:2 FTS) | EPA 533 | 1 |
| 1H,1H,2H,2H-Perfluorohexanesulfonic acid (4:2 FTS) | EPA 533 | 1 |
| 4,8-Dioxa-3H-perfluorononanoic acid (ADONA) | EPA 533 | 1 |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS) | EPA 533 | 1 |
| Hexafluoropropylene oxide dimer acid (HFPO-DA) | EPA 533 | 1 |
| Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) | EPA 533 | 1 |
| Perfluoro(2-ethoxyethane)sulfonic acid (PFEEESA) | EPA 533 | 1 |
| Perfluoro-3-methoxypropanoic acid (PFMPA) | EPA 533 | 1 |
| Perfluoro-4-methoxybutanoic acid (PFMBA) | EPA 533 | 1 |
| Perfluorobutane sulfonic acid (PFBS) | EPA 533 | 1 |
| Perfluorobutanoic acid (PFBA) | EPA 533 | 1 |
| Perfluorodecanoic acid (PFDA) | EPA 533 | 1 |
| Perfluorododecanoic acid (PFDoA) | EPA 533 | 1 |
| Perfluoroheptane sulfonic acid (PFHpS) | EPA 533 | 1 |
| Perfluoroheptanoic acid (PFHpA) | EPA 533 | 1 |
| Perfluorohexane sulfonic acid (PFHxS) | EPA 533 | 1 |
| Perfluorohexanoic acid (PFHxA) | EPA 533 | 1 |
| Perfluorononanoic acid (PFNA) | EPA 533 | 1 |
| Perfluorooctane sulfonic acid (PFOS) | EPA 533 | 1 |
| Perfluorooctanoic acid (PFOA) | EPA 533 | 1 |
| Perfluoropentane sulfonic acid (PFPeS) | EPA 533 | 1 |
| Perfluoropentanoic acid (PFPeA) | EPA 533 | 1 |
| Perfluoroundecanoic acid (PFUnA) | EPA 533 | 1 |

Advanced Environmental Laboratories, Inc.- Jacksonville

| Matrix/Analyte | Method | Notes |
|---|--------------------|-------|
| Non-Potable Water | | |
| Total Organic Carbon | EPA 415.1_1974 | 1,2 |
| pH | SM 4500-H+ B-2011 | 1 |
| Total Organic Carbon | SM 5310 C-2014 | 1 |
| 11-Chloroicosafuoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS) | EPA 1633 2nd Draft | 1 |
| 1H,1H,2H,2H,-Perfluorodecanesulfonic acid (8:2 FTS) | EPA 1633 2nd Draft | 1 |
| 1H,1H,2H,2H,-Perfluorooctanesulfonic acid (6:2 FTS) | EPA 1633 2nd Draft | 1 |
| 1H,1H,2H,2H-Perfluorohexanesulfonic acid (4:2 FTS) | EPA 1633 2nd Draft | 1 |
| 2H,2H,3H,3H-Perfluorodecanoic Acid (7:3 FTCA) | EPA 1633 2nd Draft | 1 |
| 2H,2H,3H,3H-Perfluoro-octanoic Acid (5:3 FTCA) | EPA 1633 2nd Draft | 1 |
| 4,4,5,5,6,6,6-Heptafluorohexanoic Acid (3:3 FTCA) | EPA 1633 2nd Draft | 1 |
| 4,8-Dioxa-3H-perfluorononanoic acid (ADONA) | EPA 1633 2nd Draft | 1 |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS) | EPA 1633 2nd Draft | 1 |
| Hexafluoropropylene oxide dimer acid (HFPO-DA) | EPA 1633 2nd Draft | 1 |
| N-Ethylperfluorooctane sulfonamide (EtFOSA) | EPA 1633 2nd Draft | 1 |
| N-Ethylperfluorooctane sulfonamido acetic acid (NEtFOSAA) | EPA 1633 2nd Draft | 1 |
| N-Ethylperfluorooctanesulfonamidoethanol (EtFOSE) | EPA 1633 2nd Draft | 1 |
| N-Methylperfluorooctane sulfonamide (MeFOSA) | EPA 1633 2nd Draft | 1 |
| N-Methylperfluorooctane sulfonamido acetic acid (NMeFOSAA) | EPA 1633 2nd Draft | 1 |
| N-Methylperfluorooctanesulfonamidoethanol (MeFOSE) | EPA 1633 2nd Draft | 1 |
| Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) | EPA 1633 2nd Draft | 1 |
| Perfluoro(2-ethoxyethane)sulfonic acid (PFEEESA) | EPA 1633 2nd Draft | 1 |
| Perfluoro-3-methoxypropanoic acid (PFMPA) | EPA 1633 2nd Draft | 1 |
| Perfluoro-4-methoxybutanoic acid (PFMBA) | EPA 1633 2nd Draft | 1 |
| Perfluorobutane sulfonic acid (PFBS) | EPA 1633 2nd Draft | 1 |
| Perfluorobutanoic acid (PFBA) | EPA 1633 2nd Draft | 1 |
| Perfluorodecane sulfonic acid (PFDS) | EPA 1633 2nd Draft | 1 |
| Perfluorodecanoic acid (PFDA) | EPA 1633 2nd Draft | 1 |
| Perfluorododecane sulfonic acid (PFDoS) | EPA 1633 2nd Draft | 1 |
| Perfluorododecanoic acid (PFDoA) | EPA 1633 2nd Draft | 1 |
| Perfluoroheptane sulfonic acid (PFHpS) | EPA 1633 2nd Draft | 1 |
| Perfluoroheptanoic acid (PFHpA) | EPA 1633 2nd Draft | 1 |
| Perfluorohexane sulfonic acid (PFHxS) | EPA 1633 2nd Draft | 1 |
| Perfluorohexanoic acid (PFHxA) | EPA 1633 2nd Draft | 1 |
| Perfluorononane sulfonic acid (PFNS) | EPA 1633 2nd Draft | 1 |
| Perfluorononanoic acid (PFNA) | EPA 1633 2nd Draft | 1 |
| Perfluorooctane sulfonamide (PFOSA) | EPA 1633 2nd Draft | 1 |
| Perfluorooctane sulfonic acid (PFOS) | EPA 1633 2nd Draft | 1 |

Advanced Environmental Laboratories, Inc.- Jacksonville

| Matrix/Analyte | Method | Notes |
|--|--------------------|-------|
| Non-Potable Water | | |
| Perfluorooctanoic acid (PFOA) | EPA 1633 2nd Draft | 1 |
| Perfluoropentane sulfonic acid (PFPeS) | EPA 1633 2nd Draft | 1 |
| Perfluoropentanoic acid (PFPeA) | EPA 1633 2nd Draft | 1 |
| Perfluorotetradecanoic acid (PFTeDA) | EPA 1633 2nd Draft | 1 |
| Perfluorotridecanoic acid (PFTrDA) | EPA 1633 2nd Draft | 1 |
| Perfluoroundecanoic acid (PFUnA) | EPA 1633 2nd Draft | 1 |
| Solid and Chemical Materials | | |
| pH | EPA 9045 D_2004 | 1 |
| Total Organic Carbon | EPA 9060A | 1 |
| 11-Chloroeicosafuoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS) | EPA 1633 2nd Draft | 1 |
| 1H,1H,2H,2H,-Perfluorodecanesulfonic acid (8:2 FTS) | EPA 1633 2nd Draft | 1 |
| 1H,1H,2H,2H,-Perfluorooctanesulfonic acid (6:2 FTS) | EPA 1633 2nd Draft | 1 |
| 1H,1H,2H,2H-Perfluorohexanesulfonic acid (4:2 FTS) | EPA 1633 2nd Draft | 1 |
| 2H,2H,3H,3H-Perfluorodecanoic Acid (7:3 FTCA) | EPA 1633 2nd Draft | 1 |
| 2H,2H,3H,3H-Perfluoro-octanoic Acid (5:3 FTCA) | EPA 1633 2nd Draft | 1 |
| 4,4,5,5,6,6,6-Heptafluorohexanoic Acid (3:3 FTCA) | EPA 1633 2nd Draft | 1 |
| 4,8-Dioxa-3H-perfluorononanoic acid (ADONA) | EPA 1633 2nd Draft | 1 |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS) | EPA 1633 2nd Draft | 1 |
| Hexafluoropropylene oxide dimer acid (HFPO-DA) | EPA 1633 2nd Draft | 1 |
| N-Ethylperfluorooctane sulfonamide (EtFOSA) | EPA 1633 2nd Draft | 1 |
| N-Ethylperfluorooctane sulfonamido acetic acid (NEtFOSAA) | EPA 1633 2nd Draft | 1 |
| N-Ethylperfluorooctanesulfonamidoethanol (EtFOSE) | EPA 1633 2nd Draft | 1 |
| N-Methylperfluorooctane sulfonamide (MeFOSA) | EPA 1633 2nd Draft | 1 |
| N-Methylperfluorooctane sulfonamido acetic acid (NMeFOSAA) | EPA 1633 2nd Draft | 1 |
| N-Methylperfluorooctanesulfonamido ethanol (MeFOSE) | EPA 1633 2nd Draft | 1 |
| Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) | EPA 1633 2nd Draft | 1 |
| Perfluoro(2-ethoxyethane)sulfonic acid (PFEEESA) | EPA 1633 2nd Draft | 1 |
| Perfluoro-3-methoxypropanoic acid (PFMPA) | EPA 1633 2nd Draft | 1 |
| Perfluoro-4-methoxybutanoic acid (PFMBA) | EPA 1633 2nd Draft | 1 |
| Perfluorobutane sulfonic acid (PFBS) | EPA 1633 2nd Draft | 1 |
| Perfluorobutanoic acid (PFBA) | EPA 1633 2nd Draft | 1 |
| Perfluorodecane sulfonic acid (PFDS) | EPA 1633 2nd Draft | 1 |
| Perfluorodecanoic acid (PFDA) | EPA 1633 2nd Draft | 1 |
| Perfluorododecane sulfonic acid (PFDoS) | EPA 1633 2nd Draft | 1 |
| Perfluorododecanoic acid (PFDoA) | EPA 1633 2nd Draft | 1 |
| Perfluoroheptane sulfonic acid (PFHpS) | EPA 1633 2nd Draft | 1 |
| Perfluoroheptanoic acid (PFHpA) | EPA 1633 2nd Draft | 1 |

| Matrix/Analyte | Method | Notes |
|--|--------------------|-------|
| Solid and Chemical Materials | | |
| Perfluorohexane sulfonic acid (PFHxS) | EPA 1633 2nd Draft | 1 |
| Perfluorohexanoic acid (PFHxA) | EPA 1633 2nd Draft | 1 |
| Perfluorononane sulfonic acid (PFNS) | EPA 1633 2nd Draft | 1 |
| Perfluorononanoic acid (PFNA) | EPA 1633 2nd Draft | 1 |
| Perfluorooctane sulfonamide (PFOSA) | EPA 1633 2nd Draft | 1 |
| Perfluorooctane sulfonic acid (PFOS) | EPA 1633 2nd Draft | 1 |
| Perfluorooctanoic acid (PFOA) | EPA 1633 2nd Draft | 1 |
| Perfluoropentane sulfonic acid (PFPeS) | EPA 1633 2nd Draft | 1 |
| Perfluoropentanoic acid (PFPeA) | EPA 1633 2nd Draft | 1 |
| Perfluorotetradecanoic acid (PFTeDA) | EPA 1633 2nd Draft | 1 |
| Perfluorotridecanoic acid (PFTTrDA) | EPA 1633 2nd Draft | 1 |
| Perfluoroundecanoic acid (PFUnA) | EPA 1633 2nd Draft | 1 |

Accredited Parameter Note Detail

1) Accreditation based in part on recognition of Florida NELAP accreditation. 2) Method not approved for NPDES testing.



03/22/2023

Authentication Signature
 Rebecca Wood, Lab Accreditation Unit Supervisor

Date



STATE OF WASHINGTON

DEPARTMENT OF ECOLOGY

PO Box 488 • Manchester, WA 98353-0488 • (360) 871-8840

June 30, 2023

Heather Ann Joy Quilal-lan
Advanced Environmental Laboratories, Inc.- Jacksonville
6681 Southpoint Parkway
Jacksonville, FL 32216

Dear Heather Ann Joy Quilal-lan:

Your request to add EPA 537.1 in drinking water to your WA scope of accreditation has been processed. Your revised scope is effective July 1, 2023.

Thank you for submitting the required supporting documentation:

Florida accreditation scope

PT WS-R35082 3/1/23

PT WS-030923T 3/16/23

PT WS-0423 5/25/23

Accreditation is based on third party recognition of the Labs Florida scope.

Accreditation is based in part on the Labs performance in the past twelve months in Performance Test (PT) studies.

As a reminder, continued participation in the Ecology Lab Accreditation Program requires the lab to:

- Submit a renewal application and fees annually.
- Report significant changes in facility, personnel, analytical methods, equipment, the lab's quality assurance (QA) manual or QA procedures as they occur.
- **Participate in proficiency testing studies semi-annually, with the following exception: For each parameter where all required PT were analyzed and all results were satisfactory, you are required to submit only one PT result over this next year, and in subsequent years, as long as the results are satisfactory.**

If you have any questions concerning the accreditation of your lab, please contact Kamilee Ginder by e-mail at kamilee.ginder@ecy.wa.gov.

Sincerely,

A handwritten signature in black ink that reads "Rebecca Wood".

Rebecca Wood
Lab Accreditation Unit Supervisor

RW:KG:kg
Enclosures

WASHINGTON STATE DEPARTMENT OF ECOLOGY

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

SCOPE OF ACCREDITATION

Advanced Environmental Laboratories, Inc.- Jacksonville

Jacksonville, FL

is accredited for the analytes listed below using the methods indicated. Full accreditation is granted unless stated otherwise in a note. EPA is the U.S. Environmental Protection Agency. SM is "Standard Methods for the Examination of Water and Wastewater." SM refers to EPA approved method versions. ASTM is the American Society for Testing and Materials. USGS is the U.S. Geological Survey. AOAC is the Association of Official Analytical Chemists. Other references are described in notes.

| Matrix/Analyte | Method | Notes |
|--|-----------------------------|-------|
| Drinking Water | | |
| 11-Chloroeicosafuoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS) | EPA 533 | 1 |
| 1H,1H,2H,2H,-Perfluorodecanesulfonic acid (8:2 FTS) | EPA 533 | 1 |
| 1H,1H,2H,2H,-Perfluorooctanesulfonic acid (6:2 FTS) | EPA 533 | 1 |
| 1H,1H,2H,2H-Perfluorohexanesulfonic acid (4:2 FTS) | EPA 533 | 1 |
| 4,8-Dioxa-3H-perfluorononanoic acid (ADONA) | EPA 533 | 1 |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS) | EPA 533 | 1 |
| Hexafluoropropylene oxide dimer acid (HFPO-DA) | EPA 533 | 1 |
| Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) | EPA 533 | 1 |
| Perfluoro(2-ethoxyethane)sulfonic acid (PFEEESA) | EPA 533 | 1 |
| Perfluoro-3-methoxypropanoic acid (PFMPA) | EPA 533 | 1 |
| Perfluoro-4-methoxybutanoic acid (PFMBA) | EPA 533 | 1 |
| Perfluorobutane sulfonic acid (PFBS) | EPA 533 | 1 |
| Perfluorobutanoic acid (PFBA) | EPA 533 | 1 |
| Perfluorodecanoic acid (PFDA) | EPA 533 | 1 |
| Perfluorododecanoic acid (PFDoA) | EPA 533 | 1 |
| Perfluoroheptane sulfonic acid (PFHpS) | EPA 533 | 1 |
| Perfluoroheptanoic acid (PFHpA) | EPA 533 | 1 |
| Perfluorohexane sulfonic acid (PFHxS) | EPA 533 | 1 |
| Perfluorohexanoic acid (PFHxA) | EPA 533 | 1 |
| Perfluorononanoic acid (PFNA) | EPA 533 | 1 |
| Perfluorooctane sulfonic acid (PFOS) | EPA 533 | 1 |
| Perfluorooctanoic acid (PFOA) | EPA 533 | 1 |
| Perfluoropentane sulfonic acid (PFPeS) | EPA 533 | 1 |
| Perfluoropentanoic acid (PFPeA) | EPA 533 | 1 |
| Perfluoroundecanoic acid (PFUnA) | EPA 533 | 1 |
| 11-Chloroeicosafuoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS) | EPA 537.1 revision 2 (3/20) | 1 |

Advanced Environmental Laboratories, Inc.- Jacksonville

| Matrix/Analyte | Method | Notes |
|--|-----------------------------|-------|
| Drinking Water | | |
| 4,8-Dioxa-3H-perfluorononanoic acid (ADONA) | EPA 537.1 revision 2 (3/20) | 1 |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS) | EPA 537.1 revision 2 (3/20) | 1 |
| Hexafluoropropylene oxide dimer acid (HFPO-DA) | EPA 537.1 revision 2 (3/20) | 1 |
| N-Ethylperfluorooctane sulfonamido acetic acid (NEtFOSAA) | EPA 537.1 revision 2 (3/20) | 1 |
| N-Methylperfluorooctane sulfonamido acetic acid (NMeFOSAA) | EPA 537.1 revision 2 (3/20) | 1 |
| Perfluorobutane sulfonic acid (PFBS) | EPA 537.1 revision 2 (3/20) | 1 |
| Perfluorodecanoic acid (PFDA) | EPA 537.1 revision 2 (3/20) | 1 |
| Perfluorododecanoic acid (PFDoA) | EPA 537.1 revision 2 (3/20) | 1 |
| Perfluoroheptanoic acid (PFHpA) | EPA 537.1 revision 2 (3/20) | 1 |
| Perfluorohexane sulfonic acid (PFHxS) | EPA 537.1 revision 2 (3/20) | 1 |
| Perfluorohexanoic acid (PFHxA) | EPA 537.1 revision 2 (3/20) | 1 |
| Perfluorononanoic acid (PFNA) | EPA 537.1 revision 2 (3/20) | 1 |
| Perfluorooctane sulfonic acid (PFOS) | EPA 537.1 revision 2 (3/20) | 1 |
| Perfluorooctanoic acid (PFOA) | EPA 537.1 revision 2 (3/20) | 1 |
| Perfluorotetradecanoic acid (PFTeDA) | EPA 537.1 revision 2 (3/20) | 1 |
| Perfluorotridecanoic acid (PFTrDA) | EPA 537.1 revision 2 (3/20) | 1 |
| Perfluoroundecanoic acid (PFUnA) | EPA 537.1 revision 2 (3/20) | 1 |
| Non-Potable Water | | |
| Total Organic Carbon | EPA 415.1_1974 | 1,2 |
| pH | SM 4500-H+ B-2011 | 1 |
| Total Organic Carbon | SM 5310 C-2014 | 1 |
| 11-Chloroeicosafuoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS) | EPA 1633 2nd Draft | 1 |
| 1H,1H,2H,2H,-Perfluorodecanesulfonic acid (8:2 FTS) | EPA 1633 2nd Draft | 1 |
| 1H,1H,2H,2H,-Perfluorooctanesulfonic acid (6:2 FTS) | EPA 1633 2nd Draft | 1 |
| 1H,1H,2H,2H-Perfluorohexanesulfonic acid (4:2 FTS) | EPA 1633 2nd Draft | 1 |
| 2H,2H,3H,3H-Perfluorodecanoic Acid (7:3 FTCA) | EPA 1633 2nd Draft | 1 |
| 2H,2H,3H,3H-Perfluoro-octanoic Acid (5:3 FTCA) | EPA 1633 2nd Draft | 1 |
| 4,4,5,5,6,6,6-Heptafluorohexanoic Acid (3:3 FTCA) | EPA 1633 2nd Draft | 1 |
| 4,8-Dioxa-3H-perfluorononanoic acid (ADONA) | EPA 1633 2nd Draft | 1 |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS) | EPA 1633 2nd Draft | 1 |
| Hexafluoropropylene oxide dimer acid (HFPO-DA) | EPA 1633 2nd Draft | 1 |
| N-Ethylperfluorooctane sulfonamide (EtFOSA) | EPA 1633 2nd Draft | 1 |
| N-Ethylperfluorooctane sulfonamido acetic acid (NEtFOSAA) | EPA 1633 2nd Draft | 1 |
| N-Ethylperfluorooctanesulfonamidoethanol (EtFOSE) | EPA 1633 2nd Draft | 1 |
| N-Methylperfluorooctane sulfonamide (MeFOSA) | EPA 1633 2nd Draft | 1 |
| N-Methylperfluorooctane sulfonamido acetic acid (NMeFOSAA) | EPA 1633 2nd Draft | 1 |
| N-Methylperfluorooctanesulfonamidoethanol (MeFOSE) | EPA 1633 2nd Draft | 1 |

Advanced Environmental Laboratories, Inc.- Jacksonville

| Matrix/Analyte | Method | Notes |
|--|--------------------|-------|
| Non-Potable Water | | |
| Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) | EPA 1633 2nd Draft | 1 |
| Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA) | EPA 1633 2nd Draft | 1 |
| Perfluoro-3-methoxypropanoic acid (PFMPA) | EPA 1633 2nd Draft | 1 |
| Perfluoro-4-methoxybutanoic acid (PFMBA) | EPA 1633 2nd Draft | 1 |
| Perfluorobutane sulfonic acid (PFBS) | EPA 1633 2nd Draft | 1 |
| Perfluorobutanoic acid (PFBA) | EPA 1633 2nd Draft | 1 |
| Perfluorodecane sulfonic acid (PFDS) | EPA 1633 2nd Draft | 1 |
| Perfluorodecanoic acid (PFDA) | EPA 1633 2nd Draft | 1 |
| Perfluorododecane sulfonic acid (PFDoS) | EPA 1633 2nd Draft | 1 |
| Perfluorododecanoic acid (PFDoA) | EPA 1633 2nd Draft | 1 |
| Perfluoroheptane sulfonic acid (PFHpS) | EPA 1633 2nd Draft | 1 |
| Perfluoroheptanoic acid (PFHpA) | EPA 1633 2nd Draft | 1 |
| Perfluorohexane sulfonic acid (PFHxS) | EPA 1633 2nd Draft | 1 |
| Perfluorohexanoic acid (PFHxA) | EPA 1633 2nd Draft | 1 |
| Perfluorononane sulfonic acid (PFNS) | EPA 1633 2nd Draft | 1 |
| Perfluorononanoic acid (PFNA) | EPA 1633 2nd Draft | 1 |
| Perfluorooctane sulfonamide (PFOSA) | EPA 1633 2nd Draft | 1 |
| Perfluorooctane sulfonic acid (PFOS) | EPA 1633 2nd Draft | 1 |
| Perfluorooctanoic acid (PFOA) | EPA 1633 2nd Draft | 1 |
| Perfluoropentane sulfonic acid (PFPeS) | EPA 1633 2nd Draft | 1 |
| Perfluoropentanoic acid (PFPeA) | EPA 1633 2nd Draft | 1 |
| Perfluorotetradecanoic acid (PFTeDA) | EPA 1633 2nd Draft | 1 |
| Perfluorotridecanoic acid (PFTrDA) | EPA 1633 2nd Draft | 1 |
| Perfluoroundecanoic acid (PFUnA) | EPA 1633 2nd Draft | 1 |
| Solid and Chemical Materials | | |
| pH | EPA 9045 D_2004 | 1 |
| Total Organic Carbon | EPA 9060A | 1 |
| 11-Chloroeicosafuoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS) | EPA 1633 2nd Draft | 1 |
| 1H,1H,2H,2H,-Perfluorodecanesulfonic acid (8:2 FTS) | EPA 1633 2nd Draft | 1 |
| 1H,1H,2H,2H,-Perfluorooctanesulfonic acid (6:2 FTS) | EPA 1633 2nd Draft | 1 |
| 1H,1H,2H,2H-Perfluorohexanesulfonic acid (4:2 FTS) | EPA 1633 2nd Draft | 1 |
| 2H,2H,3H,3H-Perfluorodecanoic Acid (7:3 FTCA) | EPA 1633 2nd Draft | 1 |
| 2H,2H,3H,3H-Perfluoro-octanoic Acid (5:3 FTCA) | EPA 1633 2nd Draft | 1 |
| 4,4,5,5,6,6,6-Heptafluorohexanoic Acid (3:3 FTCA) | EPA 1633 2nd Draft | 1 |
| 4,8-Dioxa-3H-perfluorononanoic acid (ADONA) | EPA 1633 2nd Draft | 1 |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS) | EPA 1633 2nd Draft | 1 |
| Hexafluoropropylene oxide dimer acid (HFPO-DA) | EPA 1633 2nd Draft | 1 |

Advanced Environmental Laboratories, Inc.- Jacksonville

| Matrix/Analyte | Method | Notes |
|--|--------------------|--------------|
| Solid and Chemical Materials | | |
| N-Ethylperfluorooctane sulfonamide (EtFOSA) | EPA 1633 2nd Draft | 1 |
| N-Ethylperfluorooctane sulfonamido acetic acid (NEtFOSAA) | EPA 1633 2nd Draft | 1 |
| N-Ethylperfluorooctanesulfonamidoethanol (EtFOSE) | EPA 1633 2nd Draft | 1 |
| N-Methylperfluorooctane sulfonamide (MeFOSA) | EPA 1633 2nd Draft | 1 |
| N-Methylperfluorooctane sulfonamido acetic acid (NMeFOSAA) | EPA 1633 2nd Draft | 1 |
| N-Methylperfluorooctanesulfonamido ethanol (MeFOSE) | EPA 1633 2nd Draft | 1 |
| Nonafluoro-3,6-dioxahexanoic acid (NFDHA) | EPA 1633 2nd Draft | 1 |
| Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA) | EPA 1633 2nd Draft | 1 |
| Perfluoro-3-methoxypropanoic acid (PFMPA) | EPA 1633 2nd Draft | 1 |
| Perfluoro-4-methoxybutanoic acid (PFMBA) | EPA 1633 2nd Draft | 1 |
| Perfluorobutane sulfonic acid (PFBS) | EPA 1633 2nd Draft | 1 |
| Perfluorobutanoic acid (PFBA) | EPA 1633 2nd Draft | 1 |
| Perfluorodecane sulfonic acid (PFDS) | EPA 1633 2nd Draft | 1 |
| Perfluorodecanoic acid (PFDA) | EPA 1633 2nd Draft | 1 |
| Perfluorododecane sulfonic acid (PFDoS) | EPA 1633 2nd Draft | 1 |
| Perfluorododecanoic acid (PFDoA) | EPA 1633 2nd Draft | 1 |
| Perfluoroheptane sulfonic acid (PFHpS) | EPA 1633 2nd Draft | 1 |
| Perfluoroheptanoic acid (PFHpA) | EPA 1633 2nd Draft | 1 |
| Perfluorohexane sulfonic acid (PFHxS) | EPA 1633 2nd Draft | 1 |
| Perfluorohexanoic acid (PFHxA) | EPA 1633 2nd Draft | 1 |
| Perfluorononane sulfonic acid (PFNS) | EPA 1633 2nd Draft | 1 |
| Perfluorononanoic acid (PFNA) | EPA 1633 2nd Draft | 1 |
| Perfluorooctane sulfonamide (PFOSA) | EPA 1633 2nd Draft | 1 |
| Perfluorooctane sulfonic acid (PFOS) | EPA 1633 2nd Draft | 1 |
| Perfluorooctanoic acid (PFOA) | EPA 1633 2nd Draft | 1 |
| Perfluoropentane sulfonic acid (PFPeS) | EPA 1633 2nd Draft | 1 |
| Perfluoropentanoic acid (PFPeA) | EPA 1633 2nd Draft | 1 |
| Perfluorotetradecanoic acid (PFTeDA) | EPA 1633 2nd Draft | 1 |
| Perfluorotridecanoic acid (PFTrDA) | EPA 1633 2nd Draft | 1 |
| Perfluoroundecanoic acid (PFUnA) | EPA 1633 2nd Draft | 1 |

Matrix/Analyte

Method

Notes

Accredited Parameter Note Detail

1) Accreditation based in part on recognition of Florida NELAP accreditation. 2) Method not approved for NPDES testing.



06/30/2023

Authentication Signature

Date

Rebecca Wood, Lab Accreditation Unit Supervisor



STANDARD OPERATING PROCEDURE

For

Sample Receipt, Handling, Storage, and Log In





SOP Revision Log for AEL SOP Admin 005

| Revision Number | Revision Date | Reason for Revision | Section(s) and Page(s) Revised |
|-----------------|---------------|--|--------------------------------|
| Revision 00 | 10/31/01 | Initial Creation | All sections affected |
| Revision 01 | 2/12/02 | Expand some detail, add references. | All sections affected |
| Revision 02 | 6/25/03 | Include detailed section on how to use software when logging in samples. | Section 5 |
| Revision 03 | 3/31/05 | Re-write to better include all Nelac language and required elements. Expand section 3. Chain of Custody added. Log-in Checklist added. | All sections affected |
| Revision 04 | 8/18/08 | Expanded procedures for splitting samples. Remove language specific to software log-in procedures. | Section 6 |
| Revision 05 | 9/15/11 | Revision to include all Nelac (TNI) and DOD ELAP language and required elements. Add in how multi-phase samples handled. | All sections affected |
| Revision 06 | 3/31/16 | Remove reference to Promium software. Update refrigerator placement at Miami and Tallahassee labs. Update references. | Sections 2, 3, 8, 9, 11 |
| Revision 07 | 7/27/18 | Define procedures for logging in and receiving samples from AEL sister labs (Sec. 9 new). Update standard references (Sec 1) Minor edits throughout SOP. Add Fort Myers to list of labs. | All sections affected |
| Revision 08 | 4/25/19 | Expand procedures for storage of “hot” samples away from other samples. Update references. | Section 1, 10 |
| Revision 09 | 07/27/22 | Tri-Annual Review. Minor grammatical updates and method updates made. Updated for Jacksonville laboratory expansion. | Multiple sections affected. |
| | | | |
| | | | |
| | | | |
| | | | |



Sample Receipt, Handling, Storage, and Log In

1.0 Scope and Application

- 1.1 This Administrative SOP in conjunction with Section 6 of the Advanced Environmental Laboratories Quality Manual covers the policies for sample receipt, handling, storage, and log in at all AEL facilities. Section 6 of the Quality Manual stands alone as AEL's Sample Acceptance Policy and should be referenced if that is the subject of interest. This SOP is for the internal activities and the procedures of such within each laboratory. AEL policies conform to the guidelines set forth in the TNI 2016 Standards, the DOD QSM ver 5.4, ISO 17025: 2005 & 2017, and the Florida Department of Health Standard Operating Procedures for Field Activities (eff 1/2017). All samples are to conform to the policies as listed in this SOP as well as the Quality Manual Section 6. Those samples submitted to AEL that do not conform to these policies are documented as non-conforming and listed as such in final reporting. See also supplemental SOP, AELSOP Admin-005a.

2.0 Summary of Procedure

- 2.1 The AEL Sample Receiving Department Staff is responsible for the receipt and login of samples that are received from clients via drop-off, AEL courier, or third-party shipping. Upon receipt, the condition of the samples are checked and documented along with the accuracy of the Chain of Custody (COC). After all non-conformances and client communications are documented the samples are logged into the LIMS software program Horizon. The samples are then stored in designated areas that are accessible to the analysts or in some cases are sub-contracted to internal or external laboratories.
- 2.2 See Section 13 of AEL's Quality Manual for **AEL's sampling plan**. **AEL's sampling plan** in general consists of implementing the procedures as outlined in the Florida Department of Health (FDEP) Standard Operating Procedures for Field Activities 01/2017 (eff 04/16/2018). A copy of these SOPs is assigned to each sampling vehicle and/or available electronically to have them present for reference by sampling personnel at the time of sampling. When a specific sampling plan is required by the client, those plans are followed. If the clients sampling plan deviates from the FDEP SOPs, this will be discussed with the client for either correction to conform to the FDEP SOPs or if the client elects not to conform, then the deviations are noted in the final report on the case narrative if the results are to be used for compliance to FDEP regulatory use. The results may also be qualified regardless, dependent on the nature of the deviations. These deviations should be documented at the time of login on non-conformation forms

3.0 Equipment

3.1 Sample Receipt

- 3.1.1 Infrared Thermometer.
- 3.1.2 Dispensing devices for adding preservatives (acids and bases).
- 3.1.3 pH test strips

3.2 Login

- 3.2.1 Login computer stations with LIMS software.
- 3.2.2 Sample label printer.
- 3.2.3 Laser Printer.



3.3 Sample Storage

3.3.1 Jacksonville

- 3.3.1.1 Walk-In Cooler located in the extractions lab
- 3.3.1.2 3-Door refrigerator located in the extractions lab (DW extractables)
- 3.3.1.3 VOC refrigerators located in the volatiles laboratory
- 3.3.1.4 SVOC refrigerators located in the semi-volatiles laboratories
- 3.3.1.5 LC refrigerator located in the LC laboratory
- 3.3.1.6 Microbiology refrigerators located in the micro lab
- 3.3.1.7 WetChem waste refrigerators located in the wet chemistry lab
- 3.3.1.8 Metals storage shelves located in the metals lab and outside hallway
- 3.3.1.9 Overflow refrigeration located in the DOD sample receiving room.

3.3.2 Tampa

- 3.3.2.1 Walk in Cooler located in garage bay area
- 3.3.2.2 Microbiology Refrigerator located in the micro lab
- 3.3.2.3 VOC refrigerator located in the volatiles laboratory
- 3.3.2.4 SVOC refrigerator located in the semi-volatiles laboratory
- 3.3.2.5 Metals storage shelves located in the metals lab

3.3.3 Gainesville

- 3.3.3.1 Walk in Cooler located on outside cement deck.
- 3.3.3.2 Short hold and shipping refrigerator located next to login.
- 3.3.3.3 Microbiology refrigerator located in the micro lab

3.3.4 Orlando

- 3.3.4.1 IC refrigerator located in the IC room for IC and turbidity samples
- 3.3.4.2 Microbiology Refrigerator located in the micro lab for micro samples
- 3.3.4.3 2-Door refrigerator located in main lab for most wetchem samples
- 3.3.4.4 3-Door refrigerator located in main lab for any overflow

3.3.5 Miami

- 3.3.5.1 Refrigerators located in the center of the lab
- 3.3.5.2 VOC refrigerators located in the volatiles laboratory
- 3.3.5.3 SVOC refrigerator located in the semi-volatiles laboratory
- 3.3.5.4 Microbiology refrigerator located in the micro lab

3.3.6 Tallahassee

- 3.3.6.1 Refrigerators located in the back room of the lab

3.3.7 Fort Myers

- 3.3.7.1 Refrigerators located in main hallway
- 3.3.7.2 Microbiology refrigerator located in the micro lab



3.4 Supplies

- 3.4.1 Latex powder free disposable gloves
- 3.4.2 pH paper
- 3.4.3 Plastic disposable pipettes
- 3.4.4 Labels for sample containers
- 3.4.5 Labels for project folders & Chain of Custody (COC)
- 3.4.6 Manila folders

4.0 Safety

- 4.1 See Standard Methods, Section 1090 (2010) Laboratory Occupation Safety and Health.
- 4.2 See AEL Chemical Hygiene Plan and Safety Manual. Rev 03, dated 09/03/2019 (or most recent revision).

5.0 Definitions:

- 5.1 Immediately: defined to be within 15 minutes.
- 5.2 Timely basis: defined to be within 2 hours.
- 5.3 Thermal preservation: preservation with certain temperature requirements. A requirement of 4°C in referenced methods is considered acceptable when measurement is within the range of 0-6°C within AEL.
- 5.4 pH Preservation: preservation using an acid or base to obtain a certain pH level.
- 5.5 For a complete listing of laboratory terms and definitions see Admin SOP 039.

6.0 Procedure

- 6.1 The conditions of samples upon receipt at AEL must be correctly accessed and documented on the COC. See attached Figure 1.0 "Chain of Custody Record." Anomalies are noted in the Sample Acceptance Discrepancies Log with project managers being notified. An NCF is generated if warranted.
- 6.2 The samples are accepted or rejected as part of the AEL Quality Manual Sample Acceptance Policy, detailed in Section 6.0 of the Quality Manual and posted in the receiving area of each laboratory. The items on the checklist listed in Table 1 found at the end of this SOP are performed/reviewed for each project received. If for DOD work, a form with check off boxes for these items will be completed.
- 6.3 Steps for accessing sample conditions:
 - 6.3.1 When the sample arrives, indicate on the COC the method shipped if shipped by third party courier service. Original shipping receipts are placed in a bin for later filing in the login file cabinet.



- 6.3.2 Examine the shipping container and note the presence/absence and condition of any custody seals.
- 6.3.3 Put on a pair of latex disposable powder free gloves. Place the cooler in a well-ventilated area, open it, determine if there is ice in the cooler, and check the appropriate boxes on the COC. The temperature measurement shall be taken from the "Temperature Blank" provided in the cooler if present. If no temperature blank(s) accompany the cooler, then the temperature is taken with an IR gun measuring the surface temperature of the sample containers.
- 6.3.4 NOTE: See preservation tables in FDEP SOP FS1000 for proper thermal preservation requirements.
 - 6.3.4.1 The target range is 0-6 degrees Celsius for samples that are thermally preserved.
 - 6.3.4.2 Turn on the thermometer. The default mode of the thermometer is degrees Celsius, however if degrees Fahrenheit is displayed press the C/F button until degrees Celsius is displayed. All recorded temperature readings will be in Celsius.
 - 6.3.4.3 Hold the IR sensor probe on the rear part of the handle. Holding the probe too far forward will cause values to drift. Point the probe sensing head at the object to be measured. The object shall be larger than the spot calculated by the measurement field/distance diagram calculated on the handle of IR probe. The sampling time is approximately one second. Record the temperature on the COC.
 - 6.3.4.4 Samples that are hand delivered to the lab on the same day that they were collected may not meet the 0-6 target range as they may not have been placed on ice long enough to reach equilibrium temperature. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.
- 6.4 Sign and document on the COC the date and time of receipt after the client has relinquished custody of the samples.
- 6.5 Sample receipt must be done in the following order:
 - 6.5.1 Projects containing samples with short hold times.
 - 6.5.2 Project corrections and projects that have a rush priority.
 - 6.5.3 Projects that have a standard turn around time.
- 6.6 After setting the sample receipt priorities remove the samples from the shipping container and organize them according to the client sample identifiers and by the tests required. Samples that exhibit a strong smell shall be kept as far away from other samples as possible given available space on the counter. Compare the number of containers received with the



number listed on the COC as well as the appropriate container types for the tests listed and document any discrepancies on the COC and in the Sample Acceptance Discrepancies Log. CAUTION: Always wear gloves when handling samples. Be careful when reaching into coolers since the glass containers sometimes break during shipment.

- 6.7 Verify the integrity and condition of all sample containers. Look for leakage, broken containers, contaminated coolers, odors, and samples past the recommended holding times, etc. Observations and exceptions are written in the Sample Acceptance Discrepancies Log kept in the log-in department.
- 6.8 Check the pH of all pH preserved samples. See preservation tables in FDEP SOP FS1000 for proper pH preservation.
 - 6.8.1 Using a disposable pipette for each sample container pull an aliquot of sample and place several drops on pH paper.
 - 6.8.2 Acid preserved samples shall have a pH of less than 2. (HCl, H₂SO₄, HNO₃).
 - 6.8.3 Base preserved samples shall have a pH of greater than 9 for sulfides (NaOH/ZnAc) and greater than 12 for cyanides (NaOH).
 - 6.8.4 Do not check the pH of VOC or Oil & Grease samples. They will be checked at the time of analysis. Also, no additional preservation checks (other than thermal) are required at this time for Microbiology samples.
 - 6.8.5 If the pH is outside criteria, adjust the pH by adding the appropriate preservative until criteria are met. Document the volume and type of preservative added on the NCF, including the lot number of the acid used. This volume cannot exceed 1% of the total volume of the sample. For example, when the sample volume is 1L (1000mL), the maximum preservative is 10mL. If maximum is reached without the appropriate pH being reached, then notify the project manager for that sample.
 - 6.8.6 Do not dispense aliquots back into the sample container and do not reuse the disposable pipettes.
 - 6.8.7 Make sure the sample is properly closed before proceeding to the next sample.
- 6.9 Check for air bubbles in all VOC vials by inverting the vial and tapping it lightly. If there are air bubbles present that are greater than one percent of the vial volume (usually 5mm in diameter) the client must be contacted, an NCF must be generated, and the deficiency must be noted in the Sample Acceptance Discrepancies Log.
- 6.10 Notify analysts and/or supervisors of receipt of samples with short-hold times or samples that the holding time is over one-half expired.
 - 6.10.1 When transferring custody of samples to an analyst before they are logged in to the LIMS system, label each container and COC with a unique ID# and make the analyst a copy of the COC for reference as to the sample ID, date/time collected, and tests required.



- 6.10.2 The analyst is responsible for having a properly labeled sample and to have appropriate paperwork before taking custody of that sample.
- 6.11 Splitting of samples: If a sample is received in a single container requesting analysis from different analytical groups, this shall be documented in the Sample Acceptance Discrepancies Log. If there is enough volume present, and the project manager approves it, the sample may be split into appropriate containers. Volatiles samples or samples of unusual matrices that require a hood or a special procedure shall be given to the appropriate department for splitting. Notify the department that such a split is needed. For volatiles, provide them with an empty container for the purpose of splitting and to act as a sample place-marker pending splitting.
- 6.12 If the nature of the liquid sample is not readily evident, a determination will need to be performed to ascertain if the sample is a water or water miscible matrix versus a solvent or non-miscible waste. This is done by taking a small portion of the sample in question (taking several drops using a borosilicate pipette) and placing the small portion in a beaker of di water. If the sample is water miscible then it is logged as water. If the sample is not water miscible (forms separate layers and does not go into the water) then it is logged as a waste.
- 6.13 Assign to each sample a unique laboratory sample identification number (Lab Sample ID). The lab sample ID is composed of five parts: the laboratory code, the year indicator, the sequential tracking number, the sequential sample number, and the bottle identifier (ex. J 22 1654 –01 A).
- 6.13.1 The laboratory code is used to distinguish between samples originating in one of the seven laboratories in the AEL laboratory system. Samples originating in the Jacksonville laboratory are assigned the lab code “J”. Similarly, lab codes are assigned to samples originating in our sister laboratories as “G” for Gainesville, “A” for Orlando (Altamonte Springs) “T” for Tampa, “M” for Miami, “F” for Fort Myers, and “S” for Tallahassee.
 - 6.13.2 The year indicator is the last two digits of the current year.
 - 6.13.3 The sequential tracking number is a five-digit number that is generated by the LIMS system.
 - 6.13.4 The sequential sample number is generally assigned to the samples in the same order as they appear on the COC. These shall be written on the right-hand side of each sample on the COC in blue or black ink.
 - 6.13.5 The bottle identifier is an alphanumeric number that is assigned to each bottle received to provide unique identifiers for each container.
 - 6.13.6 Pre-printed labels with the assigned Lab Sample ID shall be affixed to the top two copies of the COC.
 - 6.13.7 Pre-printed labels with the Lab Sample ID will be printed from the LIMS system and placed on each sample bottle after log-in.



6.14 Documenting non-conforming samples and client communications.

6.14.1 According to NELAC sample receipt protocols 5.11.3 c) “Where there is any doubt as to the items suitability for testing, where the sample does not conform to the description provided, or where the test required is not fully specified, the laboratory shall consult the client for further instruction before proceeding. The laboratory shall establish whether the sample has received all necessary preparation or whether the client requires preparation to be undertaken or arranged by the laboratory. If the sample does not meet sample acceptance criteria the laboratory shall either:

6.14.1.1 Retain correspondence and/or records of conversations concerning the final disposition of rejected samples; or

6.14.1.2 Fully document any decision to proceed with the analysis of samples not meeting acceptance criteria. If the decision is to proceed, then the condition of these samples shall, at a minimum, be noted on the LCF and COC.”

6.14.2 Documentation of any non-conforming samples for:

6.14.2.1 Any non-conformity requires the completion of a Non-Conformity Form (NCF), as explained in AEL SOP Admin-016.

6.14.2.2 Unclear analysis.

6.14.2.3 Missing containers

6.14.2.4 Samples beyond the holding time

6.14.2.5 Inadequate sample volume

6.14.2.5.1 Includes air bubbles in VOCs vials (greater than 5%)

6.14.2.6 Damaged containers

6.14.2.7 Samples out of thermal preservation (0-6 degrees Celsius)

6.14.2.8 Improperly preserved samples

6.14.2.9 Inappropriate sample container

6.14.2.10 Incomplete or missing COC

6.14.2.10.1 For coliform samples, an AEL bacteriological reporting form is not a substitute for a COC, except when for drinking water total coliform. All other stand-alone bacteriologicals must have a COC.



6.14.2.10.2 If a COC is not present, enter the IDs and times collected into the LIMS from the bacteriological form. Record this information onto a COC and place the COC in the folder.

6.14.2.10.3 The reporting form shall be given to Micro in addition to the COC in these cases.

6.14.3 Note on the COC using both the field Ids, tests required, and nonconformities.

6.14.4 Submit the COC form to project management to notify client regarding the affected sample(s) and whether client wishes to proceed with the analysis with full understanding that the data from the affected samples will be qualified accordingly on the final report.

6.14.5 Any NCF that has been generated must accompany the project folder and be routed to the supervisors of the affected departments to ensure that all qualifiers are applied to the results correctly.

6.14.6 If the samples cannot be logged into the LIMS on a timely basis place them under refrigeration or on wet ice in coolers until they can be logged in.

7.0 Samples received after hours or over the weekend

7.1 Samples received after hours or on the weekends shall be checked as follows if no short hold time samples are present. The coolers are to be checked for any volatiles samples, and if present, they will be taken to the volatiles refrigerator if waters and volatiles freezer if soils. The coolers minus any volatiles shall then be taken to inside the walk-in refrigerator, where the lids will be propped open. The regular full time staff shall then proceed with the full login checklist during normal business hours.

7.2 If a short hold sample needs to be processed and analyzed after hours or over the weekend, those handling that sample and/or the analyst shall check the preservations, labels, and all items as listed on the checklist and make note of any discrepancies in the sample discrepancy logbook and leave notification for the regular login staff so that any issues can be addressed during regular business hours.

7.3 If a discrepancy is observed on samples received after hours or over the weekend which are of such a nature that the quality of the results may be affected if an issue is not resolved quickly, then a contact number for the lab manager and/or QA officer shall be available for the after hours personnel to contact on how to proceed.

7.4 For samples and projects that contain work being analyzed under DoD ELAP certification or for a DoD ELAP client, any after hours or weekend sample receiving shall be accepted and logged in by a member of the regular full time login staff only, or someone who has been fully trained to accept samples and is so designated as member of the login staff.



7.5 All after hours and weekend received samples and projects that contain work being analyzed under DoD ELAP certification or for a DoD ELAP client shall be processed the same as those sample received during regular business hours. All checks and login procedures shall be performed at the time of receipt.

8.0 Sample Login

- 8.1 Create a new project folder by taking a manila folder and affixing a sequential pre-numbered laser label. (Matching labels shall be affixed to the top two copies of the COC.) Place all paperwork associated with the project in the folder. Project Folder review and routing is discussed in the Quality Manual.
- 8.2 Use the Horizon software to log in samples. Follow that software instruction manual for doing so.
- 8.3 If the project is being logged in from AEL Gainesville the number will start with a "G", AEL Jacksonville "J", AEL Tampa "T", AEL Orlando (Altamonte Springs) "A", AEL Miami "M" Fort Myers "F", and AEL Tallahassee "S".
- 8.4 Every project logged in must have a client name, client contact and a project name. If there is no client project name listed, name it from the sample such as weekly, quarterly or effluent.
- 8.5 Do NOT use apostrophes for any data entry in Horizon.
- 8.6 If a project is a rush, then also place in a blue folder.
- 8.7 If a project needs to be edited by the project manager, then also place in a red folder (a purple folder for Jacksonville).
- 8.8 Affix the sample labels to the containers cross-checking samples and labels to ensure against mislabeling. Place the labeled sample containers on the sample cart and transport to the designated storage sections.
 - 8.8.1 A sample requiring a sub-sample for analysis is prepared in the following manner:
 - 8.8.1.1 The sub-sample can only be taken from a sample that meets all preservative criteria listed in this SOP and shall be checked for preservative prior to sub-sample aliquot.
 - 8.8.1.2 There must be adequate sample volume to meet method-required volumes per analytical parameter.
 - 8.8.1.3 The sample must be transferred to the proper container set forth by the "Kit Prep" standard operating procedure ADMIN-023.
 - 8.8.1.4 The project manager must be notified that the sample has been split.



8.8.1.5 Transfer of volume shall be performed as accurately as possible.

8.8.1.6 Necessary contamination preventatives must be taken.

8.8.1.7 Samples are to be labeled to reflect the test code, date taken, sample ID number, and project number.

8.8.1.8 Samples must remain under proper storage guidelines.

8.8.1.9 The sample is then treated as an original sample.

8.9 If the LIMS system fails due to virus or other errors for more than 4-5 hours then proceed with manual login with lab manager's approval. See AEL SOP Admin-019.

9.0 Samples Shipped/Received from other AEL Labs.

9.1 It may be convenient for the client to drop off samples at the AEL lab closest to the sampling location rather than at the lab that will analyze the samples. Also, some analyses are only performed at certain AEL labs, such as Low Level Mercury at the Jacksonville location or Surfactants at the Gainesville location. In those situations, the receiving lab will still login samples into Horizon under their AEL letter designation (see Section 8) but then set those tests or the entire project for shipping to the sister lab in the shipping queue in Horizon. Finally, in the event of balancing workload and to expedite testing, it may be appropriate to send samples to a sister lab for analysis on tests that the receiving lab does perform.

9.2 The initial receiving lab when receiving the samples, shall follow all the steps as listed in section 6 of this SOP, and accept custody of those samples on behalf of AEL.

9.3 Any anomalies and notifications/questions about the received samples, including the writing of NCFs for those anomalies, shall be handled by the receiving labs with e-mail correspondence sent to the sister lab that will be ultimately analyzing any samples not kept in house.

9.4 Once samples have been fully received and logged in (with shipping queue set for off-loading to the sister lab) those samples are stored refrigerated or frozen per method requirements until packed for shipment to the sister lab. Normally a separate area or shelf is set aside for sister-lab shipping as an easy visual indicator for samples to be shipped.

9.4.1 Metals samples can be stored without refrigeration; however acid preservation should be checked. If further acidification is required to reach a $\text{pH} \leq 2$, then that shall be done and documentation of that shall be listed in a logbook with the date/time and lot# of the acid used. That documentation shall accompany the samples to the sister lab.

9.4.2 For Volatiles samples: samples shall be stored away from any potential contaminant sources, including away from other "hot" samples.

9.4.3 For Volatile soil samples, if samples are to ship and be received by the sister lab with 48 hours of collection, then they can be refrigerated at 0-6°C prior to



shipping. However, if to be shipped and received after 48hours, then the samples are to be stored on their sides and frozen prior to shipping. Then those frozen samples must be shipped with sufficient dry ice to have them remain frozen at minus 10°C or lower.

9.4.4 All other samples shall be refrigerated until shipping at 0-6°C.

9.5 Towards the end of the day, samples that are to be shipped to sister labs are repacked on new ice (or on dry ice for frozen volatile soil samples) and an inter-lab chain is created listing all the samples to be shipped. The inter-lab chain is reviewed against what is contained in the coolers to ensure that what is in the coolers matches exactly what is on the inter-lab chain.

9.5.1 Samples with limited and short hold times must be marked on the outside of the coolers. Notification must be made to the sister lab that these samples are being routed to their location.

9.5.2 Samples requiring special handling or special conditions must be marked on the outside of the coolers. Notification must be made to the sister lab that these samples are being routed to their location.

9.5.3 All these conditions must be notified to the sister lab who will be receiving these sub-samples.

9.5.3.1 Sub-samples to a local sub-lab (also to be noted on the cooler)

9.5.3.2 Rush samples (also to be noted on the cooler)

9.5.3.3 Samples that are not drinking water, but are logged as drinking water (can use stickers on containers)

9.5.3.4 Samples that are potentially unusually hot (separate cooler)

9.5.3.5 Samples that require other than normal reporting

9.5.3.6 Incorrect preservation or container, limited volume

9.5.3.7 Headspace in a volatile sample

9.5.3.8 Any anomalous condition with samples witnessed

9.5.3.9 Samples to arrive on other than normal Blue Streak shipping days.

9.6 Coolers are labeled and set aside for pick up by Blue Streak courier for next morning delivery. Send along with the coolers a copy of the preservation checks made upon original receipt and a copy of the inter-lab chain.

9.7 Blue Streak shall deliver the coolers overnight to the sister lab. Arrival times can vary, but all should be delivered prior to business hours or no later than 9:00am the next morning. Plan accordingly for short holds.

9.8 The samples shall be unpacked and accepted by the sister lab using the following guidelines.

9.8.1 Check first those coolers marked “short hold” or “rush”

9.8.2 Put on a pair of latex disposable powder free gloves. Place the cooler in a well-ventilated area, open it, and determine if there is ice in the cooler. The



temperature measurement shall be taken using an IR gun measuring the surface temperature of the sample containers. Note temperature on the inter-lab chain of custody.

9.8.2.1 The target range is 0-6 degrees Celsius for samples that are thermally preserved.

9.8.2.2 Frozen volatile soil samples shall be received on dry ice at temperatures at or below minus 10°C.

9.8.2.3 Acid preservation shall not need to be checked, unless for some reason the original sister lab did not check pH of the samples.

9.9 While unpacking the coolers, the lab must verify that the samples received match those listed on the inter-lab chain of custody. Sign with time and date the inter-lab chain of custody. Any anomalies must be immediately addressed with the shipping lab.

9.10 Make a copy of the inter-lab chain of custody and save that record in the login department. Send back to the original receiving lab that original copy of the inter-lab chain using an inter-lab mailer envelope.

9.11 The lab must then mark those sent samples as received in Horizon so that the samples show properly as received and ready for analysis on the analysts' backlogs.

10.0 Sample Storage

10.1 Jacksonville

10.1.1 VOC samples

10.1.1.1 All samples shall be placed in the refrigerator in volatiles. There are designated soil and water refrigerators.

10.1.1.1.1 Waste VOC samples are placed in the same refrigerator as soils.

10.1.1.1.2 If samples are "hot", as in of a concentration that could possibly cross contaminate to other samples, those samples are to be segregated. For volatiles that could consist of placing them in a sealed Coleman type cooler and placing them in the walk-in-cooler or having a separate refrigerator just for their storage. Upon expansion of the lab in 2019, a separate refrigerator was purchased solely for their storage.

10.1.2 Micro samples

10.1.2.1 Shall be placed in the refrigerator in micro. Samples shall be separated as to whether they are for fecal coliform, total coliform, or other micro tests.



10.1.2.2 Due to short holding times, notify the micro analyst of the collection time with every sample delivered to microbiology.

10.1.3 Metals

10.1.3.1 Place soils in the walk-in cooler. Also place unpreserved waste samples or water samples when also to be used in other departments in the walk-in.

10.1.3.2 Place metals-only water samples on the storage shelves in the metals department.

10.1.4 Wet Chemistry

10.1.4.1 Deliver the samples to the inorganic side of the walk-in cooler.

10.1.4.2 Due to short holding times, notify the wetchem analyst if a delivered sample is nearing the holding time. CBOD/BOD, TSS, and IC samples are often delivered to the wetchem analyst assigned as primary to those methods. CBOD/BOD and TSS samples have their own refrigeration units assigned in the wetchem laboratory.

10.1.5 Extractable Organics

10.1.5.1 Deliver the samples to the organic side of the walk-in cooler. Seal in a Coleman style cooler any samples capable of cross contamination to other samples.

10.2 Tampa

10.2.1 Micro samples

10.2.1.1 Shall be placed in the refrigerator in micro. Samples shall be separated as to whether they are for fecal coliform, total coliform, or other micro tests.

10.2.1.2 Due to short holding times, notify the micro analyst of the collection time with every sample delivered to microbiology.

10.2.2 Wet Chemistry

10.2.2.1 Deliver the samples to the inorganic side of the walk-in cooler.

10.2.2.2 Due to short holding times, notify the wetchem analyst if a delivered sample is nearing the holding time.

10.2.3 VOC samples

10.2.3.1 All samples shall be placed in the refrigerator in volatiles. There are designated soil and water refrigerators.



10.2.3.2 Waste VOC samples are placed in the same refrigerator as soils.

10.2.4 Extractable Organics

8.2.4.1 Deliver the samples to the organic side of the walk-in cooler

10.2.5 Metals

10.2.5.1 Place soils in the walk-in cooler.

Place water samples on the storage shelves in the metals department.

10.3 Orlando (micro and wet chemistry analysis only)

10.3.1 Micro samples

10.3.1.1 Shall be placed in the refrigerator in micro. Samples shall be separated as to whether they are for fecal coliform, total coliform, or other micro tests.

10.3.1.2 Due to short holding times, notify the micro analyst of the collection time with every sample delivered to microbiology.

10.3.2 Wet Chemistry

10.3.2.1 Deliver the samples to the storage refrigerator.

10.3.2.2 Due to short holding times, notify the wetchem analyst if a delivered sample is nearing the holding time.

10.4 Gainesville

10.4.1 Micro samples

10.4.1.1 Shall be placed in the refrigerator in micro. Samples shall be separated as to whether they are for fecal coliform, total coliform, or other micro tests.

10.4.1.2 Due to short holding times, notify the micro analyst of the collection time with every sample delivered to microbiology.

10.4.2 Wet Chemistry

10.4.2.1 Deliver the samples to the walk-in storage refrigerator.

10.4.2.2 Due to short holding times, notify the wetchem analyst if a delivered sample is nearing the holding time.



10.5 Miami

10.5.1 VOC samples

10.5.1.1 All samples shall be placed in the refrigerator in volatiles. There are designated soil and water refrigerators.

10.5.1.1.1 Waste VOC samples are placed in the same refrigerator as soils.

10.5.2 Micro samples

10.5.2.1 Shall be placed in the refrigerator in micro. Samples shall be separated as to whether they are for fecal coliform, total coliform, or other micro tests.

10.5.2.2 Due to short holding times, notify the micro analyst of the collection time with every sample delivered to microbiology.

10.5.3 Metals

10.5.3.1 Place soils in the designated refrigerators. Also place unpreserved waste samples or water samples when also to be used in other departments in the walk-in.

10.5.3.2 Place water samples on the storage shelves in the metals department.

10.5.4 Wet Chemistry

10.5.4.1 Deliver the samples the designated refrigerators.

10.5.4.2 Due to short holding times, notify the wetchem analyst if a delivered sample is nearing the holding time.

10.5.5 Extractable Organics

10.5.5.1 Deliver the samples to the designated refrigerators.

10.6 Tallahassee

10.6.1 All samples are to be placed in the refrigerator or stored in iced coolers for later shipment.

10.7 Fort Myers

10.7.1 All samples are to be placed in the refrigerator or stored in iced coolers for later shipment.



10.8 If the samples are to be sent to another laboratory in the AEL network, see Section 9 for procedures to follow.

10.9 If the samples are sent to an approved subcontracted laboratory, the samples will be shipped using a third-party shipping agency and will become the responsibility of the subcontracted laboratory to ensure the samples are correctly stored and preserved.

11.0 Pollution Prevention

11.1 See Standard Methods, Section 1100 (2010) Waste Minimization and Disposal.

11.2 See the AEL SOP for Hazardous Waste Management (ADMIN-018) and the AEL Safety Manual.

12.0 References

- 12.1 AEL SOP Admin-005a, Manual Log-in.
- 12.2 AEL SOP Admin-019, Manual Log-in.
- 12.3 AEL SOP Admin-023, Sample Kit Preparation.
- 12.4 AEL SOP Admin-016, Non-Conformities.
- 12.5 AEL SOP Admin-018, Waste Disposal.
- 12.6 AEL SOP Admin-031, Receipt of Consumable Items.
- 12.7 AEL Quality Manual, newest revision.
- 12.8 AEL Safety & Health Manual, newest revision.
- 12.9 Standard Methods, 1090 (2010), 1100 (2010)
- 12.10 TNI 2016 Standards.
- 12.11 DoD ELAP QSM rev. 5.4, January 2021.
- 12.12 ISO 17025: 2005 & 2017 Standards
- 12.13 Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples -- EPA/600/R-03/027 November 2003 by Robert W. Gerlach Lockheed Martin Environmental Services.
- 12.14 Florida Department of Environmental Protection Standard Operating Procedures for Field Activities (eff 04/16/2018)



Table 1

Log-in Checklist Items.

1. Sample temperature checked and recorded.
2. Chain of Custody received and properly filled out.
3. Chain of Custody signed when relinquished and received.
4. If received with custody seals, seals were intact.
5. All samples arrived in good condition. (All unbroken)
6. Sample labels agree with sample Chain of Custody.
7. Bottle labels contain all necessary information.
8. Sample container labels filled out using indelible ink or Sharpie.
9. The correct containers were used for the test indicated.
10. The proper preservative listed on the sample container.
11. Samples were received within holding times.
12. All VOA vials checked for the presence of air bubbles.
13. Samples were in direct contact with wet ice.
14. Sufficient sample volume to perform tests requested.
15. Sample cooler was less than 6 degrees centigrade.
16. Sample pHs checked and recorded by sample log-in when applicable.
17. When non-conventional sample container used, noted on Chain.
18. Samples accepted into the laboratory.

If any of the above items from the checklist are not met, it is to be noted in the "Sample Acceptance Discrepancies Log".

Also use the "Sample Acceptance Discrepancies Log" to note any other anomalous events or issues arising in sample log-in.



Advanced
Environmental Laboratories, Inc.

SOP No.: Admin-018
Revision No. 10
Effective Date: 10/09/2020
Revised By: K. Bortle
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STANDARD OPERATING PROCEDURE

For

Waste Disposal and Pollution Prevention





SOP Revision Log for AEL SOP Admin 018

| Revision Number | Revision Date | Reason for Revision | Section(s) and Page(s) Revised |
|-----------------|---------------|---|--------------------------------|
| Revision 00 | 12/01/01 | Initial Creation | All sections affected |
| Revision 01 | 02/20/03 | Complete revision, change language throughout | All sections affected |
| Revision 02 | 04/30/04 | Add cover sheet, revised to add reference to JEA compliance, add F tables from CFR. | Sections 4 and 5 |
| Revision 03 | 09/25/07 | Revised to expand references to JEA compliance for Jacksonville Laboratory sections 2, 6. Add Appendix 1 JEA permit (as required by JEA audit) | Sections 2, 6, Appendix 1 |
| Revision 04 | 04/02/10 | Add revision log, remove CFR tables. Complete re-write throughout | All sections affected |
| Revision 05 | 05/26/16 | Update format. Minor language changes. Remove copy of JEA permit, reference it only. | All sections affected |
| Revision 06 | 06/28/19 | Update references. Minor language changes. Edit to allow for combining hazardous water and solvents with corrosive liquids into a single drum. Remove weekly inspection documentation requirement. | All sections affected |
| Revision 07 | 07/15/20 | Water containers glass and plastic emptied. Vials and soil jars fully destroyed by mechanical crusher. Infectious waste to be handle per SM9020 biosafety level 1. Add language throughout. Define period of time samples must be held prior to disposal. | All sections affected |
| Revision 08 | 07/31/20 | Further define waste streams and testing of waste streams. Increase TCLP testing. Update training materials. Update documentation requirements. Add PPE waste category. Add emergency procedures to training outline. | Sections 2, 3, 4, Appendix 2 |
| Revision 09 | 09/22/20 | Complete revision. Change language throughout. Add new sections. All sections affected. | All sections affected |
| Revision 10 | 10/09/20 | Change language throughout. All sections affected. Add P and U waste code tables. | All sections affected |
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Waste Disposal and Pollution Prevention

1.0 Scope and Application

1.1 Advanced Environmental Laboratories Inc (AEL) has seven laboratories. Four are defined as very small quantity generators (VSQG) of hazardous waste, while the three full service laboratories are small-quantity generators (SQG) under EPA definitions. AEL is committed to the responsible handling, treatment, and disposal of any hazardous or potentially hazardous materials associated with our laboratory activities. The firm makes every effort to minimize the amounts of hazardous materials necessitating disposal, and to treat, label, and dispose of such wastes in a manner which is within the regulatory guidelines and which minimizes the negative impact on the environment. Whenever possible, Advanced Environmental Laboratories Inc. will dispose of waste materials by recycling as opposed to disposal.

1.2 Wastes requiring disposal generally fall into one of five categories:

- 1) Samples received for analysis.
- 2) Sample extracts, distillates, and digestates.
- 3) Spent solvents and expired reagents and standards.
- 4) Waste generated by lab processes
- 5) Field generated wastes

1.3 This standard operating procedure (SOP) describes the procedures used by Advanced Environmental Laboratories Inc. Jacksonville (AEL, JAX) and by all AEL laboratories to dispose of all hazardous and non-hazardous sample waste. Administration of this SOP is assigned to the Hazardous Waste Coordinator at the Jacksonville facility. The Hazardous Waste Coordinator at each lab signs all manifests and routinely informs all disposal operations to the owner and operations manager.

1.4 Contractors selected by the Jax Lab (and all AEL facilities) for treatment, transportation, and disposal are strictly limited to those who meet all applicable legal, administrative, and financial criteria. Only approved organizations are used for the purpose.

1.5 Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Managers are responsible for ensuring adherence to the SOP by the laboratory staff and providing adequate explanation of the material contained.

1.6 Annual training is required in the month of January. Annual training is to include as part of the Chemical Hygiene Training, a section pertaining to the proper handling of lab generated waste. Training shall also occur anytime there



are major revisions to this SOP. New staff upon hire, shall be given full training. All training shall include the reading and discussion of this SOP.

- 1.7 Only the three full service labs of AEL (Jacksonville, Miami, and Tampa) are classified as small quantity generators (SQG) of waste. The Tallahassee, Gainesville, Orlando, and Fort Myers labs are classified as very small quantity generators (VSQG) of waste. That is, they generate < 100Kg of waste per month. Those VSQG labs still must follow the procedures as listed in this SOP for waste collection and documentation, however, that collected waste can be brought to their local city's or county's household hazardous waste facility or next collection event. There may be a small fee associated with this.

2.0 Defining Hazardous Waste and Waste Characteristics.

- 2.1 A material only becomes a waste after it is no longer being used in a process, and needs to be recycled, thrown away, or stored until enough volume is collected to treat or dispose of.
- 2.2 Once the material (used and excess sample, spent solvent, used reagents, etc.) is deemed to be a waste, an accurate determination is required as to whether that waste is a hazardous waste in order to ensure it is properly managed according to applicable Resource Conservation and Recovery Act (RCRA) regulations. A hazardous waste determination is made using the following steps:
 - 2.2.1 The hazardous waste determination for each waste product must be made at the point of waste generation, before any dilution, mixing, or other alteration of the waste occurs.
 - 2.2.2 The person making the determination must use knowledge of the waste to determine whether the waste is to be deemed hazardous. The person must apply knowledge of the hazard characteristic of the waste in light of the materials or the processes used to generate the waste. In the lab, the most common knowledge is that gained from testing samples or the direct knowledge of the reagents and solvents used in the testing process.

Characteristics of Hazardous Waste: Ignitable, Corrosive, Reactive, Toxic

2.3 Ignitable

- 2.3.1 If liquid with a flashpoint of <140°F, it is considered ignitable. If a solid is capable of ignition through friction or by chemical change (such as spontaneous combustion), it is considered ignitable. The most common source of an ignitable waste in the lab would be spent solvents used in the lab, such as used Hexane and Acetone.



These would first be collected into a satellite container labeled “Hazardous Waste” “Ignitable” with a “D001” hazardous waste number (can also be referred to as EPA hazardous waste code). Note that satellite containers can have more than one classification. Example: If also containing toxic substances, the satellite should also be labeled “Toxic” with the hazardous waste numbers of those substances.

2.4 Corrosive

- 2.4.1 If the waste is aqueous with a $\text{pH} \leq 2$ or ≥ 12.5 as determined by pH meter under method 9040C, it is corrosive. Corrosivity can also be determined by test Method 1110A (steel corrosive test). If waste is deemed to be corrosive, it would be collected first into a satellite container labeled “Hazardous Waste” “Corrosive” with a “D002” hazardous waste number. (Note: again, satellite containers can have more than one classification.)

2.5 Reactive

- 2.5.1 A waste would be classified reactive if it undergoes violent change without detonating, reacts violently with water, forms potentially explosive mixtures with water, when mixed with water generates toxic gases, is a cyanide or sulfide bearing waste that would produce toxic gas if mixed with a corrosive, or can explode. If waste is deemed to be reactive, it would be collected first into a satellite container labeled “Hazardous Waste” “Reactive” with a “D003” hazardous waste number. (Note: again, satellite containers can have more than one classification.)

2.6 Toxic

- 2.6.1 Toxicity characteristics are covered under the test method for TCLP 1311. The waste does not have to be tested by TCLP to reach a determination that it is Toxic in character. Other knowledge can be used, such as regular sample testing or bottle labeling.
- 2.6.2 Samples that have tested above the TCLP limits for any compounds on the TCLP list (see Appendix 1, Table 1) shall be segregated to a hazardous waste satellite container labeled as “Hazardous Waste” “Toxic” along with the exceeded compound(s) hazardous waste number. (example “D006” for cadmium)



2.7 Hazardous Waste from non-specific sources.

- 2.7.1 If the waste is determined to be hazardous, the lab must identify all applicable EPA hazardous waste numbers (EPA hazardous waste codes) for that waste. Prior to shipping the waste off site, the generator must mark its containers with all applicable EPA hazardous waste numbers (EPA hazardous waste codes) and waste characteristics.
- 2.7.2 Therefore, further classification of hazardous waste may be required when segregating a waste to a satellite container. If the waste contains a substance from the F001-F005 Hazardous Waste Table (See Appendix 1, Table 2) at or above 10% by volume, then that waste is also to be segregated into a satellite container and labeled with its hazardous waste number. That satellite container shall also be labeled as “Hazardous Waste”, then “Toxic” and/or “Ignitable” based on the waste properties.
- 2.7.2.1 Note that any non-hazardous samples/wastes that come into contact with any F-listed wastes are then considered themselves to be an F-listed waste regardless of the quantity of the F-listed waste involved (i.e., mixture rule).
- 2.7.3 If a waste is seen that does not seem to fit into any of the categories seen above, then seek out the hazardous waste coordinator.

3.0 Waste Accumulation

- 3.1 **Non-hazardous waters.** For the labs, these would be any drinking water vials or samples, wastewater treatment plant samples, and other waste waters not otherwise identified in the lab as hazardous. If a sample tests with results that indicate that it is hazardous, that sample shall be segregated and collected into a satellite container. (Note: also notify your supervisor as no such hazardous samples are expected to be associated with drinking water or wastewater samples). These non-hazardous water samples can be disposed of down the sink if pH neutral or neutralized to a pH range of 5.5 to 12.0. If unsure of the characteristic of the aqueous waste, set it aside to be classified. Always err on the side of caution.
- 3.1.1 For the three full service laboratories, the local utility company must be informed of the type and quantity of these discharges. The utility may elect to set up a compositor to test the lab’s non-hazardous wastewater stream and/or issue specific instructions for



wastewater disposal. The lab must then follow those instructions if/when issued.

- 3.1.2 Non-aqueous samples or any aqueous samples **not meeting the criteria as defined in 3.1 above** shall be disposed of with the hazardous waters, solvents, and liquid wastes (see section 3.7). This would include waste that do not fail TCLP but could potentially contain other contaminants. Paint waste and oils would be disposed of under section 3.7 criteria.

- 3.2 **Infectious waste.** These wastes are those generated in the Microbiology Department. Whenever samples have colony growth or an organism is cultured as controls or comparison samples, after use and analysis, these must be sterilized by autoclaving before disposal. Autoclave waste is not considered hazardous waste and can be disposed of in the normal trash pickup.

Some waste generated from the microbiology department is designated as infectious waste. The lab is to follow procedure as listed in Standard Methods 9020B under Section 2.0, Biosafety Criteria. In short, there are two main pathways for disposal. Any excess sample or unused volumes can be disposed of in the manner they were collected, as in wastewater can be disposed of down the toilet. If the waste comes from any positive growth of any kind, then the waste must be autoclaved using an autoclave bio-bag before disposal with the normal trash pickup.

- 3.3 **Satellite containers** are for the collection of any waste that is deemed to be hazardous by either lab testing or by product container labeling. (See section 2.0 above). Satellite containers can be any volume from 4 ounces up to 55 gallons. If 55 gallons or more of waste is collected into a satellite container, it must receive a start date, be re-designated a waste accumulation container, be moved to the designated hazardous waste accumulation area, and be removed for disposal within 180 days of the start date.

- 3.3.1 If a container holding hazardous waste is not in good condition, or if it begins to leak, the lab must immediately transfer the hazardous waste from this container to a container that is in good condition and does not leak, or immediately transfer and manage the waste in a central accumulation area. Good condition means that there are no severe dents or rust, especially in the seam areas, which could weaken the container and allow it to leak. The container must close and seal properly.
- 3.3.2 The generator must use a container made of or lined with materials that will not react with, and are otherwise compatible with, the



hazardous waste to be accumulated, so that the ability of the container to contain the waste is not impaired.

- 3.3.3 Incompatible wastes, or incompatible wastes and materials must not be placed in the same container.
- 3.3.4 A container holding a hazardous waste that is incompatible with any waste or other materials accumulated nearby in other containers must be separated from the other materials or protected from them by any practical means.
- 3.3.5 A container holding hazardous waste must be closed at all times during accumulation, except:
 - 3.3.5.1 When adding, removing, or consolidating waste
 - 3.3.5.2 When temporary venting of a container is necessary
 - 3.3.5.3 For the proper operation of equipment
 - 3.3.5.4 To prevent dangerous situations, such as build-up of extreme pressure.
- 3.3.6 The lab must mark or label its container with the words “Hazardous Waste” and the applicable hazardous waste characteristic(s) (*i.e.*, ignitable, corrosive, reactive, toxic).
- 3.3.7 The labeling for satellite containers should also include the hazardous waste numbers of the compounds they contain. See Appendix 1, tables 1 & 2. (The D and F waste codes)
- 3.3.8 All satellite accumulation areas operated must have available equipment to contain a release of the hazardous waste and to minimize the likely hood of fire or explosion. Spill kit(s) are needed adequate for the wastes accumulated. Access to nearby phone and fire extinguisher are required in the vicinity of the satellite accumulation areas. Decontamination equipment such as an eyewash station must also be nearby.
- 3.3.9 Waste for each batch of samples are first segregated by the known chemical(s) used in processing the samples. As example, a waste generated from extracting with methylene chloride is first collected in a satellite container labeled “Hazardous waste” “Toxic” “F002”. The waste for that batch (or that day) is set aside until analysis has been completed for the samples in that batch (or day). Those batch-day satellite containers shall be temporarily labeled with the batch ID or date. Once final analysis has returned results that show no other hazards are present, as happens in most cases, then that



satellite container (traceable to the sample processing by batch/date on the processing logs) is emptied into the main larger satellite container labeled with the known chemical codes used in the sample processing. In the cases where analysis does discover a new hazardous characteristic(s) on a sample(s), the sample(s) hazardous characteristic(s) are communicated to the lab by e-mail. Those processed waste are then segregated into their own satellite container with the hazardous labeling of the known hazards and newly discovered hazards. The original batch containers are now empty (wiped clean with cleaning materials disposed of with waste stream) and ready for re-use. In this way the generated processing waste and any discovered sample hazards can be known across the different lab departments.

3.3.10 Satellite accumulation areas may be consolidated so that processed samples and the waste generated from processing can be accumulated into common satellite containers. As example, the Jacksonville lab will have the satellite accumulation areas in the metals and extraction departments with wetchem waste accumulated in metals and TCLP/SPLP waste accumulated in extractions.

3.3.11 The unused portions of the original samples (normally enough extra volume is collected for re-analysis if needed) are then disposed of based on the final analytical results. This extra volume can be held up to 30 days after analysis (more if contracted by client). Unused sample portions will be disposed of by sample ID and date after reviewing the final project result reports so it is known what samples go to what hazard satellite container and that the labeling reflects the hazards properly.

For Sections 3.4 thru 3.10: All other waste whether hazardous or non-hazardous will be collected and drummed up for disposal.



3.4 **Non-hazardous soils.** The soil from vials and sample jars are dumped into a 55-gallon steel drum labeled for disposal as a "non-hazardous soil" drum. In most cases this entails putting the vials and jars through a mechanical crusher temporarily attached to the top of the disposal drum. (Ear protection and other PPE required) This has the dual benefit of compacting the waste and destroying the labeling. **Only soils that are non-hazardous can go into this drum.** The waste drum should be marked with a green non-hazardous label instead of the yellow hazardous labels. Once annually, as a check of our procedures, a non-hazardous soil drum shall be cored and analyzed for a full TCLP. Save the TCLP test results for as supporting documentation for this waste stream's waste profile.



- 3.5 **Hazardous soils and solids.** Any hazardous soils or solids shall be segregated from the general sample storage areas when analysis determines that they are hazardous. As example, samples that have tested above the TCLP limits for any compounds on the TCLP list (see Appendix 1, Table 1) shall be segregated to a hazardous waste satellite container labeled as “Toxic” along with the exceeded compound(s) hazardous waste number. (example D006 for cadmium). (See section 2 for a full listing of hazardous waste classifications) If only a small quantity of a single hazardous substance is expected over an extended time period, then a small satellite container can be used for accumulating this waste. Jars, pails, buckets, or drums can be used as satellite containers as long as they comply with section 3.3 requirements.



As needed, such as when a container is filled, satellite containers shall be moved to the hazardous waste accumulation area and transferred into a hazardous waste drum. All the hazardous waste numbers and waste characteristics of the satellite container(s) shall be transferred to the label of the collecting hazardous waste drum. Multiple satellites will most likely result in listing multiple hazardous waste numbers and possibly multiple waste characteristics on the drum label. The start date of accumulation will be listed on the label and then that drum must be transported for disposal within 180 days of that start date.

- 3.6 **Solvent vials and waste containers.** Any container than is 5 gallons or less can be Lab Packed in a 55-gallon drum for disposal. All the autosampler vials from the instruments, even if they have gone dry, are disposed of in this manner. Old expired chemicals and standards are also placed unbroken into the drum. This drum will be labeled "Hazardous Waste" “Ignitable” “Toxic” and “Corrosive” dependent on all that is added to this drum. The hazards label will also list all the hazardous waste codes of the contents of the drum. If any container to be lab packed contains greater than 10% by volume of used solvent, use the appropriate F001-F005 hazardous code from Appendix 2, table 2. As with hazardous soils and solids, transfer the hazardous waste numbers from any satellite containers that are emptied into the drum.

- 3.6.1 Note that any non-hazardous samples/wastes that come into contact with any F-listed wastes are then considered themselves to be an F-listed waste regardless of the quantity of the F-listed waste involved (i.e., mixture rule).
- 3.6.2 Any expired, off-spec, or unused commercial chemical products (such as any unused expired acetone, chloroform, methanol, etc.) that require disposal are to be compared to the chemicals listed in the CFR 40 261.33 (and listed at the end of this SOP) to determine whether a P-list or U-list waste code would be applicable to labeled



on this waste drum. Use the D codes and F codes for used chemical product waste. As example an old bottle of chloroform unopened but expired, would be disposed of listing waste code U044 on the hazardous waste label. Used chloroform and waste associated with its use would have waste code D022 and would be disposed of in the hazardous waters, solvents, and liquid wastes drum.

3.7 Hazardous waters, solvents, and liquid wastes. Any samples that contain hazardous water or solvent shall be segregated from the general sample storage areas when analysis determines that they are hazardous. As example, samples that have tested above the TCLP limits for any compounds on the TCLP list (see Appendix 1, Table 1) shall be segregated to a hazardous waste satellite container labeled as “Toxic” along with the exceeded compound(s) hazardous waste number. (example D006 for cadmium) at the time of disposal. Note that samples can be held for up to 30 days pending client review and possible request for further testing. (See section 2 for a full listing of hazardous waste classifications) If only a small quantity of a single hazardous substance is expected over an extended time period, then a small satellite container should be used for accumulating this waste. Jars, pails, buckets, or drums can be used as satellite containers as long as they comply with section 3.3 requirements.

- 3.7.1 When needed, a hazardous water and liquid solvent drum will be started in the hazardous waste accumulation area and the contents of the satellite containers will go into this drum. This drum will be labeled "Hazardous Waste" "Ignitable" "Toxic" and "Corrosive" dependent on all that is added to this drum. The hazardous label will also list all the hazardous waste codes of the contents of the drum. If any original source waste (determination made at the point of waste generation) contained greater than 10% by volume of solvent, use the appropriate F001-F005 hazardous code from Appendix 2, table 2. Transfer the hazardous waste numbers (and waste characteristics) from any satellite containers that are emptied into the drum.
- 3.7.2 Non-aqueous samples or any aqueous samples not strictly defined as non-hazardous water (see section 3.1) shall be disposed of with the hazardous waters, solvents, and liquid. This would include waste that do not fail TCLP but could potentially contain other contaminants. Paint waste and oils would be disposed of here.
- 3.7.3 Note that any non-hazardous samples/wastes that come into contact with any F-listed wastes are then considered themselves to be an F-listed waste regardless of the quantity of the F-listed waste involved (i.e., mixture rule).



3.8 Corrosive Liquids/Acidic Liquids. Acids should go into this accumulation drum. It should be noted that acids are reactive, so take extreme care when disposing in this drum. As with any disposal into a drum, full PPE must be worn. Dump liquid waste at a slow pour into the waste drum to prevent splashing. Also watch for reaction and spillover. (Note all waste areas must have spill containment). No one should be alone when disposing. All HAA vial extractions would be in this drum. This drum will be labeled as “Hazardous Waste” “Corrosive” at a minimum. Transfer the hazardous waste numbers (and waste characteristics) from any satellite containers that are emptied into this drum.

Hazardous waters and Solvents can be combined with Corrosive Liquids/Acidic Liquids to one drum if there is not enough volume of each to justify separate drums. Label appropriately with all the hazardous waste numbers and waste characteristics that apply.

3.9 Mercury Waste. A separate waste stream is designated for Mercury as it is the costliest to dispose of. Any satellite containers and accumulation containers will be labeled “Hazardous Waste” “D009-Mercury” “Toxic” “Corrosive” at a minimum. If any other metals are detected at hazardous waste levels as seen in Appendix 1, Table 1, then those hazardous waste numbers shall also be required to be listed on those satellite and accumulation containers as well. For the final accumulation container, the smallest container that can be used and still contain all the mercury waste accumulated in that 180 day period should be used to save on cost of disposal. Please note that the cost of using or contaminating a 55-gallon drum well exceeds \$1000. Costs go down incrementally in relation to the container size.

3.10 PPE, wipes, and collected waste. Gloves, paper towels, wipes, wooden stirrers, and any materials that come in contact with hazardous samples are to be treated as if it is a sample of that hazardous material. Those materials should be collected into satellite containers in the same manner as the hazardous solids, with the hazardous waste numbers and waste characteristics listed on the satellite containers. If unsure if those samples that came in contact with the working materials are actually hazardous, those working materials can be held for disposal until sample results are returned. PPE used in conjunction with extraction solvents will be treated as being contaminated with those solvents and contained accordingly.

4.0 Drum Storage in the hazardous waste accumulation area.

4.1 Once hazardous waste is removed from the satellite containers to accumulation containers, the clock starts ticking and these containers (drums) must be disposed of within 180 days.





- 4.2 Drums for each category of waste shall be used. This is due to the cost of disposals of each category of waste. Pouring Mercury waste into a drum designated for hazardous liquids and solvents will then push that drum to a more expensive category for disposal and the label would need to be changed on the drum to reflect its now more toxic characteristic.
- 4.3 Drums are stored in a designated area away from the lab, such as an outside storage shed or separately ventilated room. The storage area must have adequate ventilation to prevent the accumulation of fumes. The spaces between the drums and the aisle space must be adequate for inspecting fully all around the drums and to allow the unobstructed movement of personnel. Spill containment must be of adequate design to be able to contain all the volume of a leaking drum. Most labs are to use PIG poly spill containment pallets (or equivalent) under the drums. A spill kit(s) should also be available in the hazardous waste accumulation area. Spillage inside and outside the PIG containment pallets shall be cleaned up and that waste disposed of appropriately under this SOP procedures.
- 4.4 The emergency contact phone numbers and information as listed in section 6.2 must be posted in the hazardous waste accumulation area.
- 4.5 The hazardous waste accumulation area shall also have a fire extinguisher and decontamination equipment such as an eyewash.
- 4.6 Drums should be chosen to the size estimated for accumulation of six months worth of waste. A 55-gallon drum should not be used if it would only be 1/4 or 1/2 full in that time period. 15 gallon and 30-gallon waste containers are available choices.
 - 4.6.1 Drums must be in good condition. Good condition means that there are no severe dents or rust, especially in the seam areas, which could weaken the container and allow it to leak. The container must close and seal properly.
- 4.7 Drum construction should be based on what it will contain. A steel drum would be appropriate for non-hazardous and hazardous soils and solids but not appropriate for corrosive waste. Seek advice of the licensed company which will provide permitted transportation and treatment/disposal of the waste. They will also most likely provide the waste containers to the lab.
- 4.8 Specific rules for sample storage and labeling are covered in the Hazardous in various regulating documents including the Federal Code of Regulations 40-261,262. The following specific points are to be followed:



- 4.8.1 Containers and drums must be kept in good condition, must be compatible with contents, and must be kept closed.
- 4.8.2 The date of first accumulation of waste in the drum must be marked on the drum. Once the start date has been assigned (when waste is first introduced into the waste container) the waste drum **must be disposed of within 180 days**.
- 4.8.3 Each drum must be properly labeled. The rules for labeling are listed in sections 2 and 3 above. The rules for containment also follow those listed for satellite containers as listed in section 3.3.
- 4.8.4 The laboratory's proper EPA Identification Number must be plainly marked on each drum.
- 4.9 When disposing of samples using the vial crusher ear protection and other PPE are required. The use of crusher has the dual benefit of compacting the waste while also destroying the labeling.
- 4.10 The waste area shall be kept clean. Waste buckets and waste shall not be left lying around loose. The area shall be maintained in a clean manner with all drums and waste containers labeled. Drums will be maintained clean on the outside surfaces, otherwise waste disposal companies will not remove them when scheduled, delaying their removal.
- 4.11 Normal waste disposal is roughly weekly or bi-weekly. If there are any damages or problems seen during the normal disposal or at any other time, they are handled immediately, and the hazardous waste coordinator is to be notified.
- 5.0 **Documentation.** For items 5.1 through 5.6 listed below, proper documentation is required. For those samples collected for shipping and disposal, the labels on the drums must reflect the waste material profiles set by our waste transportation and disposal company. The final profile sheet (and any testing done to properly fill out the profile sheet) shall represent the full documentation for those drums. Waste Manifests are completed every time shipping and storage companies pick up drums. These forms are retained for 5 years.

Documentation for waste streams should be clear and easy to follow. The lab should be able to clearly connect the dots from sample-satellite-drum-profile-transporter.

- 5.1 A sample inventory and copies of final project result reports shall be kept for samples with hazardous characteristics and be traceable to the drum where they are accumulated. The hazardous waste label shall list all the waste codes and characteristics for those samples and sample processing wastes associated with those project reports. The drum profile shall be made with the transporter



accordingly. All documentation shall be readily available for any internal or external assessment reviews for 5 years.

- 5.2 Inspect containers at least once a week and keep a written log of container inspections.
- 5.3 Keep a record of larger spills and use this information to identify the spill prevention options that might help your lab.
- 5.4 Keep training and inspection records for five years.
- 5.5 Keep manifests and shipping receipts for five years.
- 5.6 AEL's default policy is to only hold samples for 30 days past when the final report is released. This is when no other instructions are given by the client. Some projects and those under DoD may/will define a longer period and the disposal team must be aware of what that period is before disposal. Mark or segregate those samples that must be retained for a longer period of time. The lab must follow client request and any contract guidelines where it concerns sample disposal (as long as those requests do not conflict with EPA regulations and these SOP procedures). These held samples shall be disposed in the appropriate waste stream based on the hazards listed on final project result reports.

6.0 Emergency Procedures.

- 6.1 The lab must have a designated emergency coordinator who knows what to do in case of a fire, spill, or other emergency. They must be on the premises or on call 24 hours a day. The designated coordinator responsibilities can be transferred from one person to another so as to cover an absence or vacation and not leave the lab without an emergency contact.
- 6.2 Emergency contact phone numbers and information are posted in every room.
 - 6.2.1 Fire department phone number.
 - 6.2.2 Emergency coordinator's name and phone number.
 - 6.2.3 Map with the locations of fire alarms and extinguishers.
 - 6.2.4 Locations of spill control material

6.3 Spill Response and Clean-up Procedures

- 6.3.1 In the event of a chemical or waste spill, the individual(s) who caused the spill is responsible for prompt and proper clean-up. It is also their responsibility to have readily available the spill control



and personal protective equipment appropriate for the chemicals being handled.

6.3.2 AEL's spill response assumes that spill control materials are close to the areas where wastes and hazardous chemicals are stored. It also assumes that Health and Safety training is given to all new employees and refreshed on an annual basis. The person working with the waste or solvents should have a working knowledge of the possible hazards of the spill material and how to use the spill kit/materials prior beginning work in that area.

6.3.3 The spill response should be as follows:

6.3.3.1 Determine the severity of the spill and react proportionately.

6.3.3.2 Ascertain the risk to yourself and those around you.

6.3.3.3 Evacuate to an area free of vapors if vapors are suspected. Inform the supervisor, lab manager, and emergency coordinator.

6.3.3.4 A member of the managerial staff will determine if a complete building evacuation is required.

6.3.3.5 If it is an obvious dangerous situation, the person first noting the spill should ask in a loud calm voice for all to leave the area while at the same time sending someone to inform a member of the managerial staff.

6.3.3.6 If not already wearing appropriate PPE to work with the waste, don the PPE prior to first containing and then cleaning up the spill. (see evacuation plans below if a severe spill)

6.3.3.7 If a small spill, use the spill control materials located nearby to contain and then clean up the spill.

6.3.3.8 Dispose of the spill control materials and cleanup materials appropriately including any PPE worn.

6.3.3.9 Decontaminate the area following the cleanup. An internal incident report will need to be documented by the emergency coordinator.

6.3.3.10 Take any corrective action stated in the report to prevent a future re-occurrence of the spill.

6.3.4 For Evacuation.

6.3.4.1 Each lab shall designate a safe area where all employees are to collect in the event the lab needs to be evacuated. If the lab is to be evacuated, it should be announced through the phone system. All employees should then evacuate through the closest safe exit and meet in the designated safe area.



The sign in/sign out sheet shall then be used to make sure everyone has been evacuated. Proceed with any cleanup as directed by the emergency coordinator.

7.0 Hazardous Waste Coordinator

- 7.1 The Hazardous Waste Coordinator shall be responsible for all record keeping and ensure that the proper documentation is kept for all required processes. This includes the keeping of manifests, weekly hazardous waste accumulation area inspection logs, and documents of this nature. In coordination with the lab's Health and Safety Officer, the Hazardous Waste Coordinator shall ensure that a schedule of routine inspection of the emergency equipment (spill kits, eye wash, etc.) and an annual inspection of the fire extinguisher from an outside vendor are performed with records kept. As it is now required the at a minimum, labs shall be inspected by FDEP once every four years, and that an inspection can occur at any time, all records should be kept up to date and easily accessible for review.
- 7.2 The Hazardous Waste Coordinator shall be responsible for ensuring that the local utility is informed of the type of waste and quantity of waste that the lab is discharging into the sewer system. Records of any correspondence and any instructions received shall be kept on file. Any instructions received shall be passed onto the lab through new hire and annual training.
- 7.3 The Hazardous Waste Coordinator shall inform local authorities (Fire Department, Police, Hospital, Emergency Manager) of what hazardous waste the lab has on hand. Also provided to the local authorities shall be the laboratory's layout, the types of hazards and hazardous waste, and the quantities of those hazards. The information (lab's contact included) shall be kept up to date with the local authorities.
- 7.4 The Hazardous Waste Coordinator shall ensure that the lab has emergency information posted next to each phone near a waste generation and accumulation area. This information will be displayed along with a map with emergency exits, eye wash stations, and fire extinguishers listed for the lab.
- 7.5 The Hazardous Waste Coordinator shall ensure that proper training is carried out and that through inspection that the procedures as outlined in this SOP are being followed through random inspection.

8.0 References

- 8.1 DEP Standard Operating Procedures for Laboratory Operations and Sample Collection Activities, DEP – QA-002/92.
- 8.2 40 CFR Parts 260-272 (RCRA Programs).



- 8.3 AEL Quality Manual, latest revision.
- 8.4 AEL Chemical Hygiene Plan and Safety Manual
- 8.5 From Federal Code of Regulations 40-261,262
- 8.6 Standard Methods, Online Edition
 - 8.6.1 Section 1090, laboratory Occupational Safety and Health.
 - 8.6.2 Section 1100, Waste Minimization and Disposal.
 - 8.6.3 Section 9020B Quality Assurance, Biosafety Criteria
- 8.7 *A Guide on Hazardous Waste Management for Florida's Laboratories*, FDEP Hazardous Waste Compliance Assistance Program, June 2003.

Appendix 1, **TABLE 1**

MAXIMUM CONCENTRATION OF CONTAMINANTS FOR TOXICITY CHARACTERISTIC (TCLP)

TABLE 1—MAXIMUM CONCENTRATION OF CONTAMINANTS FOR THE TOXICITY CHARACTERISTIC

| EPA HW No. ¹ | Contaminant | CAS No. ² | Regulatory Level (mg/L) |
|-------------------------|------------------------------|----------------------|-------------------------|
| D004 | Arsenic | 7440-38-2 | 5.0 |
| D005 | Barium | 7440-39-3 | 100.0 |
| D018 | Benzene | 71-43-2 | 0.5 |
| D006 | Cadmium | 7440-43-9 | 1.0 |
| D019 | Carbon tetrachloride | 56-23-5 | 0.5 |
| D020 | Chlordane | 57-74-9 | 0.03 |
| D021 | Chlorobenzene | 108-90-7 | 100.0 |
| D022 | Chloroform | 67-66-3 | 6.0 |
| D007 | Chromium | 7440-47-3 | 5.0 |
| D023 | o-Cresol | 95-48-7 | ⁴ 200.0 |
| D024 | m-Cresol | 108-39-4 | ⁴ 200.0 |
| D025 | p-Cresol | 106-44-5 | ⁴ 200.0 |
| D026 | Cresol | | ⁴ 200.0 |
| D016 | 2,4-D | 94-75-7 | 10.0 |
| D027 | 1,4-Dichlorobenzene | 106-46-7 | 7.5 |
| D028 | 1,2-Dichloroethane | 107-06-2 | 0.5 |
| D029 | 1,1-Dichloroethylene | 75-35-4 | 0.7 |
| D030 | 2,4-Dinitrotoluene | 121-14-2 | ³ 0.13 |
| D012 | Endrin | 72-20-8 | 0.02 |
| D031 | Heptachlor (and its epoxide) | 76-44-8 | 0.008 |
| D032 | Hexachlorobenzene | 118-74-1 | ³ 0.13 |



| | | | |
|------|-----------------------|-----------|------------------|
| D033 | Hexachlorobutadiene | 87-68-3 | 0.5 |
| D034 | Hexachloroethane | 67-72-1 | 3.0 |
| D008 | Lead | 7439-92-1 | 5.0 |
| D013 | Lindane | 58-89-9 | 0.4 |
| D009 | Mercury | 7439-97-6 | 0.2 |
| D014 | Methoxychlor | 72-43-5 | 10.0 |
| D035 | Methyl ethyl ketone | 78-93-3 | 200.0 |
| D036 | Nitrobenzene | 98-95-3 | 2.0 |
| D037 | Pentachlorophenol | 87-86-5 | 100.0 |
| D038 | Pyridine | 110-86-1 | ³ 5.0 |
| D010 | Selenium | 7782-49-2 | 1.0 |
| D011 | Silver | 7440-22-4 | 5.0 |
| D039 | Tetrachloroethylene | 127-18-4 | 0.7 |
| D015 | Toxaphene | 8001-35-2 | 0.5 |
| D040 | Trichloroethylene | 79-01-6 | 0.5 |
| D041 | 2,4,5-Trichlorophenol | 95-95-4 | 400.0 |
| D042 | 2,4,6-Trichlorophenol | 88-06-2 | 2.0 |
| D017 | 2,4,5-TP (Silvex) | 93-72-1 | 1.0 |
| D043 | Vinyl chloride | 75-01-4 | 0.2 |

1 If o-, m-, and p-cresol concentrations cannot be differentiated, the total cresol (D026) concentration is used. The regulatory level of total cresol is 200 mg/L.

2 Quantitation limit is greater than the calculated regulatory level. The quantitation limit therefore becomes the regulatory level.

Appendix 1, Table 2 **Hazardous wastes from non-specific sources.**

| | | |
|------|---|-----|
| F001 | The following spent halogenated solvents used in degreasing: Tetrachloroethylene, trichloroethylene, methylene chloride, 1,1,1-trichloroethane, carbon tetrachloride, and chlorinated fluorocarbons; all spent solvent mixtures/blends used in degreasing containing, before use, a total of ten percent or more (by volume) of one or more of the above halogenated solvents or those solvents listed in F002, F004, and F005; and still bottoms from the recovery of these spent solvents and spent solvent mixtures | (T) |
| F002 | The following spent halogenated solvents: Tetrachloroethylene, methylene chloride, trichloroethylene, 1,1,1-trichloroethane, chlorobenzene, 1,1,2-trichloro-1,2,2-trifluoroethane, ortho-dichlorobenzene, trichlorofluoromethane, and 1,1,2-trichloroethane; all spent solvent mixtures/blends containing, before use, a total of ten percent or more (by volume) of one or more of the above halogenated solvents or those listed in F001, F004, or F005; and still bottoms from the recovery of these spent solvents and spent solvent mixtures | (T) |



| | | |
|------|---|--------|
| F003 | The following spent non-halogenated solvents: Xylene, acetone, ethyl acetate, ethyl benzene, ethyl ether, methyl isobutyl ketone, n-butyl alcohol, cyclohexanone, and methanol; all spent solvent mixtures/blends containing, before use, only the above spent non-halogenated solvents; and all spent solvent mixtures/blends containing, before use, one or more of the above non-halogenated solvents, and, a total of ten percent or more (by volume) of one or more of those solvents listed in F001, F002, F004, and F005; and still bottoms from the recovery of these spent solvents and spent solvent mixtures | (I)* |
| F004 | The following spent non-halogenated solvents: Cresols and cresylic acid, and nitrobenzene; all spent solvent mixtures/blends containing, before use, a total of ten percent or more (by volume) of one or more of the above non-halogenated solvents or those solvents listed in F001, F002, and F005; and still bottoms from the recovery of these spent solvents and spent solvent mixtures | (T) |
| F005 | The following spent non-halogenated solvents: Toluene, methyl ethyl ketone, carbon disulfide, isobutanol, pyridine, benzene, 2-ethoxyethanol, and 2-nitropropane; all spent solvent mixtures/blends containing, before use, a total of ten percent or more (by volume) of one or more of the above non-halogenated solvents or those solvents listed in F001, F002, or F004; and still bottoms from the recovery of these spent solvents and spent solvent mixtures | (I, T) |

P-list and U-list Hazardous Waste Numbers for unused commercial chemical products.

These wastes and their corresponding EPA Hazardous Waste Numbers are:

| Hazardous waste No. | Chemical abstracts No. | Substance |
|---------------------|------------------------|-----------------------------------|
| P023 | 107-20-0 | Acetaldehyde, chloro- |
| P002 | 591-08-2 | Acetamide, N-(aminothioxomethyl)- |
| P057 | 640-19-7 | Acetamide, 2-fluoro- |
| P058 | 62-74-8 | Acetic acid, fluoro-, sodium salt |
| P002 | 591-08-2 | 1-Acetyl-2-thiourea |
| P003 | 107-02-8 | Acrolein |
| P070 | 116-06-3 | Aldicarb |
| P203 | 1646-88-4 | Aldicarb sulfone. |
| P004 | 309-00-2 | Aldrin |
| P005 | 107-18-6 | Allyl alcohol |
| P006 | 20859-73-8 | Aluminum phosphide (R,T) |
| P007 | 2763-96-4 | 5-(Aminomethyl)-3-isoxazolol |
| P008 | 504-24-5 | 4-Aminopyridine |



| | | |
|------|----------------------|---|
| P009 | 131-74-8 | Ammonium picrate (R) |
| P119 | 7803-55-6 | Ammonium vanadate |
| P099 | 506-61-6 | Argentate(1-), bis(cyano-C)-, potassium |
| P010 | 7778-39-4 | Arsenic acid H ₃ AsO ₄ |
| P012 | 1327-53-3 | Arsenic oxide As ₂ O ₃ |
| P011 | 1303-28-2 | Arsenic oxide As ₂ O ₅ |
| P011 | 1303-28-2 | Arsenic pentoxide |
| P012 | 1327-53-3 | Arsenic trioxide |
| P038 | 692-42-2 | Arsine, diethyl- |
| P036 | 696-28-6 | Arsonous dichloride, phenyl- |
| P054 | 151-56-4 | Aziridine |
| P067 | 75-55-8 | Aziridine, 2-methyl- |
| P013 | 542-62-1 | Barium cyanide |
| P024 | 106-47-8 | Benzenamine, 4-chloro- |
| P077 | 100-01-6 | Benzenamine, 4-nitro- |
| P028 | 100-44-7 | Benzene, (chloromethyl)- |
| P042 | 51-43-4 | 1,2-Benzenediol, 4-[1-hydroxy-2-(methylamino)ethyl]-, (R)- |
| P046 | 122-09-8 | Benzeneethanamine, alpha,alpha-dimethyl- |
| P014 | 108-98-5 | Benzenethiol |
| P127 | 1563-66-2 | 7-Benzofuranol, 2,3-dihydro-2,2-dimethyl-, methylcarbamate. |
| P188 | 57-64-7 | Benzoic acid, 2-hydroxy-, compd. with (3aS-cis)-1,2,3,3a,8,8a-hexahydro-1,3a,8-trimethylpyrrolo[2,3-b]indol-5-yl methylcarbamate ester (1:1). |
| P001 | ¹ 81-81-2 | 2H-1-Benzopyran-2-one, 4-hydroxy-3-(3-oxo-1-phenylbutyl)-, & salts, when present at concentrations greater than 0.3% |
| P028 | 100-44-7 | Benzyl chloride |
| P015 | 7440-41-7 | Beryllium powder |
| P017 | 598-31-2 | Bromoacetone |
| P018 | 357-57-3 | Brucine |
| P045 | 39196-18-4 | 2-Butanone, 3,3-dimethyl-1-(methylthio)-, O-[(methylamino)carbonyl] oxime |
| P021 | 592-01-8 | Calcium cyanide |
| P021 | 592-01-8 | Calcium cyanide Ca(CN) ₂ |
| P189 | 55285-14-8 | Carbamic acid, [(dibutylamino)- thio]methyl-, 2,3-dihydro-2,2-dimethyl- 7-benzofuranyl ester. |
| P191 | 644-64-4 | Carbamic acid, dimethyl-, 1-[(dimethyl-amino)carbonyl]- 5-methyl-1H- pyrazol-3-yl ester. |



| | | |
|------|----------------------|--|
| P192 | 119-38-0 | Carbamic acid, dimethyl-, 3-methyl-1- (1-methylethyl)-1H-pyrazol-5-yl ester. |
| P190 | 1129-41-5 | Carbamic acid, methyl-, 3-methylphenyl ester. |
| P127 | 1563-66-2 | Carbofuran. |
| P022 | 75-15-0 | Carbon disulfide |
| P095 | 75-44-5 | Carbonic dichloride |
| P189 | 55285-14-8 | Carbosulfan. |
| P023 | 107-20-0 | Chloroacetaldehyde |
| P024 | 106-47-8 | p-Chloroaniline |
| P026 | 5344-82-1 | 1-(o-Chlorophenyl)thiourea |
| P027 | 542-76-7 | 3-Chloropropionitrile |
| P029 | 544-92-3 | Copper cyanide |
| P029 | 544-92-3 | Copper cyanide Cu(CN) |
| P202 | 64-00-6 | m-Cumenyl methylcarbamate. |
| P030 | | Cyanides (soluble cyanide salts), not otherwise specified |
| P031 | 460-19-5 | Cyanogen |
| P033 | 506-77-4 | Cyanogen chloride |
| P033 | 506-77-4 | Cyanogen chloride (CN)Cl |
| P034 | 131-89-5 | 2-Cyclohexyl-4,6-dinitrophenol |
| P016 | 542-88-1 | Dichloromethyl ether |
| P036 | 696-28-6 | Dichlorophenylarsine |
| P037 | 60-57-1 | Dieldrin |
| P038 | 692-42-2 | Diethylarsine |
| P041 | 311-45-5 | Diethyl-p-nitrophenyl phosphate |
| P040 | 297-97-2 | O,O-Diethyl O-pyrazinyl phosphorothioate |
| P043 | 55-91-4 | Diisopropylfluorophosphate (DFP) |
| P004 | 309-00-2 | 1,4,5,8-Dimethanonaphthalene, 1,2,3,4,10,10-hexa- chloro-1,4,4a,5,8,8a,-hexahydro-, (1alpha,4alpha,4abeta,5alpha,8alpha,8abeta)- |
| P060 | 465-73-6 | 1,4,5,8-Dimethanonaphthalene, 1,2,3,4,10,10-hexa- chloro-1,4,4a,5,8,8a-hexahydro-, (1alpha,4alpha,4abeta,5beta,8beta,8abeta)- |
| P037 | 60-57-1 | 2,7:3,6-Dimethanonaphth[2,3-b]oxirene, 3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a-octahydro-, (1aalpha,2beta,2aalpha,3beta,6beta,6aalpha,7beta, 7aalpha)- |
| P051 | ¹ 72-20-8 | 2,7:3,6-Dimethanonaphth [2,3-b]oxirene, 3,4,5,6,9,9-hexachloro- 1a,2,2a,3,6,6a,7,7a-octahydro-, (1aalpha,2beta,2abeta,3alpha,6alpha,6abeta,7beta, 7aalpha)-, & |



| | | |
|------|-----------------------|---|
| | | metabolites |
| P044 | 60-51-5 | Dimethoate |
| P046 | 122-09-8 | alpha,alpha-Dimethylphenethylamine |
| P191 | 644-64-4 | Dimetilan. |
| P047 | ¹ 534-52-1 | 4,6-Dinitro-o-cresol, & salts |
| P048 | 51-28-5 | 2,4-Dinitrophenol |
| P020 | 88-85-7 | Dinoseb |
| P085 | 152-16-9 | Diphosphoramidate, octamethyl- |
| P111 | 107-49-3 | Diphosphoric acid, tetraethyl ester |
| P039 | 298-04-4 | Disulfoton |
| P049 | 541-53-7 | Dithiobiuret |
| P185 | 26419-73-8 | 1,3-Dithiolane-2-carboxaldehyde, 2,4-dimethyl-, O-[(methylamino)- carbonyl]oxime. |
| P050 | 115-29-7 | Endosulfan |
| P088 | 145-73-3 | Endothall |
| P051 | 72-20-8 | Endrin |
| P051 | 72-20-8 | Endrin, & metabolites |
| P042 | 51-43-4 | Epinephrine |
| P031 | 460-19-5 | Ethanedinitrile |
| P194 | 23135-22-0 | Ethanimidothioic acid, 2-(dimethylamino)-N-[[[(methylamino) carbonyl]oxy]-2-oxo-, methyl ester. |
| P066 | 16752-77-5 | Ethanimidothioic acid, N-[[[(methylamino)carbonyl]oxy]-, methyl ester |
| P101 | 107-12-0 | Ethyl cyanide |
| P054 | 151-56-4 | Ethyleneimine |
| P097 | 52-85-7 | Famphur |
| P056 | 7782-41-4 | Fluorine |
| P057 | 640-19-7 | Fluoroacetamide |
| P058 | 62-74-8 | Fluoroacetic acid, sodium salt |
| P198 | 23422-53-9 | Formetanate hydrochloride. |
| P197 | 17702-57-7 | Formparanate. |
| P065 | 628-86-4 | Fulminic acid, mercury(2 +) salt (R,T) |
| P059 | 76-44-8 | Heptachlor |
| P062 | 757-58-4 | Hexaethyl tetraphosphate |
| P116 | 79-19-6 | Hydrazinecarbothioamide |
| P068 | 60-34-4 | Hydrazine, methyl- |



| | | |
|------|------------|---|
| P063 | 74-90-8 | Hydrocyanic acid |
| P063 | 74-90-8 | Hydrogen cyanide |
| P096 | 7803-51-2 | Hydrogen phosphide |
| P060 | 465-73-6 | Isodrin |
| P192 | 119-38-0 | Isolan. |
| P202 | 64-00-6 | 3-Isopropylphenyl N-methylcarbamate. |
| P007 | 2763-96-4 | 3(2H)-Isoxazolone, 5-(aminomethyl)- |
| P196 | 15339-36-3 | Manganese, bis(dimethylcarbamodithioato-S,S')-, |
| P196 | 15339-36-3 | Manganese dimethyldithiocarbamate. |
| P092 | 62-38-4 | Mercury, (acetato-O)phenyl- |
| P065 | 628-86-4 | Mercury fulminate (R,T) |
| P082 | 62-75-9 | Methanamine, N-methyl-N-nitroso- |
| P064 | 624-83-9 | Methane, isocyanato- |
| P016 | 542-88-1 | Methane, oxybis[chloro- |
| P112 | 509-14-8 | Methane, tetranitro- (R) |
| P118 | 75-70-7 | Methanethiol, trichloro- |
| P198 | 23422-53-9 | Methanimidamide, N,N-dimethyl-N'-[3-[[methylamino)- carbonyl]oxy]phenyl]-, monohydrochloride. |
| P197 | 17702-57-7 | Methanimidamide, N,N-dimethyl-N'-[2-methyl-4- [[methylamino)carbonyl]oxy]phenyl]- |
| P050 | 115-29-7 | 6,9-Methano-2,4,3-benzodioxathiepin, 6,7,8,9,10,10- hexachloro-1,5,5a,6,9,9a-hexahydro-, 3-oxide |
| P059 | 76-44-8 | 4,7-Methano-1H-indene, 1,4,5,6,7,8,8-heptachloro- 3a,4,7,7a-tetrahydro- |
| P199 | 2032-65-7 | Methiocarb. |
| P066 | 16752-77-5 | Methomyl |
| P068 | 60-34-4 | Methyl hydrazine |
| P064 | 624-83-9 | Methyl isocyanate |
| P069 | 75-86-5 | 2-Methylactonitrile |
| P071 | 298-00-0 | Methyl parathion |
| P190 | 1129-41-5 | Metolcarb. |
| P128 | 315-8-4 | Mexacarbate. |
| P072 | 86-88-4 | alpha-Naphthylthiourea |
| P073 | 13463-39-3 | Nickel carbonyl |
| P073 | 13463-39-3 | Nickel carbonyl Ni(CO) ₄ , (T-4)- |
| P074 | 557-19-7 | Nickel cyanide |
| P074 | 557-19-7 | Nickel cyanide Ni(CN) ₂ |



| | | |
|------|-----------------------|---|
| P075 | ¹ 54-11-5 | Nicotine, & salts (this listing does not include patches, gums and lozenges that are FDA-approved over-the-counter nicotine replacement therapies). |
| P076 | 10102-43-9 | Nitric oxide |
| P077 | 100-01-6 | p-Nitroaniline |
| P078 | 10102-44-0 | Nitrogen dioxide |
| P076 | 10102-43-9 | Nitrogen oxide NO |
| P078 | 10102-44-0 | Nitrogen oxide NO ₂ |
| P081 | 55-63-0 | Nitroglycerine (R) |
| P082 | 62-75-9 | N-Nitrosodimethylamine |
| P084 | 4549-40-0 | N-Nitrosomethylvinylamine |
| P085 | 152-16-9 | Octamethylpyrophosphoramidate |
| P087 | 20816-12-0 | Osmium oxide OsO ₄ , (T-4)- |
| P087 | 20816-12-0 | Osmium tetroxide |
| P088 | 145-73-3 | 7-Oxabicyclo[2.2.1]heptane-2,3-dicarboxylic acid |
| P194 | 23135-22-0 | Oxamyl. |
| P089 | 56-38-2 | Parathion |
| P034 | 131-89-5 | Phenol, 2-cyclohexyl-4,6-dinitro- |
| P048 | 51-28-5 | Phenol, 2,4-dinitro- |
| P047 | ¹ 534-52-1 | Phenol, 2-methyl-4,6-dinitro-, & salts |
| P020 | 88-85-7 | Phenol, 2-(1-methylpropyl)-4,6-dinitro- |
| P009 | 131-74-8 | Phenol, 2,4,6-trinitro-, ammonium salt (R) |
| P128 | 315-18-4 | Phenol, 4-(dimethylamino)-3,5-dimethyl-, methylcarbamate (ester). |
| P199 | 2032-65-7 | Phenol, (3,5-dimethyl-4-(methylthio)-), methylcarbamate |
| P202 | 64-00-6 | Phenol, 3-(1-methylethyl)-, methyl carbamate. |
| P201 | 2631-37-0 | Phenol, 3-methyl-5-(1-methylethyl)-, methyl carbamate. |
| P092 | 62-38-4 | Phenylmercury acetate |
| P093 | 103-85-5 | Phenylthiourea |
| P094 | 298-02-2 | Phorate |
| P095 | 75-44-5 | Phosgene |
| P096 | 7803-51-2 | Phosphine |
| P041 | 311-45-5 | Phosphoric acid, diethyl 4-nitrophenyl ester |
| P039 | 298-04-4 | Phosphorodithioic acid, O,O-diethyl S-[2-(ethylthio)ethyl] ester |
| P094 | 298-02-2 | Phosphorodithioic acid, O,O-diethyl S-[(ethylthio)methyl] ester |



| | | |
|------|----------------------|---|
| P044 | 60-51-5 | Phosphorodithioic acid, O,O-dimethyl S-[2-(methylamino)-2-oxoethyl] ester |
| P043 | 55-91-4 | Phosphorofluoridic acid, bis(1-methylethyl) ester |
| P089 | 56-38-2 | Phosphorothioic acid, O,O-diethyl O-(4-nitrophenyl) ester |
| P040 | 297-97-2 | Phosphorothioic acid, O,O-diethyl O-pyrazinyl ester |
| P097 | 52-85-7 | Phosphorothioic acid, O-[4-[(dimethylamino)sulfonyl]phenyl] O,O-dimethyl ester |
| P071 | 298-00-0 | Phosphorothioic acid, O,O,-dimethyl O-(4-nitrophenyl) ester |
| P204 | 57-47-6 | Physostigmine. |
| P188 | 57-64-7 | Physostigmine salicylate. |
| P110 | 78-00-2 | Plumbane, tetraethyl- |
| P098 | 151-50-8 | Potassium cyanide |
| P098 | 151-50-8 | Potassium cyanide K(CN) |
| P099 | 506-61-6 | Potassium silver cyanide |
| P201 | 2631-37-0 | Promecarb |
| P070 | 116-06-3 | Propanal, 2-methyl-2-(methylthio)-, O-[(methylamino)carbonyl]oxime |
| P203 | 1646-88-4 | Propanal, 2-methyl-2-(methyl-sulfonyl)-, O- [(methylamino)carbonyl] oxime. |
| P101 | 107-12-0 | Propanenitrile |
| P027 | 542-76-7 | Propanenitrile, 3-chloro- |
| P069 | 75-86-5 | Propanenitrile, 2-hydroxy-2-methyl- |
| P081 | 55-63-0 | 1,2,3-Propanetriol, trinitrate (R) |
| P017 | 598-31-2 | 2-Propanone, 1-bromo- |
| P102 | 107-19-7 | Propargyl alcohol |
| P003 | 107-02-8 | 2-Propenal |
| P005 | 107-18-6 | 2-Propen-1-ol |
| P067 | 75-55-8 | 1,2-Propylenimine |
| P102 | 107-19-7 | 2-Propyn-1-ol |
| P008 | 504-24-5 | 4-Pyridinamine |
| P075 | ¹ 54-11-5 | Pyridine, 3-(1-methyl-2-pyrrolidinyl)-, (S)-, & salts (this listing does not include patches, gums and lozenges that are FDA-approved over-the-counter nicotine replacement therapies). |
| P204 | 57-47-6 | Pyrrolo[2,3-b]indol-5-ol, 1,2,3,3a,8,8a-hexahydro-1,3a,8-trimethyl-, methylcarbamate (ester), (3aS-cis)-. |
| P114 | 12039-52-0 | Selenious acid, dithallium(1 +) salt |



| | | |
|------|----------------------|--|
| P103 | 630-10-4 | Selenourea |
| P104 | 506-64-9 | Silver cyanide |
| P104 | 506-64-9 | Silver cyanide Ag(CN) |
| P105 | 26628-22-8 | Sodium azide |
| P106 | 143-33-9 | Sodium cyanide |
| P106 | 143-33-9 | Sodium cyanide Na(CN) |
| P108 | ¹ 57-24-9 | Strychnidin-10-one, & salts |
| P018 | 357-57-3 | Strychnidin-10-one, 2,3-dimethoxy- |
| P108 | ¹ 57-24-9 | Strychnine, & salts |
| P115 | 7446-18-6 | Sulfuric acid, dithallium(1 +) salt |
| P109 | 3689-24-5 | Tetraethyldithiopyrophosphate |
| P110 | 78-00-2 | Tetraethyl lead |
| P111 | 107-49-3 | Tetraethyl pyrophosphate |
| P112 | 509-14-8 | Tetranitromethane (R) |
| P062 | 757-58-4 | Tetraphosphoric acid, hexaethyl ester |
| P113 | 1314-32-5 | Thallic oxide |
| P113 | 1314-32-5 | Thallium oxide Tl ₂ O ₃ |
| P114 | 12039-52-0 | Thallium(I) selenite |
| P115 | 7446-18-6 | Thallium(I) sulfate |
| P109 | 3689-24-5 | Thiodiphosphoric acid, tetraethyl ester |
| P045 | 39196-18-4 | Thiofanox |
| P049 | 541-53-7 | Thioimidodicarbonic diamide [(H ₂ N)C(S)] ₂ NH |
| P014 | 108-98-5 | Thiophenol |
| P116 | 79-19-6 | Thiosemicarbazide |
| P026 | 5344-82-1 | Thiourea, (2-chlorophenyl)- |
| P072 | 86-88-4 | Thiourea, 1-naphthalenyl- |
| P093 | 103-85-5 | Thiourea, phenyl- |
| P185 | 26419-73-8 | Tirpate. |
| P123 | 8001-35-2 | Toxaphene |
| P118 | 75-70-7 | Trichloromethanethiol |
| P119 | 7803-55-6 | Vanadic acid, ammonium salt |
| P120 | 1314-62-1 | Vanadium oxide V ₂ O ₅ |
| P120 | 1314-62-1 | Vanadium pentoxide |
| P084 | 4549-40-0 | Vinylamine, N-methyl-N-nitroso- |
| P001 | ¹ 81-81-2 | Warfarin, & salts, when present at concentrations greater than 0.3% |



| | | |
|------|------------|---|
| P205 | 137-30-4 | Zinc, bis(dimethylcarbamo-dithioato-S,S')-, |
| P121 | 557-21-1 | Zinc cyanide |
| P121 | 557-21-1 | Zinc cyanide $Zn(CN)_2$ |
| P122 | 1314-84-7 | Zinc phosphide $Zn_3 P_2$, when present at concentrations greater than 10% (R,T) |
| P205 | 137-30-4 | Ziram. |
| P001 | 181-81-2 | 2H-1-Benzopyran-2-one, 4-hydroxy-3-(3-oxo-1-phenylbutyl)-, & salts, when present at concentrations greater than 0.3% |
| P001 | 181-81-2 | Warfarin, & salts, when present at concentrations greater than 0.3% |
| P002 | 591-08-2 | Acetamide, -(aminothioxomethyl)- |
| P002 | 591-08-2 | 1-Acetyl-2-thiourea |
| P003 | 107-02-8 | Acrolein |
| P003 | 107-02-8 | 2-Propenal |
| P004 | 309-00-2 | Aldrin |
| P004 | 309-00-2 | 1,4,5,8-Dimethanonaphthalene, 1,2,3,4,10,10-hexa-chloro-1,4,4a,5,8,8a,-hexahydro-, (1alpha,4alpha,4abeta,5alpha,8alpha,8abeta)- |
| P005 | 107-18-6 | Allyl alcohol |
| P005 | 107-18-6 | 2-Propen-1-ol |
| P006 | 20859-73-8 | Aluminum phosphide (R,T) |
| P007 | 2763-96-4 | 5-(Aminomethyl)-3-isoxazolol |
| P007 | 2763-96-4 | 3(2H)-Isoxazolone, 5-(aminomethyl)- |
| P008 | 504-24-5 | 4-Aminopyridine |
| P008 | 504-24-5 | 4-Pyridinamine |
| P009 | 131-74-8 | Ammonium picrate (R) |
| P009 | 131-74-8 | Phenol, 2,4,6-trinitro-, ammonium salt (R) |
| P010 | 7778-39-4 | Arsenic acid $H_3 AsO_4$ |
| P011 | 1303-28-2 | Arsenic oxide $As_2 O_5$ |
| P011 | 1303-28-2 | Arsenic pentoxide |
| P012 | 1327-53-3 | Arsenic oxide $As_2 O_3$ |
| P012 | 1327-53-3 | Arsenic trioxide |
| P013 | 542-62-1 | Barium cyanide |
| P014 | 108-98-5 | Benzenethiol |
| P014 | 108-98-5 | Thiophenol |
| P015 | 7440-41-7 | Beryllium powder |
| P016 | 542-88-1 | Dichloromethyl ether |



| | | |
|------|-----------|---|
| P016 | 542-88-1 | Methane, oxybis[chloro- |
| P017 | 598-31-2 | Bromoacetone |
| P017 | 598-31-2 | 2-Propanone, 1-bromo- |
| P018 | 357-57-3 | Brucine |
| P018 | 357-57-3 | Strychnidin-10-one, 2,3-dimethoxy- |
| P020 | 88-85-7 | Dinoseb |
| P020 | 88-85-7 | Phenol, 2-(1-methylpropyl)-4,6-dinitro- |
| P021 | 592-01-8 | Calcium cyanide |
| P021 | 592-01-8 | Calcium cyanide Ca(CN) ₂ |
| P022 | 75-15-0 | Carbon disulfide |
| P023 | 107-20-0 | Acetaldehyde, chloro- |
| P023 | 107-20-0 | Chloroacetaldehyde |
| P024 | 106-47-8 | Benzenamine, 4-chloro- |
| P024 | 106-47-8 | p-Chloroaniline |
| P026 | 5344-82-1 | 1-(o-Chlorophenyl)thiourea |
| P026 | 5344-82-1 | Thiourea, (2-chlorophenyl)- |
| P027 | 542-76-7 | 3-Chloropropionitrile |
| P027 | 542-76-7 | Propanenitrile, 3-chloro- |
| P028 | 100-44-7 | Benzene, (chloromethyl)- |
| P028 | 100-44-7 | Benzyl chloride |
| P029 | 544-92-3 | Copper cyanide |
| P029 | 544-92-3 | Copper cyanide Cu(CN) |
| P030 | | Cyanides (soluble cyanide salts), not otherwise specified |
| P031 | 460-19-5 | Cyanogen |
| P031 | 460-19-5 | Ethanedinitrile |
| P033 | 506-77-4 | Cyanogen chloride |
| P033 | 506-77-4 | Cyanogen chloride (CN)Cl |
| P034 | 131-89-5 | 2-Cyclohexyl-4,6-dinitrophenol |
| P034 | 131-89-5 | Phenol, 2-cyclohexyl-4,6-dinitro- |
| P036 | 696-28-6 | Arsonous dichloride, phenyl- |
| P036 | 696-28-6 | Dichlorophenylarsine |
| P037 | 60-57-1 | Dieldrin |
| P037 | 60-57-1 | 2,7:3,6-Dimethanonaphth[2,3-b]oxirene, 3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a-octahydro-, (1aalpha,2beta,2aalpha,3beta,6beta,6aalpha,7beta, 7aalpha)- |
| P038 | 692-42-2 | Arsine, diethyl- |



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| P038 | 692-42-2 | Diethylarsine |
| P039 | 298-04-4 | Disulfoton |
| P039 | 298-04-4 | Phosphorodithioic acid, O,O-diethyl S-[2-(ethylthio)ethyl] ester |
| P040 | 297-97-2 | O,O-Diethyl O-pyrazinyl phosphorothioate |
| P040 | 297-97-2 | Phosphorothioic acid, O,O-diethyl O-pyrazinyl ester |
| P041 | 311-45-5 | Diethyl-p-nitrophenyl phosphate |
| P041 | 311-45-5 | Phosphoric acid, diethyl 4-nitrophenyl ester |
| P042 | 51-43-4 | 1,2-Benzenediol, 4-[1-hydroxy-2-(methylamino)ethyl]-, (R)- |
| P042 | 51-43-4 | Epinephrine |
| P043 | 55-91-4 | Diisopropylfluorophosphate (DFP) |
| P043 | 55-91-4 | Phosphorofluoridic acid, bis(1-methylethyl) ester |
| P044 | 60-51-5 | Dimethoate |
| P044 | 60-51-5 | Phosphorodithioic acid, O,O-dimethyl S-[2-(methyl amino)-2-oxoethyl] ester |
| P045 | 39196-18-4 | 2-Butanone, 3,3-dimethyl-1-(methylthio)-, O-[(methylamino)carbonyl] oxime |
| P045 | 39196-18-4 | Thiofanox |
| P046 | 122-09-8 | Benzeneethanamine, alpha,alpha-dimethyl- |
| P046 | 122-09-8 | alpha,alpha-Dimethylphenethylamine |
| P047 | ¹ 534-52-1 | 4,6-Dinitro-o-cresol, & salts |
| P047 | ¹ 534-52-1 | Phenol, 2-methyl-4,6-dinitro-, & salts |
| P048 | 51-28-5 | 2,4-Dinitrophenol |
| P048 | 51-28-5 | Phenol, 2,4-dinitro- |
| P049 | 541-53-7 | Dithiobiuret |
| P049 | 541-53-7 | Thioimidodicarbonic diamide [(H ₂ N)C(S)] ₂ NH |
| P050 | 115-29-7 | Endosulfan |
| P050 | 115-29-7 | 6,9-Methano-2,4,3-benzodioxathiepin, 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-, 3-oxide |
| P051 | ¹ 72-20-8 | 2,7:3,6-Dimethanonaphth [2,3-b]oxirene, 3,4,5,6,9,9-hexachloro-1a,2,2a,3,6,6a,7,7a-octahydro-, (1aalpha,2beta,2beta,3alpha,6alpha,6alpha,7beta,7beta,7aalpha)-, & metabolites |
| P051 | 72-20-8 | Endrin |
| P051 | 72-20-8 | Endrin, & metabolites |
| P054 | 151-56-4 | Aziridine |
| P054 | 151-56-4 | Ethyleneimine |
| P056 | 7782-41-4 | Fluorine |



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| P057 | 640-19-7 | Acetamide, 2-fluoro- |
| P057 | 640-19-7 | Fluoroacetamide |
| P058 | 62-74-8 | Acetic acid, fluoro-, sodium salt |
| P058 | 62-74-8 | Fluoroacetic acid, sodium salt |
| P059 | 76-44-8 | Heptachlor |
| P059 | 76-44-8 | 4,7-Methano-1H-indene, 1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro- |
| P060 | 465-73-6 | 1,4,5,8-Dimethanonaphthalene, 1,2,3,4,10,10-hexa-chloro-1,4,4a,5,8,8a-hexahydro-, (1alpha,4alpha,4beta,5beta,8beta,8beta)- |
| P060 | 465-73-6 | Isodrin |
| P062 | 757-58-4 | Hexaethyl tetraphosphate |
| P062 | 757-58-4 | Tetraphosphoric acid, hexaethyl ester |
| P063 | 74-90-8 | Hydrocyanic acid |
| P063 | 74-90-8 | Hydrogen cyanide |
| P064 | 624-83-9 | Methane, isocyanato- |
| P064 | 624-83-9 | Methyl isocyanate |
| P065 | 628-86-4 | Fulminic acid, mercury(2 +) salt (R,T) |
| P065 | 628-86-4 | Mercury fulminate (R,T) |
| P066 | 16752-77-5 | Ethanimidothioic acid, N-[[[(methylamino)carbonyl]oxy]-, methyl ester |
| P066 | 16752-77-5 | Methomyl |
| P067 | 75-55-8 | Aziridine, 2-methyl- |
| P067 | 75-55-8 | 1,2-Propylenimine |
| P068 | 60-34-4 | Hydrazine, methyl- |
| P068 | 60-34-4 | Methyl hydrazine |
| P069 | 75-86-5 | 2-Methylactonitrile |
| P069 | 75-86-5 | Propanenitrile, 2-hydroxy-2-methyl- |
| P070 | 116-06-3 | Aldicarb |
| P070 | 116-06-3 | Propanal, 2-methyl-2-(methylthio)-, O-[(methylamino)carbonyl]oxime |
| P071 | 298-00-0 | Methyl parathion |
| P071 | 298-00-0 | Phosphorothioic acid, O,O,-dimethyl O-(4-nitrophenyl) ester |
| P072 | 86-88-4 | alpha-Naphthylthiourea |
| P072 | 86-88-4 | Thiourea, 1-naphthalenyl- |
| P073 | 13463-39-3 | Nickel carbonyl |
| P073 | 13463-39-3 | Nickel carbonyl Ni(CO) ₄ , (T-4)- |



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| P074 | 557-19-7 | Nickel cyanide |
| P074 | 557-19-7 | Nickel cyanide Ni(CN) ₂ |
| P075 | ¹ 54-11-5 | Nicotine, & salts (this listing does not include patches, gums and lozenges that are FDA-approved over-the-counter nicotine replacement therapies). |
| P075 | ¹ 54-11-5 | Pyridine, 3-(1-methyl-2-pyrrolidinyl)-, (S)-, & salts (this listing does not include patches, gums and lozenges that are FDA-approved over-the-counter nicotine replacement therapies). |
| P076 | 10102-43-9 | Nitric oxide |
| P076 | 10102-43-9 | Nitrogen oxide NO |
| P077 | 100-01-6 | Benzenamine, 4-nitro- |
| P077 | 100-01-6 | p-Nitroaniline |
| P078 | 10102-44-0 | Nitrogen dioxide |
| P078 | 10102-44-0 | Nitrogen oxide NO ₂ |
| P081 | 55-63-0 | Nitroglycerine (R) |
| P081 | 55-63-0 | 1,2,3-Propanetriol, trinitrate (R) |
| P082 | 62-75-9 | Methanamine, -methyl-N-nitroso- |
| P082 | 62-75-9 | N-Nitrosodimethylamine |
| P084 | 4549-40-0 | N-Nitrosomethylvinylamine |
| P084 | 4549-40-0 | Vinylamine, -methyl-N-nitroso- |
| P085 | 152-16-9 | Diphosphoramidate, octamethyl- |
| P085 | 152-16-9 | Octamethylpyrophosphoramidate |
| P087 | 20816-12-0 | Osmium oxide OsO ₄ , (T-4)- |
| P087 | 20816-12-0 | Osmium tetroxide |
| P088 | 145-73-3 | Endothall |
| P088 | 145-73-3 | 7-Oxabicyclo[2.2.1]heptane-2,3-dicarboxylic acid |
| P089 | 56-38-2 | Parathion |
| P089 | 56-38-2 | Phosphorothioic acid, O,O-diethyl O-(4-nitrophenyl) ester |
| P092 | 62-38-4 | Mercury, (acetato-O)phenyl- |
| P092 | 62-38-4 | Phenylmercury acetate |
| P093 | 103-85-5 | Phenylthiourea |
| P093 | 103-85-5 | Thiourea, phenyl- |
| P094 | 298-02-2 | Phorate |
| P094 | 298-02-2 | Phosphorodithioic acid, O,O-diethyl S-[(ethylthio)methyl] ester |
| P095 | 75-44-5 | Carbonic dichloride |
| P095 | 75-44-5 | Phosgene |
| P096 | 7803-51-2 | Hydrogen phosphide |



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| P096 | 7803-51-2 | Phosphine |
| P097 | 52-85-7 | Famphur |
| P097 | 52-85-7 | Phosphorothioic acid, O-[4-[(dimethylamino)sulfonyl]phenyl] O,O-dimethyl ester |
| P098 | 151-50-8 | Potassium cyanide |
| P098 | 151-50-8 | Potassium cyanide K(CN) |
| P099 | 506-61-6 | Argentate(1-), bis(cyano-C)-, potassium |
| P099 | 506-61-6 | Potassium silver cyanide |
| P101 | 107-12-0 | Ethyl cyanide |
| P101 | 107-12-0 | Propanenitrile |
| P102 | 107-19-7 | Propargyl alcohol |
| P102 | 107-19-7 | 2-Propyn-1-ol |
| P103 | 630-10-4 | Selenourea |
| P104 | 506-64-9 | Silver cyanide |
| P104 | 506-64-9 | Silver cyanide Ag(CN) |
| P105 | 26628-22-8 | Sodium azide |
| P106 | 143-33-9 | Sodium cyanide |
| P106 | 143-33-9 | Sodium cyanide Na(CN) |
| P108 | ¹ 157-24-9 | Strychnidin-10-one, & salts |
| P108 | ¹ 157-24-9 | Strychnine, & salts |
| P109 | 3689-24-5 | Tetraethyldithiopyrophosphate |
| P109 | 3689-24-5 | Thiodiphosphoric acid, tetraethyl ester |
| P110 | 78-00-2 | Plumbane, tetraethyl- |
| P110 | 78-00-2 | Tetraethyl lead |
| P111 | 107-49-3 | Diphosphoric acid, tetraethyl ester |
| P111 | 107-49-3 | Tetraethyl pyrophosphate |
| P112 | 509-14-8 | Methane, tetranitro-(R) |
| P112 | 509-14-8 | Tetranitromethane (R) |
| P113 | 1314-32-5 | Thallic oxide |
| P113 | 1314-32-5 | Thallium oxide Tl ₂ O ₃ |
| P114 | 12039-52-0 | Selenious acid, dithallium(1 +) salt |
| P114 | 12039-52-0 | Tetraethyldithiopyrophosphate |
| P115 | 7446-18-6 | Thiodiphosphoric acid, tetraethyl ester |
| P115 | 7446-18-6 | Plumbane, tetraethyl- |
| P116 | 79-19-6 | Tetraethyl lead |
| P116 | 79-19-6 | Thiosemicarbazide |



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| P118 | 75-70-7 | Methanethiol, trichloro- |
| P118 | 75-70-7 | Trichloromethanethiol |
| P119 | 7803-55-6 | Ammonium vanadate |
| P119 | 7803-55-6 | Vanadic acid, ammonium salt |
| P120 | 1314-62-1 | Vanadium oxide V ₂ O ₅ |
| P120 | 1314-62-1 | Vanadium pentoxide |
| P121 | 557-21-1 | Zinc cyanide |
| P121 | 557-21-1 | Zinc cyanide Zn(CN) ₂ |
| P122 | 1314-84-7 | Zinc phosphide Zn ₃ P ₂ , when present at concentrations greater than 10% (R,T) |
| P123 | 8001-35-2 | Toxaphene |
| P127 | 1563-66-2 | 7-Benzofuranol, 2,3-dihydro-2,2-dimethyl-, methylcarbamate. |
| P127 | 1563-66-2 | Carbofuran |
| P128 | 315-8-4 | Mexacarbate |
| P128 | 315-18-4 | Phenol, 4-(dimethylamino)-3,5-dimethyl-, methylcarbamate (ester) |
| P185 | 26419-73-8 | 1,3-Dithiolane-2-carboxaldehyde, 2,4-dimethyl-, O-[(methylamino)-carbonyl]oxime. |
| P185 | 26419-73-8 | Tirpate |
| P188 | 57-64-7 | Benzoic acid, 2-hydroxy-, compd. with (3a <i>S</i> - <i>cis</i>)-1,2,3,3a,8,8a-hexahydro-1,3a,8-trimethylpyrrolo[2,3- <i>b</i>]indol-5-yl methylcarbamate ester (1:1) |
| P188 | 57-64-7 | Physostigmine salicylate |
| P189 | 55285-14-8 | Carbamic acid, [(dibutylamino)-thio]methyl-, 2,3-dihydro-2,2-dimethyl-7-benzofuranyl ester |
| P189 | 55285-14-8 | Carbosulfan |
| P190 | 1129-41-5 | Carbamic acid, methyl-, 3-methylphenyl ester |
| P190 | 1129-41-5 | Metolcarb |
| P191 | 644-64-4 | Carbamic acid, dimethyl-, 1-[(dimethyl-amino)carbonyl]-5-methyl-1 <i>H</i> -pyrazol-3-yl ester |
| P191 | 644-64-4 | Dimetilan |
| P192 | 119-38-0 | Carbamic acid, dimethyl-, 3-methyl-1-(1-methylethyl)-1 <i>H</i> -pyrazol-5-yl ester |
| P192 | 119-38-0 | Isolan |
| P194 | 23135-22-0 | Ethanimidthioic acid, 2-(dimethylamino)- <i>N</i> -[[[(methylamino)carbonyl]oxy]-2-oxo-, methyl ester |
| P194 | 23135-22-0 | Oxamyl |
| P196 | 15339-36-3 | Manganese, bis(dimethylcarbamodithioato- <i>S,S'</i>)-, |



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| P196 | 15339-36-3 | Manganese dimethyldithiocarbamate |
| P197 | 17702-57-7 | Formparanate |
| P197 | 17702-57-7 | Methanimidamide, N,N-dimethyl-N'-[2-methyl-4- [[[(methylamino)carbonyl]oxy]phenyl]- |
| P198 | 23422-53-9 | Formetanate hydrochloride |
| P198 | 23422-53-9 | Methanimidamide, N,N-dimethyl-N'-[3-[[[(methylamino)- carbonyl]oxy]phenyl]-monohydrochloride |
| P199 | 2032-65-7 | Methiocarb |
| P199 | 2032-65-7 | Phenol, (3,5-dimethyl-4-(methylthio)-, methylcarbamate |
| P201 | 2631-37-0 | Phenol, 3-methyl-5-(1-methylethyl)-, methyl carbamate |
| P201 | 2631-37-0 | Promecarb |
| P202 | 64-00-6 | m-Cumenyl methylcarbamate |
| P202 | 64-00-6 | 3-Isopropylphenyl N-methylcarbamate |
| P202 | 64-00-6 | Phenol, 3-(1-methylethyl)-, methyl carbamate |
| P203 | 1646-88-4 | Aldicarb sulfone |
| P203 | 1646-88-4 | Propanal, 2-methyl-2-(methyl-sulfonyl)-, O- [[[(methylamino)carbonyl] oxime |
| P204 | 57-47-6 | Physostigmine |
| P204 | 57-47-6 | Pyrrolo[2,3-b]indol-5-ol, 1,2,3,3a,8,8a-hexahydro-1,3a,8- trimethyl-, methylcarbamate (ester), (3aS-cis)- |
| P205 | 137-30-4 | Zinc, bis(dimethylcarbamo-dithioato-S,S')-, |
| P205 | 137-30-4 | Ziram |

¹CAS Number given for parent compound only.

(f) The commercial chemical products, manufacturing chemical intermediates, or off-specification commercial chemical products referred to in paragraphs (a) through (d) of this section, are identified as toxic wastes (T) unless otherwise designated.

[*Comment:* For the convenience of the regulated community, the primary hazardous properties of these materials have been indicated by the letters T (Toxicity), R (Reactivity), I (Ignitability) and C (Corrosivity). Absence of a letter indicates that the compound is only listed for toxicity. Wastes are first listed in alphabetical order by substance and then listed again in numerical order by Hazardous Waste Number.]

These wastes and their corresponding EPA Hazardous Waste Numbers are:

| Hazardous waste No. | Chemical abstracts No. | Substance |
|---------------------|------------------------|------------------|
| U394 | 30558-43-1 | A2213. |
| U001 | 75-07-0 | Acetaldehyde (I) |



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| U034 | 75-87-6 | Acetaldehyde, trichloro- |
| U187 | 62-44-2 | Acetamide, N-(4-ethoxyphenyl)- |
| U005 | 53-96-3 | Acetamide, N-9H-fluoren-2-yl- |
| U240 | ¹ 94-75-7 | Acetic acid, (2,4-dichlorophenoxy)-, salts & esters |
| U112 | 141-78-6 | Acetic acid ethyl ester (I) |
| U144 | 301-04-2 | Acetic acid, lead(2 +) salt |
| U214 | 563-68-8 | Acetic acid, thallium(1 +) salt |
| see F027 | 93-76-5 | Acetic acid, (2,4,5-trichlorophenoxy)- |
| U002 | 67-64-1 | Acetone (I) |
| U003 | 75-05-8 | Acetonitrile (I,T) |
| U004 | 98-86-2 | Acetophenone |
| U005 | 53-96-3 | 2-Acetylaminofluorene |
| U006 | 75-36-5 | Acetyl chloride (C,R,T) |
| U007 | 79-06-1 | Acrylamide |
| U008 | 79-10-7 | Acrylic acid (I) |
| U009 | 107-13-1 | Acrylonitrile |
| U011 | 61-82-5 | Amitrole |
| U012 | 62-53-3 | Aniline (I,T) |
| U136 | 75-60-5 | Arsinic acid, dimethyl- |
| U014 | 492-80-8 | Auramine |
| U015 | 115-02-6 | Azaserine |
| U010 | 50-07-7 | Azirino[2',3':3,4]pyrrolo[1,2-a]indole-4,7-dione, 6-amino-8-[[aminocarbonyl]oxy]methyl]-1,1a,2,8,8a,8b-hexahydro-8a-methoxy-5-methyl-, [1aS-(1aalpha, 8beta,8aalpha,8balpha)]- |
| U280 | 101-27-9 | Barban. |
| U278 | 22781-23-3 | Bendiocarb. |
| U364 | 22961-82-6 | Bendiocarb phenol. |
| U271 | 17804-35-2 | Benomyl. |
| U157 | 56-49-5 | Benz[j]aceanthrylene, 1,2-dihydro-3-methyl- |
| U016 | 225-51-4 | Benz[c]acridine |
| U017 | 98-87-3 | Benzal chloride |
| U192 | 23950-58-5 | Benzamide, 3,5-dichloro-N-(1,1-dimethyl-2-propynyl)- |
| U018 | 56-55-3 | Benz[a]anthracene |
| U094 | 57-97-6 | Benz[a]anthracene, 7,12-dimethyl- |
| U012 | 62-53-3 | Benzenamine (I,T) |



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|------|------------|---|
| U014 | 492-80-8 | Benzenamine, 4,4'-carbonimidoylbis[N,N-dimethyl- |
| U049 | 3165-93-3 | Benzenamine, 4-chloro-2-methyl-, hydrochloride |
| U093 | 60-11-7 | Benzenamine, N,N-dimethyl-4-(phenylazo)- |
| U328 | 95-53-4 | Benzenamine, 2-methyl- |
| U353 | 106-49-0 | Benzenamine, 4-methyl- |
| U158 | 101-14-4 | Benzenamine, 4,4'-methylenebis[2-chloro- |
| U222 | 636-21-5 | Benzenamine, 2-methyl-, hydrochloride |
| U181 | 99-55-8 | Benzenamine, 2-methyl-5-nitro- |
| U019 | 71-43-2 | Benzene (I,T) |
| U038 | 510-15-6 | Benzeneacetic acid, 4-chloro-alpha-(4-chlorophenyl)- alpha-hydroxy-, ethyl ester |
| U030 | 101-55-3 | Benzene, 1-bromo-4-phenoxy- |
| U035 | 305-03-3 | Benzenebutanoic acid, 4-[bis(2-chloroethyl)amino]- |
| U037 | 108-90-7 | Benzene, chloro- |
| U221 | 25376-45-8 | Benzenediamine, ar-methyl- |
| U028 | 117-81-7 | 1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester |
| U069 | 84-74-2 | 1,2-Benzenedicarboxylic acid, dibutyl ester |
| U088 | 84-66-2 | 1,2-Benzenedicarboxylic acid, diethyl ester |
| U102 | 131-11-3 | 1,2-Benzenedicarboxylic acid, dimethyl ester |
| U107 | 117-84-0 | 1,2-Benzenedicarboxylic acid, dioctyl ester |
| U070 | 95-50-1 | Benzene, 1,2-dichloro- |
| U071 | 541-73-1 | Benzene, 1,3-dichloro- |
| U072 | 106-46-7 | Benzene, 1,4-dichloro- |
| U060 | 72-54-8 | Benzene, 1,1'-(2,2-dichloroethylidene)bis[4-chloro- |
| U017 | 98-87-3 | Benzene, (dichloromethyl)- |
| U223 | 26471-62-5 | Benzene, 1,3-diisocyanatomethyl- (R,T) |
| U239 | 1330-20-7 | Benzene, dimethyl- (I) |
| U201 | 108-46-3 | 1,3-Benzenediol |
| U127 | 118-74-1 | Benzene, hexachloro- |
| U056 | 110-82-7 | Benzene, hexahydro- (I) |
| U220 | 108-88-3 | Benzene, methyl- |
| U105 | 121-14-2 | Benzene, 1-methyl-2,4-dinitro- |
| U106 | 606-20-2 | Benzene, 2-methyl-1,3-dinitro- |
| U055 | 98-82-8 | Benzene, (1-methylethyl)- (I) |
| U169 | 98-95-3 | Benzene, nitro- |
| U183 | 608-93-5 | Benzene, pentachloro- |



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| U185 | 82-68-8 | Benzene, pentachloronitro- |
| U020 | 98-09-9 | Benzenesulfonic acid chloride (C,R) |
| U020 | 98-09-9 | Benzenesulfonyl chloride (C,R) |
| U207 | 95-94-3 | Benzene, 1,2,4,5-tetrachloro- |
| U061 | 50-29-3 | Benzene, 1,1'-(2,2,2-trichloroethylidene)bis[4-chloro- |
| U247 | 72-43-5 | Benzene, 1,1'-(2,2,2-trichloroethylidene)bis[4-methoxy- |
| U023 | 98-07-7 | Benzene, (trichloromethyl)- |
| U234 | 99-35-4 | Benzene, 1,3,5-trinitro- |
| U021 | 92-87-5 | Benzidine |
| U278 | 22781-23-3 | 1,3-Benzodioxol-4-ol, 2,2-dimethyl-, methyl carbamate. |
| U364 | 22961-82-6 | 1,3-Benzodioxol-4-ol, 2,2-dimethyl-, |
| U203 | 94-59-7 | 1,3-Benzodioxole, 5-(2-propenyl)- |
| U141 | 120-58-1 | 1,3-Benzodioxole, 5-(1-propenyl)- |
| U367 | 1563-38-8 | 7-Benzofuranol, 2,3-dihydro-2,2-dimethyl- |
| U090 | 94-58-6 | 1,3-Benzodioxole, 5-propyl- |
| U064 | 189-55-9 | Benzo[rst]pentaphene |
| U248 | ¹ 81-81-2 | 2H-1-Benzopyran-2-one, 4-hydroxy-3-(3-oxo-1-phenyl-butyl)-, & salts, when present at concentrations of 0.3% or less |
| U022 | 50-32-8 | Benzo[a]pyrene |
| U197 | 106-51-4 | p-Benzoquinone |
| U023 | 98-07-7 | Benzotrichloride (C,R,T) |
| U085 | 1464-53-5 | 2,2'-Bioxirane |
| U021 | 92-87-5 | [1,1'-Biphenyl]-4,4'-diamine |
| U073 | 91-94-1 | [1,1'-Biphenyl]-4,4'-diamine, 3,3'-dichloro- |
| U091 | 119-90-4 | [1,1'-Biphenyl]-4,4'-diamine, 3,3'-dimethoxy- |
| U095 | 119-93-7 | [1,1'-Biphenyl]-4,4'-diamine, 3,3'-dimethyl- |
| U225 | 75-25-2 | Bromoform |
| U030 | 101-55-3 | 4-Bromophenyl phenyl ether |
| U128 | 87-68-3 | 1,3-Butadiene, 1,1,2,3,4,4-hexachloro- |
| U172 | 924-16-3 | 1-Butanamine, N-butyl-N-nitroso- |
| U031 | 71-36-3 | 1-Butanol (I) |
| U159 | 78-93-3 | 2-Butanone (I,T) |
| U160 | 1338-23-4 | 2-Butanone, peroxide (R,T) |



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| U053 | 4170-30-3 | 2-Butenal |
| U074 | 764-41-0 | 2-Butene, 1,4-dichloro- (I,T) |
| U143 | 303-34-4 | 2-Butenoic acid, 2-methyl-, 7-[[2,3-dihydroxy-2-(1-methoxyethyl)-3-methyl-1-oxobutoxy]methyl]-2,3,5,7a-tetrahydro-1H-pyrrolizin-1-yl ester, [1S-[1alpha(Z),7(2S*,3R*),7aalpha]]- |
| U031 | 71-36-3 | n-Butyl alcohol (I) |
| U136 | 75-60-5 | Cacodylic acid |
| U032 | 13765-19-0 | Calcium chromate |
| U372 | 10605-21-7 | Carbamic acid, 1H-benzimidazol-2-yl, methyl ester. |
| U271 | 17804-35-2 | Carbamic acid, [1-[(butylamino)carbonyl]-1H-benzimidazol-2-yl]-, methyl ester. |
| U280 | 101-27-9 | Carbamic acid, (3-chlorophenyl)-, 4-chloro-2-butynyl ester. |
| U238 | 51-79-6 | Carbamic acid, ethyl ester |
| U178 | 615-53-2 | Carbamic acid, methylnitroso-, ethyl ester |
| U373 | 122-42-9 | Carbamic acid, phenyl-, 1-methylethyl ester. |
| U409 | 23564-05-8 | Carbamic acid, [1,2-phenylenebis(iminocarbonothioyl)]bis-, dimethyl ester. |
| U097 | 79-44-7 | Carbamic chloride, dimethyl- |
| U389 | 2303-17-5 | Carbamothioic acid, bis(1-methylethyl)-, S-(2,3,3-trichloro-2-propenyl) ester. |
| U387 | 52888-80-9 | Carbamothioic acid, dipropyl-, S-(phenylmethyl) ester. |
| U114 | ¹ 111-54-6 | Carbamodithioic acid, 1,2-ethanediylbis-, salts & esters |
| U062 | 2303-16-4 | Carbamothioic acid, bis(1-methylethyl)-, S-(2,3-dichloro-2-propenyl) ester |
| U279 | 63-25-2 | Carbaryl. |
| U372 | 10605-21-7 | Carbendazim. |
| U367 | 1563-38-8 | Carbofuran phenol. |
| U215 | 6533-73-9 | Carbonic acid, dithallium(1 +) salt |
| U033 | 353-50-4 | Carbonic difluoride |
| U156 | 79-22-1 | Carbonochloridic acid, methyl ester (I,T) |
| U033 | 353-50-4 | Carbon oxyfluoride (R,T) |
| U211 | 56-23-5 | Carbon tetrachloride |
| U034 | 75-87-6 | Chloral |



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| U035 | 305-03-3 | Chlorambucil |
| U036 | 57-74-9 | Chlordane, alpha & gamma isomers |
| U026 | 494-03-1 | Chlornaphazin |
| U037 | 108-90-7 | Chlorobenzene |
| U038 | 510-15-6 | Chlorobenzilate |
| U039 | 59-50-7 | p-Chloro-m-cresol |
| U042 | 110-75-8 | 2-Chloroethyl vinyl ether |
| U044 | 67-66-3 | Chloroform |
| U046 | 107-30-2 | Chloromethyl methyl ether |
| U047 | 91-58-7 | beta-Chloronaphthalene |
| U048 | 95-57-8 | o-Chlorophenol |
| U049 | 3165-93-3 | 4-Chloro-o-toluidine, hydrochloride |
| U032 | 13765-19-0 | Chromic acid H ₂ CrO ₄ , calcium salt |
| U050 | 218-01-9 | Chrysene |
| U051 | | Creosote |
| U052 | 1319-77-3 | Cresol (Cresylic acid) |
| U053 | 4170-30-3 | Crotonaldehyde |
| U055 | 98-82-8 | Cumene (I) |
| U246 | 506-68-3 | Cyanogen bromide (CN)Br |
| U197 | 106-51-4 | 2,5-Cyclohexadiene-1,4-dione |
| U056 | 110-82-7 | Cyclohexane (I) |
| U129 | 58-89-9 | Cyclohexane, 1,2,3,4,5,6-hexachloro-, (1alpha,2alpha,3beta,4alpha,5alpha,6beta)- |
| U057 | 108-94-1 | Cyclohexanone (I) |
| U130 | 77-47-4 | 1,3-Cyclopentadiene, 1,2,3,4,5,5-hexachloro- |
| U058 | 50-18-0 | Cyclophosphamide |
| U240 | ¹ 94-75-7 | 2,4-D, salts & esters |
| U059 | 20830-81-3 | Daunomycin |
| U060 | 72-54-8 | DDD |
| U061 | 50-29-3 | DDT |
| U062 | 2303-16-4 | Diallate |
| U063 | 53-70-3 | Dibenz[a,h]anthracene |
| U064 | 189-55-9 | Dibenzo[a,i]pyrene |
| U066 | 96-12-8 | 1,2-Dibromo-3-chloropropane |
| U069 | 84-74-2 | Dibutyl phthalate |
| U070 | 95-50-1 | o-Dichlorobenzene |



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| U071 | 541-73-1 | m-Dichlorobenzene |
| U072 | 106-46-7 | p-Dichlorobenzene |
| U073 | 91-94-1 | 3,3'-Dichlorobenzidine |
| U074 | 764-41-0 | 1,4-Dichloro-2-butene (I,T) |
| U075 | 75-71-8 | Dichlorodifluoromethane |
| U078 | 75-35-4 | 1,1-Dichloroethylene |
| U079 | 156-60-5 | 1,2-Dichloroethylene |
| U025 | 111-44-4 | Dichloroethyl ether |
| U027 | 108-60-1 | Dichloroisopropyl ether |
| U024 | 111-91-1 | Dichloromethoxy ethane |
| U081 | 120-83-2 | 2,4-Dichlorophenol |
| U082 | 87-65-0 | 2,6-Dichlorophenol |
| U084 | 542-75-6 | 1,3-Dichloropropene |
| U085 | 1464-53-5 | 1,2:3,4-Diepoxybutane (I,T) |
| U108 | 123-91-1 | 1,4-Diethyleneoxide |
| U028 | 117-81-7 | Diethylhexyl phthalate |
| U395 | 5952-26-1 | Diethylene glycol, dicarbamate. |
| U086 | 1615-80-1 | N,N'-Diethylhydrazine |
| U087 | 3288-58-2 | O,O-Diethyl S-methyl dithiophosphate |
| U088 | 84-66-2 | Diethyl phthalate |
| U089 | 56-53-1 | Diethylstilbesterol |
| U090 | 94-58-6 | Dihydrosafrole |
| U091 | 119-90-4 | 3,3'-Dimethoxybenzidine |
| U092 | 124-40-3 | Dimethylamine (I) |
| U093 | 60-11-7 | p-Dimethylaminoazobenzene |
| U094 | 57-97-6 | 7,12-Dimethylbenz[a]anthracene |
| U095 | 119-93-7 | 3,3'-Dimethylbenzidine |
| U096 | 80-15-9 | alpha,alpha-Dimethylbenzylhydroperoxide (R) |
| U097 | 79-44-7 | Dimethylcarbamoyl chloride |
| U098 | 57-14-7 | 1,1-Dimethylhydrazine |
| U099 | 540-73-8 | 1,2-Dimethylhydrazine |
| U101 | 105-67-9 | 2,4-Dimethylphenol |
| U102 | 131-11-3 | Dimethyl phthalate |
| U103 | 77-78-1 | Dimethyl sulfate |
| U105 | 121-14-2 | 2,4-Dinitrotoluene |
| U106 | 606-20-2 | 2,6-Dinitrotoluene |



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| U107 | 117-84-0 | Di-n-octyl phthalate |
| U108 | 123-91-1 | 1,4-Dioxane |
| U109 | 122-66-7 | 1,2-Diphenylhydrazine |
| U110 | 142-84-7 | Dipropylamine (I) |
| U111 | 621-64-7 | Di-n-propylnitrosamine |
| U041 | 106-89-8 | Epichlorohydrin |
| U001 | 75-07-0 | Ethanal (I) |
| U404 | 121-44-8 | Ethanamine, N,N-diethyl- |
| U174 | 55-18-5 | Ethanamine, N-ethyl-N-nitroso- |
| U155 | 91-80-5 | 1,2-Ethanediamine, N,N-dimethyl-N'-2-pyridinyl-N'-(2-thienylmethyl)- |
| U067 | 106-93-4 | Ethane, 1,2-dibromo- |
| U076 | 75-34-3 | Ethane, 1,1-dichloro- |
| U077 | 107-06-2 | Ethane, 1,2-dichloro- |
| U131 | 67-72-1 | Ethane, hexachloro- |
| U024 | 111-91-1 | Ethane, 1,1'-[methylenebis(oxy)]bis[2-chloro- |
| U117 | 60-29-7 | Ethane, 1,1'-oxybis-(I) |
| U025 | 111-44-4 | Ethane, 1,1'-oxybis[2-chloro- |
| U184 | 76-01-7 | Ethane, pentachloro- |
| U208 | 630-20-6 | Ethane, 1,1,1,2-tetrachloro- |
| U209 | 79-34-5 | Ethane, 1,1,2,2-tetrachloro- |
| U218 | 62-55-5 | Ethanethioamide |
| U226 | 71-55-6 | Ethane, 1,1,1-trichloro- |
| U227 | 79-00-5 | Ethane, 1,1,2-trichloro- |
| U410 | 59669-26-0 | Ethanimidothioic acid, N,N'-[thiobis[(methylimino)carbonyloxy]]bis-, dimethyl ester |
| U394 | 30558-43-1 | Ethanimidothioic acid, 2-(dimethylamino)-N-hydroxy-2-oxo-, methyl ester. |
| U359 | 110-80-5 | Ethanol, 2-ethoxy- |
| U173 | 1116-54-7 | Ethanol, 2,2'-(nitrosoimino)bis- |
| U395 | 5952-26-1 | Ethanol, 2,2'-oxybis-, dicarbamate. |
| U004 | 98-86-2 | Ethanone, 1-phenyl- |
| U043 | 75-01-4 | Ethene, chloro- |
| U042 | 110-75-8 | Ethene, (2-chloroethoxy)- |
| U078 | 75-35-4 | Ethene, 1,1-dichloro- |
| U079 | 156-60-5 | Ethene, 1,2-dichloro-, (E)- |



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| U210 | 127-18-4 | Ethene, tetrachloro- |
| U228 | 79-01-6 | Ethene, trichloro- |
| U112 | 141-78-6 | Ethyl acetate (I) |
| U113 | 140-88-5 | Ethyl acrylate (I) |
| U238 | 51-79-6 | Ethyl carbamate (urethane) |
| U117 | 60-29-7 | Ethyl ether (I) |
| U114 | ¹ 111-54-6 | Ethylenebisdithiocarbamic acid, salts & esters |
| U067 | 106-93-4 | Ethylene dibromide |
| U077 | 107-06-2 | Ethylene dichloride |
| U359 | 110-80-5 | Ethylene glycol monoethyl ether |
| U115 | 75-21-8 | Ethylene oxide (I,T) |
| U116 | 96-45-7 | Ethylenethiourea |
| U076 | 75-34-3 | Ethylidene dichloride |
| U118 | 97-63-2 | Ethyl methacrylate |
| U119 | 62-50-0 | Ethyl methanesulfonate |
| U120 | 206-44-0 | Fluoranthene |
| U122 | 50-00-0 | Formaldehyde |
| U123 | 64-18-6 | Formic acid (C,T) |
| U124 | 110-00-9 | Furan (I) |
| U125 | 98-01-1 | 2-Furancarboxaldehyde (I) |
| U147 | 108-31-6 | 2,5-Furandione |
| U213 | 109-99-9 | Furan, tetrahydro-(I) |
| U125 | 98-01-1 | Furfural (I) |
| U124 | 110-00-9 | Furfuran (I) |
| U206 | 18883-66-4 | Glucopyranose, 2-deoxy-2-(3-methyl-3-nitrosoureido)-, D- |
| U206 | 18883-66-4 | D-Glucose, 2-deoxy-2-[[methylnitrosoamino)-carbonyl]amino]- |
| U126 | 765-34-4 | Glycidylaldehyde |
| U163 | 70-25-7 | Guanidine, N-methyl-N'-nitro-N-nitroso- |
| U127 | 118-74-1 | Hexachlorobenzene |
| U128 | 87-68-3 | Hexachlorobutadiene |
| U130 | 77-47-4 | Hexachlorocyclopentadiene |
| U131 | 67-72-1 | Hexachloroethane |
| U132 | 70-30-4 | Hexachlorophene |
| U243 | 1888-71-7 | Hexachloropropene |



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| U133 | 302-01-2 | Hydrazine (R,T) |
| U086 | 1615-80-1 | Hydrazine, 1,2-diethyl- |
| U098 | 57-14-7 | Hydrazine, 1,1-dimethyl- |
| U099 | 540-73-8 | Hydrazine, 1,2-dimethyl- |
| U109 | 122-66-7 | Hydrazine, 1,2-diphenyl- |
| U134 | 7664-39-3 | Hydrofluoric acid (C,T) |
| U134 | 7664-39-3 | Hydrogen fluoride (C,T) |
| U135 | 7783-06-4 | Hydrogen sulfide |
| U135 | 7783-06-4 | Hydrogen sulfide H ₂ S |
| U096 | 80-15-9 | Hydroperoxide, 1-methyl-1-phenylethyl- (R) |
| U116 | 96-45-7 | 2-Imidazolidinethione |
| U137 | 193-39-5 | Indeno[1,2,3-cd]pyrene |
| U190 | 85-44-9 | 1,3-Isobenzofurandione |
| U140 | 78-83-1 | Isobutyl alcohol (I,T) |
| U141 | 120-58-1 | Isosafrole |
| U142 | 143-50-0 | Kepone |
| U143 | 303-34-4 | Lasiocarpine |
| U144 | 301-04-2 | Lead acetate |
| U146 | 1335-32-6 | Lead, bis(acetato-O)tetrahydroxytri- |
| U145 | 7446-27-7 | Lead phosphate |
| U146 | 1335-32-6 | Lead subacetate |
| U129 | 58-89-9 | Lindane |
| U163 | 70-25-7 | MNNG |
| U147 | 108-31-6 | Maleic anhydride |
| U148 | 123-33-1 | Maleic hydrazide |
| U149 | 109-77-3 | Malononitrile |
| U150 | 148-82-3 | Melphalan |
| U151 | 7439-97-6 | Mercury |
| U152 | 126-98-7 | Methacrylonitrile (I, T) |
| U092 | 124-40-3 | Methanamine, N-methyl- (I) |
| U029 | 74-83-9 | Methane, bromo- |
| U045 | 74-87-3 | Methane, chloro- (I, T) |
| U046 | 107-30-2 | Methane, chloromethoxy- |
| U068 | 74-95-3 | Methane, dibromo- |
| U080 | 75-09-2 | Methane, dichloro- |
| U075 | 75-71-8 | Methane, dichlorodifluoro- |



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| U138 | 74-88-4 | Methane, iodo- |
| U119 | 62-50-0 | Methanesulfonic acid, ethyl ester |
| U211 | 56-23-5 | Methane, tetrachloro- |
| U153 | 74-93-1 | Methanethiol (I, T) |
| U225 | 75-25-2 | Methane, tribromo- |
| U044 | 67-66-3 | Methane, trichloro- |
| U121 | 75-69-4 | Methane, trichlorofluoro- |
| U036 | 57-74-9 | 4,7-Methano-1H-indene, 1,2,4,5,6,7,8,8-octachloro-2,3,3a,4,7,7a-hexahydro- |
| U154 | 67-56-1 | Methanol (I) |
| U155 | 91-80-5 | Methapyrilene |
| U142 | 143-50-0 | 1,3,4-Metheno-2H-cyclobuta[cd]pentalen-2-one, 1,1a,3,3a,4,5,5,5a,5b,6-decachlorooctahydro- |
| U247 | 72-43-5 | Methoxychlor |
| U154 | 67-56-1 | Methyl alcohol (I) |
| U029 | 74-83-9 | Methyl bromide |
| U186 | 504-60-9 | 1-Methylbutadiene (I) |
| U045 | 74-87-3 | Methyl chloride (I,T) |
| U156 | 79-22-1 | Methyl chlorocarbonate (I,T) |
| U226 | 71-55-6 | Methyl chloroform |
| U157 | 56-49-5 | 3-Methylcholanthrene |
| U158 | 101-14-4 | 4,4'-Methylenebis(2-chloroaniline) |
| U068 | 74-95-3 | Methylene bromide |
| U080 | 75-09-2 | Methylene chloride |
| U159 | 78-93-3 | Methyl ethyl ketone (MEK) (I,T) |
| U160 | 1338-23-4 | Methyl ethyl ketone peroxide (R,T) |
| U138 | 74-88-4 | Methyl iodide |
| U161 | 108-10-1 | Methyl isobutyl ketone (I) |
| U162 | 80-62-6 | Methyl methacrylate (I,T) |
| U161 | 108-10-1 | 4-Methyl-2-pentanone (I) |
| U164 | 56-04-2 | Methylthiouracil |
| U010 | 50-07-7 | Mitomycin C |
| U059 | 20830-81-3 | 5,12-Naphthacenedione, 8-acetyl-10-[(3-amino-2,3,6-trideoxy)-alpha-L-lyxo-hexopyranosyl]oxy]-7,8,9,10-tetrahydro-6,8,11-trihydroxy-1-methoxy-, (8S-cis)- |
| U167 | 134-32-7 | 1-Naphthalenamine |



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| U168 | 91-59-8 | 2-Naphthalenamine |
| U026 | 494-03-1 | Naphthalenamine, N,N'-bis(2-chloroethyl)- |
| U165 | 91-20-3 | Naphthalene |
| U047 | 91-58-7 | Naphthalene, 2-chloro- |
| U166 | 130-15-4 | 1,4-Naphthalenedione |
| U236 | 72-57-1 | 2,7-Naphthalenedisulfonic acid, 3,3'-[(3,3'-dimethyl[1,1'-biphenyl]-4,4'-diyl)bis(azo)bis[5-amino-4-hydroxy]-, tetrasodium salt |
| U279 | 63-25-2 | 1-Naphthalenol, methylcarbamate. |
| U166 | 130-15-4 | 1,4-Naphthoquinone |
| U167 | 134-32-7 | alpha-Naphthylamine |
| U168 | 91-59-8 | beta-Naphthylamine |
| U217 | 10102-45-1 | Nitric acid, thallium(1 +) salt |
| U169 | 98-95-3 | Nitrobenzene (I,T) |
| U170 | 100-02-7 | p-Nitrophenol |
| U171 | 79-46-9 | 2-Nitropropane (I,T) |
| U172 | 924-16-3 | N-Nitrosodi-n-butylamine |
| U173 | 1116-54-7 | N-Nitrosodiethanolamine |
| U174 | 55-18-5 | N-Nitrosodiethylamine |
| U176 | 759-73-9 | N-Nitroso-N-ethylurea |
| U177 | 684-93-5 | N-Nitroso-N-methylurea |
| U178 | 615-53-2 | N-Nitroso-N-methylurethane |
| U179 | 100-75-4 | N-Nitrosopiperidine |
| U180 | 930-55-2 | N-Nitrosopyrrolidine |
| U181 | 99-55-8 | 5-Nitro-o-toluidine |
| U193 | 1120-71-4 | 1,2-Oxathiolane, 2,2-dioxide |
| U058 | 50-18-0 | 2H-1,3,2-Oxazaphosphorin-2-amine, N,N-bis(2-chloroethyl)tetrahydro-, 2-oxide |
| U115 | 75-21-8 | Oxirane (I,T) |
| U126 | 765-34-4 | Oxiranecarboxyaldehyde |
| U041 | 106-89-8 | Oxirane, (chloromethyl)- |
| | U182 | 123-63-7 Paraldehyde |
| U183 | 608-93-5 | Pentachlorobenzene |
| U184 | 76-01-7 | Pentachloroethane |
| U185 | 82-68-8 | Pentachloronitrobenzene (PCNB) |
| See F027 | 87-86-5 | Pentachlorophenol |



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| U161 | 108-10-1 | Pentanol, 4-methyl- |
| U186 | 504-60-9 | 1,3-Pentadiene (I) |
| U187 | 62-44-2 | Phenacetin |
| U188 | 108-95-2 | Phenol |
| U048 | 95-57-8 | Phenol, 2-chloro- |
| U039 | 59-50-7 | Phenol, 4-chloro-3-methyl- |
| U081 | 120-83-2 | Phenol, 2,4-dichloro- |
| U082 | 87-65-0 | Phenol, 2,6-dichloro- |
| U089 | 56-53-1 | Phenol, 4,4'-(1,2-diethyl-1,2-ethenediyl)bis-, (E)- |
| U101 | 105-67-9 | Phenol, 2,4-dimethyl- |
| U052 | 1319-77-3 | Phenol, methyl- |
| U132 | 70-30-4 | Phenol, 2,2'-methylenebis[3,4,6-trichloro- |
| U411 | 114-26-1 | Phenol, 2-(1-methylethoxy)-, methylcarbamate. |
| U170 | 100-02-7 | Phenol, 4-nitro- |
| See F027 | 87-86-5 | Phenol, pentachloro- |
| See F027 | 58-90-2 | Phenol, 2,3,4,6-tetrachloro- |
| See F027 | 95-95-4 | Phenol, 2,4,5-trichloro- |
| See F027 | 88-06-2 | Phenol, 2,4,6-trichloro- |
| U150 | 148-82-3 | L-Phenylalanine, 4-[bis(2-chloroethyl)amino]- |
| U145 | 7446-27-7 | Phosphoric acid, lead(2 +) salt (2:3) |
| U087 | 3288-58-2 | Phosphorodithioic acid, O,O-diethyl S-methyl ester |
| U189 | 1314-80-3 | Phosphorus sulfide (R) |
| U190 | 85-44-9 | Phthalic anhydride |
| U191 | 109-06-8 | 2-Picoline |
| U179 | 100-75-4 | Piperidine, 1-nitroso- |
| U192 | 23950-58-5 | Pronamide |
| U194 | 107-10-8 | 1-Propanamine (I,T) |
| U111 | 621-64-7 | 1-Propanamine, N-nitroso-N-propyl- |
| U110 | 142-84-7 | 1-Propanamine, N-propyl- (I) |
| U066 | 96-12-8 | Propane, 1,2-dibromo-3-chloro- |
| U083 | 78-87-5 | Propane, 1,2-dichloro- |
| U149 | 109-77-3 | Propanedinitrile |
| U171 | 79-46-9 | Propane, 2-nitro- (I,T) |
| U027 | 108-60-1 | Propane, 2,2'-oxybis[2-chloro- |
| U193 | 1120-71-4 | 1,3-Propane sultone |
| See F027 | 93-72-1 | Propanoic acid, 2-(2,4,5-trichlorophenoxy)- |



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| U235 | 126-72-7 | 1-Propanol, 2,3-dibromo-, phosphate (3:1) |
| U140 | 78-83-1 | 1-Propanol, 2-methyl- (I,T) |
| U002 | 67-64-1 | 2-Propanone (I) |
| U007 | 79-06-1 | 2-Propenamide |
| U084 | 542-75-6 | 1-Propene, 1,3-dichloro- |
| U243 | 1888-71-7 | 1-Propene, 1,1,2,3,3,3-hexachloro- |
| U009 | 107-13-1 | 2-Propenenitrile |
| U152 | 126-98-7 | 2-Propenenitrile, 2-methyl- (I,T) |
| U008 | 79-10-7 | 2-Propenoic acid (I) |
| U113 | 140-88-5 | 2-Propenoic acid, ethyl ester (I) |
| U118 | 97-63-2 | 2-Propenoic acid, 2-methyl-, ethyl ester |
| U162 | 80-62-6 | 2-Propenoic acid, 2-methyl-, methyl ester (I,T) |
| U373 | 122-42-9 | Propham. |
| U411 | 114-26-1 | Propoxur. |
| U387 | 52888-80-9 | Prosulfocarb. |
| U194 | 107-10-8 | n-Propylamine (I,T) |
| U083 | 78-87-5 | Propylene dichloride |
| U148 | 123-33-1 | 3,6-Pyridazinedione, 1,2-dihydro- |
| U196 | 110-86-1 | Pyridine |
| U191 | 109-06-8 | Pyridine, 2-methyl- |
| U237 | 66-75-1 | 2,4-(1H,3H)-Pyrimidinedione, 5-[bis(2-chloroethyl)amino]- |
| U164 | 56-04-2 | 4(1H)-Pyrimidinone, 2,3-dihydro-6-methyl-2-thioxo- |
| U180 | 930-55-2 | Pyrrolidine, 1-nitroso- |
| U200 | 50-55-5 | Reserpine |
| U201 | 108-46-3 | Resorcinol |
| U203 | 94-59-7 | Safrole |
| U204 | 7783-00-8 | Selenious acid |
| U204 | 7783-00-8 | Selenium dioxide |
| U205 | 7488-56-4 | Selenium sulfide |
| U205 | 7488-56-4 | Selenium sulfide SeS ₂ (R,T) |
| U015 | 115-02-6 | L-Serine, diazoacetate (ester) |
| See F027 | 93-72-1 | Silvex (2,4,5-TP) |
| U206 | 18883-66-4 | Streptozotocin |
| U103 | 77-78-1 | Sulfuric acid, dimethyl ester |
| U189 | 1314-80-3 | Sulfur phosphide (R) |



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| See F027 | 93-76-5 | 2,4,5-T |
| U207 | 95-94-3 | 1,2,4,5-Tetrachlorobenzene |
| U208 | 630-20-6 | 1,1,1,2-Tetrachloroethane |
| U209 | 79-34-5 | 1,1,2,2-Tetrachloroethane |
| U210 | 127-18-4 | Tetrachloroethylene |
| See F027 | 58-90-2 | 2,3,4,6-Tetrachlorophenol |
| U213 | 109-99-9 | Tetrahydrofuran (I) |
| U214 | 563-68-8 | Thallium(I) acetate |
| U215 | 6533-73-9 | Thallium(I) carbonate |
| U216 | 7791-12-0 | Thallium(I) chloride |
| U216 | 7791-12-0 | thallium chloride TlCl |
| U217 | 10102-45-1 | Thallium(I) nitrate |
| U218 | 62-55-5 | Thioacetamide |
| U410 | 59669-26-0 | Thiodicarb. |
| U153 | 74-93-1 | Thiomethanol (I,T) |
| U244 | 137-26-8 | Thioperoxydicarbonic diamide [(H ₂ N)C(S)] ₂ S ₂ , tetramethyl- |
| U409 | 23564-05-8 | Thiophanate-methyl. |
| U219 | 62-56-6 | Thiourea |
| U244 | 137-26-8 | Thiram |
| U220 | 108-88-3 | Toluene |
| U221 | 25376-45-8 | Toluenediamine |
| U223 | 26471-62-5 | Toluene diisocyanate (R,T) |
| U328 | 95-53-4 | o-Toluidine |
| U353 | 106-49-0 | p-Toluidine |
| U222 | 636-21-5 | o-Toluidine hydrochloride |
| U389 | 2303-17-5 | Triallate. |
| U011 | 61-82-5 | 1H-1,2,4-Triazol-3-amine |
| U226 | 71-55-6 | 1,1,1-Trichloroethane |
| U227 | 79-00-5 | 1,1,2-Trichloroethane |
| U228 | 79-01-6 | Trichloroethylene |
| U121 | 75-69-4 | Trichloromonofluoromethane |
| See F027 | 95-95-4 | 2,4,5-Trichlorophenol |
| See F027 | 88-06-2 | 2,4,6-Trichlorophenol |
| U404 | 121-44-8 | Triethylamine. |
| U234 | 99-35-4 | 1,3,5-Trinitrobenzene (R,T) |



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| U182 | 123-63-7 | 1,3,5-Trioxane, 2,4,6-trimethyl- |
| U235 | 126-72-7 | Tris(2,3-dibromopropyl) phosphate |
| U236 | 72-57-1 | Trypan blue |
| U237 | 66-75-1 | Uracil mustard |
| U176 | 759-73-9 | Urea, N-ethyl-N-nitroso- |
| U177 | 684-93-5 | Urea, N-methyl-N-nitroso- |
| U043 | 75-01-4 | Vinyl chloride |
| U248 | 181-81-2 | Warfarin, & salts, when present at concentrations of 0.3% or less |
| U239 | 1330-20-7 | Xylene (I) |
| U200 | 50-55-5 | Yohimban-16-carboxylic acid, 11,17-dimethoxy-18-[(3,4,5-trimethoxybenzoyl)oxy]-, methyl ester, (3beta,16beta,17alpha,18beta,20alpha)- |
| U249 | 1314-84-7 | Zinc phosphide Zn ₃ P ₂ , when present at concentrations of 10% or less |
| U001 | 75-07-0 | Acetaldehyde (I) |
| U001 | 75-07-0 | Ethanal (I) |
| U002 | 67-64-1 | Acetone (I) |
| U002 | 67-64-1 | 2-Propanone (I) |
| U003 | 75-05-8 | Acetonitrile (I,T) |
| U004 | 98-86-2 | Acetophenone |
| U004 | 98-86-2 | Ethanone, 1-phenyl- |
| U005 | 53-96-3 | Acetamide, -9H-fluoren-2-yl- |
| U005 | 53-96-3 | 2-Acetylaminofluorene |
| U006 | 75-36-5 | Acetyl chloride (C,R,T) |
| U007 | 79-06-1 | Acrylamide |
| U007 | 79-06-1 | 2-Propenamamide |
| U008 | 79-10-7 | Acrylic acid (I) |
| U008 | 79-10-7 | 2-Propenoic acid (I) |
| U009 | 107-13-1 | Acrylonitrile |
| U009 | 107-13-1 | 2-Propenenitrile |
| U010 | 50-07-7 | Azirino[2',3':3,4]pyrrolo[1,2-a]indole-4,7-dione, 6-amino-8-[[aminocarbonyl]oxy]methyl]-1,1a,2,8,8a,8b-hexahydro-8a-methoxy-5-methyl-, [1aS-(1aalpha, 8beta,8aalpha,8balpha)]- |
| U010 | 50-07-7 | Mitomycin C |
| U011 | 61-82-5 | Amitrole |



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| U011 | 61-82-5 | 1H-1,2,4-Triazol-3-amine |
| U012 | 62-53-3 | Aniline (I,T) |
| U012 | 62-53-3 | Benzenamine (I,T) |
| U014 | 492-80-8 | Auramine |
| U014 | 492-80-8 | Benzenamine, 4,4'-carbonimidoylbis[N,N-dimethyl- |
| U015 | 115-02-6 | Azaserine |
| U015 | 115-02-6 | L-Serine, diazoacetate (ester) |
| U016 | 225-51-4 | Benz[c]acridine |
| U017 | 98-87-3 | Benzal chloride |
| U017 | 98-87-3 | Benzene, (dichloromethyl)- |
| U018 | 56-55-3 | Benz[a]anthracene |
| U019 | 71-43-2 | Benzene (I,T) |
| U020 | 98-09-9 | Benzenesulfonic acid chloride (C,R) |
| U020 | 98-09-9 | Benzenesulfonyl chloride (C,R) |
| U021 | 92-87-5 | Benzidine |
| U021 | 92-87-5 | [1,1'-Biphenyl]-4,4'-diamine |
| U022 | 50-32-8 | Benzo[a]pyrene |
| U023 | 98-07-7 | Benzene, (trichloromethyl)- |
| U023 | 98-07-7 | Benzotrichloride (C,R,T) |
| U024 | 111-91-1 | Dichloromethoxy ethane |
| U024 | 111-91-1 | Ethane, 1,1'-[methylenebis(oxy)]bis[2-chloro- |
| U025 | 111-44-4 | Dichloroethyl ether |
| U025 | 111-44-4 | Ethane, 1,1'-oxybis[2-chloro- |
| U026 | 494-03-1 | Chlornaphazin |
| U026 | 494-03-1 | Naphthalenamine, N,N'-bis(2-chloroethyl)- |
| U027 | 108-60-1 | Dichloroisopropyl ether |
| U027 | 108-60-1 | Propane, 2,2'-oxybis[2-chloro- |
| U028 | 117-81-7 | 1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester |
| U028 | 117-81-7 | Diethylhexyl phthalate |
| U029 | 74-83-9 | Methane, bromo- |
| U029 | 74-83-9 | Methyl bromide |
| U030 | 101-55-3 | Benzene, 1-bromo-4-phenoxy- |
| U030 | 101-55-3 | 4-Bromophenyl phenyl ether |
| U031 | 71-36-3 | 1-Butanol (I) |
| U031 | 71-36-3 | n-Butyl alcohol (I) |
| U032 | 13765-19-0 | Calcium chromate |



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| U032 | 13765-19-0 | Chromic acid H ₂ CrO ₄ , calcium salt |
| U033 | 353-50-4 | Carbonic difluoride |
| U033 | 353-50-4 | Carbon oxyfluoride (R,T) |
| U034 | 75-87-6 | Acetaldehyde, trichloro- |
| U034 | 75-87-6 | Chloral |
| U035 | 305-03-3 | Benzenebutanoic acid, 4-[bis(2-chloroethyl)amino]- |
| U035 | 305-03-3 | Chlorambucil |
| U036 | 57-74-9 | Chlordane, alpha & gamma isomers |
| U036 | 57-74-9 | 4,7-Methano-1H-indene, 1,2,4,5,6,7,8,8-octachloro-2,3,3a,4,7,7a-hexahydro- |
| U037 | 108-90-7 | Benzene, chloro- |
| U037 | 108-90-7 | Chlorobenzene |
| U038 | 510-15-6 | Benzeneacetic acid, 4-chloro-alpha-(4-chlorophenyl)-alpha-hydroxy-, ethyl ester |
| U038 | 510-15-6 | Chlorobenzilate |
| U039 | 59-50-7 | p-Chloro-m-cresol |
| U039 | 59-50-7 | Phenol, 4-chloro-3-methyl- |
| U041 | 106-89-8 | Epichlorohydrin |
| U041 | 106-89-8 | Oxirane, (chloromethyl)- |
| U042 | 110-75-8 | 2-Chloroethyl vinyl ether |
| U042 | 110-75-8 | Ethene, (2-chloroethoxy)- |
| U043 | 75-01-4 | Ethene, chloro- |
| U043 | 75-01-4 | Vinyl chloride |
| U044 | 67-66-3 | Chloroform |
| U044 | 67-66-3 | Methane, trichloro- |
| U045 | 74-87-3 | Methane, chloro- (I,T) |
| U045 | 74-87-3 | Methyl chloride (I,T) |
| U046 | 107-30-2 | Chloromethyl methyl ether |
| U046 | 107-30-2 | Methane, chloromethoxy- |
| U047 | 91-58-7 | beta-Chloronaphthalene |
| U047 | 91-58-7 | Naphthalene, 2-chloro- |
| U048 | 95-57-8 | o-Chlorophenol |
| U048 | 95-57-8 | Phenol, 2-chloro- |
| U049 | 3165-93-3 | Benzenamine, 4-chloro-2-methyl-, hydrochloride |
| U049 | 3165-93-3 | 4-Chloro-o-toluidine, hydrochloride |
| U050 | 218-01-9 | Chrysene |



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| U051 | | Creosote |
| U052 | 1319-77-3 | Cresol (Cresylic acid) |
| U052 | 1319-77-3 | Phenol, methyl- |
| U053 | 4170-30-3 | 2-Butenal |
| U053 | 4170-30-3 | Crotonaldehyde |
| U055 | 98-82-8 | Benzene, (1-methylethyl)-(I) |
| U055 | 98-82-8 | Cumene (I) |
| U056 | 110-82-7 | Benzene, hexahydro-(I) |
| U056 | 110-82-7 | Cyclohexane (I) |
| U057 | 108-94-1 | Cyclohexanone (I) |
| U058 | 50-18-0 | Cyclophosphamide |
| U058 | 50-18-0 | 2H-1,3,2-Oxazaphosphorin-2-amine, N,N-bis(2-chloroethyl)tetrahydro-, 2-oxide |
| U059 | 20830-81-3 | Daunomycin |
| U059 | 20830-81-3 | 5,12-Naphthacenedione, 8-acetyl-10-[(3-amino-2,3,6-trideoxy)-alpha-L-lyxo-hexopyranosyl]oxy]-7,8,9,10-tetrahydro-6,8,11-trihydroxy-1-methoxy-, (8S-cis)- |
| U060 | 72-54-8 | Benzene, 1,1'-(2,2-dichloroethylidene)bis[4-chloro- |
| U060 | 72-54-8 | DDD |
| U061 | 50-29-3 | Benzene, 1,1'-(2,2,2-trichloroethylidene)bis[4-chloro- |
| U061 | 50-29-3 | DDT |
| U062 | 2303-16-4 | Carbamothioic acid, bis(1-methylethyl)-, S-(2,3-dichloro-2-propenyl) ester |
| U062 | 2303-16-4 | Diallate |
| U063 | 53-70-3 | Dibenz[a,h]anthracene |
| U064 | 189-55-9 | Benzo[rs]pentaphene |
| U064 | 189-55-9 | Dibenzo[a,i]pyrene |
| U066 | 96-12-8 | 1,2-Dibromo-3-chloropropane |
| U066 | 96-12-8 | Propane, 1,2-dibromo-3-chloro- |
| U067 | 106-93-4 | Ethane, 1,2-dibromo- |
| U067 | 106-93-4 | Ethylene dibromide |
| U068 | 74-95-3 | Methane, dibromo- |
| U068 | 74-95-3 | Methylene bromide |
| U069 | 84-74-2 | 1,2-Benzenedicarboxylic acid, dibutyl ester |
| U069 | 84-74-2 | Dibutyl phthalate |



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| U070 | 95-50-1 | Benzene, 1,2-dichloro- |
| U070 | 95-50-1 | o-Dichlorobenzene |
| U071 | 541-73-1 | Benzene, 1,3-dichloro- |
| U071 | 541-73-1 | m-Dichlorobenzene |
| U072 | 106-46-7 | Benzene, 1,4-dichloro- |
| U072 | 106-46-7 | p-Dichlorobenzene |
| U073 | 91-94-1 | [1,1'-Biphenyl]-4,4'-diamine, 3,3'-dichloro- |
| U073 | 91-94-1 | 3,3'-Dichlorobenzidine |
| U074 | 764-41-0 | 2-Butene, 1,4-dichloro-(I,T) |
| U074 | 764-41-0 | 1,4-Dichloro-2-butene (I,T) |
| U075 | 75-71-8 | Dichlorodifluoromethane |
| U075 | 75-71-8 | Methane, dichlorodifluoro- |
| U076 | 75-34-3 | Ethane, 1,1-dichloro- |
| U076 | 75-34-3 | Ethylidene dichloride |
| U077 | 107-06-2 | Ethane, 1,2-dichloro- |
| U077 | 107-06-2 | Ethylene dichloride |
| U078 | 75-35-4 | 1,1-Dichloroethylene |
| U078 | 75-35-4 | Ethene, 1,1-dichloro- |
| U079 | 156-60-5 | 1,2-Dichloroethylene |
| U079 | 156-60-5 | Ethene, 1,2-dichloro-, (E)- |
| U080 | 75-09-2 | Methane, dichloro- |
| U080 | 75-09-2 | Methylene chloride |
| U081 | 120-83-2 | 2,4-Dichlorophenol |
| U081 | 120-83-2 | Phenol, 2,4-dichloro- |
| U082 | 87-65-0 | 2,6-Dichlorophenol |
| U082 | 87-65-0 | Phenol, 2,6-dichloro- |
| U083 | 78-87-5 | Propane, 1,2-dichloro- |
| U083 | 78-87-5 | Propylene dichloride |
| U084 | 542-75-6 | 1,3-Dichloropropene |
| U084 | 542-75-6 | 1-Propene, 1,3-dichloro- |
| U085 | 1464-53-5 | 2,2'-Bioxirane |
| U085 | 1464-53-5 | 1,2:3,4-Diepoxybutane (I,T) |
| U086 | 1615-80-1 | N,N'-Diethylhydrazine |
| U086 | 1615-80-1 | Hydrazine, 1,2-diethyl- |
| U087 | 3288-58-2 | O,O-Diethyl S-methyl dithiophosphate |
| U087 | 3288-58-2 | Phosphorodithioic acid, O,O-diethyl S-methyl ester |



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| U088 | 84-66-2 | 1,2-Benzenedicarboxylic acid, diethyl ester |
| U088 | 84-66-2 | Diethyl phthalate |
| U089 | 56-53-1 | Diethylstilbesterol |
| U089 | 56-53-1 | Phenol, 4,4'-(1,2-diethyl-1,2-ethenediyl)bis-, (E)- |
| U090 | 94-58-6 | 1,3-Benzodioxole, 5-propyl- |
| U090 | 94-58-6 | Dihydrosafrole |
| U091 | 119-90-4 | [1,1'-Biphenyl]-4,4'-diamine, 3,3'-dimethoxy- |
| U091 | 119-90-4 | 3,3'-Dimethoxybenzidine |
| U092 | 124-40-3 | Dimethylamine (I) |
| U092 | 124-40-3 | Methanamine, -methyl-(I) |
| U093 | 60-11-7 | Benzenamine, N,N-dimethyl-4-(phenylazo)- |
| U093 | 60-11-7 | p-Dimethylaminoazobenzene |
| U094 | 57-97-6 | Benz[a]anthracene, 7,12-dimethyl- |
| U094 | 57-97-6 | 7,12-Dimethylbenz[a]anthracene |
| U095 | 119-93-7 | [1,1'-Biphenyl]-4,4'-diamine, 3,3'-dimethyl- |
| U095 | 119-93-7 | 3,3'-Dimethylbenzidine |
| U096 | 80-15-9 | alpha,alpha-Dimethylbenzylhydroperoxide (R) |
| U096 | 80-15-9 | Hydroperoxide, 1-methyl-1-phenylethyl-(R) |
| U097 | 79-44-7 | Carbamic chloride, dimethyl- |
| U097 | 79-44-7 | Dimethylcarbamoyl chloride |
| U098 | 57-14-7 | 1,1-Dimethylhydrazine |
| U098 | 57-14-7 | Hydrazine, 1,1-dimethyl- |
| U099 | 540-73-8 | 1,2-Dimethylhydrazine |
| U099 | 540-73-8 | Hydrazine, 1,2-dimethyl- |
| U101 | 105-67-9 | 2,4-Dimethylphenol |
| U101 | 105-67-9 | Phenol, 2,4-dimethyl- |
| U102 | 131-11-3 | 1,2-Benzenedicarboxylic acid, dimethyl ester |
| U102 | 131-11-3 | Dimethyl phthalate |
| U103 | 77-78-1 | Dimethyl sulfate |
| U103 | 77-78-1 | Sulfuric acid, dimethyl ester |
| U105 | 121-14-2 | Benzene, 1-methyl-2,4-dinitro- |
| U105 | 121-14-2 | 2,4-Dinitrotoluene |
| U106 | 606-20-2 | Benzene, 2-methyl-1,3-dinitro- |
| U106 | 606-20-2 | 2,6-Dinitrotoluene |
| U107 | 117-84-0 | 1,2-Benzenedicarboxylic acid, dioctyl ester |
| U107 | 117-84-0 | Di-n-octyl phthalate |



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| U108 | 123-91-1 | 1,4-Diethyleneoxide |
| U108 | 123-91-1 | 1,4-Dioxane |
| U109 | 122-66-7 | 1,2-Diphenylhydrazine |
| U109 | 122-66-7 | Hydrazine, 1,2-diphenyl- |
| U110 | 142-84-7 | Dipropylamine (I) |
| U110 | 142-84-7 | 1-Propanamine, N-propyl-(I) |
| U111 | 621-64-7 | Di-n-propylnitrosamine |
| U111 | 621-64-7 | 1-Propanamine, N-nitroso-N-propyl- |
| U112 | 141-78-6 | Acetic acid ethyl ester (I) |
| U112 | 141-78-6 | Ethyl acetate (I) |
| U113 | 140-88-5 | Ethyl acrylate (I) |
| U113 | 140-88-5 | 2-Propenoic acid, ethyl ester (I) |
| U114 | ¹ 111-54-6 | Carbamodithioic acid, 1,2-ethanediybis-, salts & esters |
| U114 | ¹ 111-54-6 | Ethylenebisdithiocarbamic acid, salts & esters |
| U115 | 75-21-8 | Ethylene oxide (I,T) |
| U115 | 75-21-8 | Oxirane (I,T) |
| U116 | 96-45-7 | Ethylenethiourea |
| U116 | 96-45-7 | 2-Imidazolidinethione |
| U117 | 60-29-7 | Ethane, 1,1'-oxybis-(I) |
| U117 | 60-29-7 | Ethyl ether (I) |
| U118 | 97-63-2 | Ethyl methacrylate |
| U118 | 97-63-2 | 2-Propenoic acid, 2-methyl-, ethyl ester |
| U119 | 62-50-0 | Ethyl methanesulfonate |
| U119 | 62-50-0 | Methanesulfonic acid, ethyl ester |
| U120 | 206-44-0 | Fluoranthene |
| U121 | 75-69-4 | Methane, trichlorofluoro- |
| U121 | 75-69-4 | Trichloromonofluoromethane |
| U122 | 50-00-0 | Formaldehyde |
| U123 | 64-18-6 | Formic acid (C,T) |
| U124 | 110-00-9 | Furan (I) |
| U124 | 110-00-9 | Furfuran (I) |
| U125 | 98-01-1 | 2-Furancarboxaldehyde (I) |
| U125 | 98-01-1 | Furfural (I) |
| U126 | 765-34-4 | Glycidylaldehyde |
| U126 | 765-34-4 | Oxiranecarboxyaldehyde |



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| U127 | 118-74-1 | Benzene, hexachloro- |
| U127 | 118-74-1 | Hexachlorobenzene |
| U128 | 87-68-3 | 1,3-Butadiene, 1,1,2,3,4,4-hexachloro- |
| U128 | 87-68-3 | Hexachlorobutadiene |
| U129 | 58-89-9 | Cyclohexane, 1,2,3,4,5,6-hexachloro-, (1alpha,2alpha,3beta,4alpha,5alpha,6beta)- |
| U129 | 58-89-9 | Lindane |
| U130 | 77-47-4 | 1,3-Cyclopentadiene, 1,2,3,4,5,5-hexachloro- |
| U130 | 77-47-4 | Hexachlorocyclopentadiene |
| U131 | 67-72-1 | Ethane, hexachloro- |
| U131 | 67-72-1 | Hexachloroethane |
| U132 | 70-30-4 | Hexachlorophene |
| U132 | 70-30-4 | Phenol, 2,2'-methylenebis[3,4,6-trichloro- |
| U133 | 302-01-2 | Hydrazine (R,T) |
| U134 | 7664-39-3 | Hydrofluoric acid (C,T) |
| U134 | 7664-39-3 | Hydrogen fluoride (C,T) |
| U135 | 7783-06-4 | Hydrogen sulfide |
| U135 | 7783-06-4 | Hydrogen sulfide H ₂ S |
| U136 | 75-60-5 | Arsinic acid, dimethyl- |
| U136 | 75-60-5 | Cacodylic acid |
| U137 | 193-39-5 | Indeno[1,2,3-cd]pyrene |
| U138 | 74-88-4 | Methane, iodo- |
| U138 | 74-88-4 | Methyl iodide |
| U140 | 78-83-1 | Isobutyl alcohol (I,T) |
| U140 | 78-83-1 | 1-Propanol, 2-methyl- (I,T) |
| U141 | 120-58-1 | 1,3-Benzodioxole, 5-(1-propenyl)- |
| U141 | 120-58-1 | Isosafrole |
| U142 | 143-50-0 | Kepone |
| U142 | 143-50-0 | 1,3,4-Metheno-2H-cyclobuta[cd]pentalen-2-one, 1,1a,3,3a,4,5,5,5a,5b,6-decachlorooctahydro- |
| U143 | 303-34-4 | 2-Butenoic acid, 2-methyl-, 7-[[2,3-dihydroxy-2-(1- methoxyethyl)-3-methyl-1-oxobutoxy]methyl]- 2,3,5,7a-tetrahydro-1H-pyrrolizin-1-yl ester, [1S- [1alpha(Z),7(2S*,3R*),7aalpha]]- |
| U143 | 303-34-4 | Lasiocarpine |
| U144 | 301-04-2 | Acetic acid, lead(2 +) salt |
| U144 | 301-04-2 | Lead acetate |



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| U145 | 7446-27-7 | Lead phosphate |
| U145 | 7446-27-7 | Phosphoric acid, lead(2 +) salt (2:3) |
| U146 | 1335-32-6 | Lead, bis(acetato-O)tetrahydroxytri- |
| U146 | 1335-32-6 | Lead subacetate |
| U147 | 108-31-6 | 2,5-Furandione |
| U147 | 108-31-6 | Maleic anhydride |
| U148 | 123-33-1 | Maleic hydrazide |
| U148 | 123-33-1 | 3,6-Pyridazinedione, 1,2-dihydro- |
| U149 | 109-77-3 | Malononitrile |
| U149 | 109-77-3 | Propanedinitrile |
| U150 | 148-82-3 | Melphalan |
| U150 | 148-82-3 | L-Phenylalanine, 4-[bis(2-chloroethyl)amino]- |
| U151 | 7439-97-6 | Mercury |
| U152 | 126-98-7 | Methacrylonitrile (I,T) |
| U152 | 126-98-7 | 2-Propenenitrile, 2-methyl- (I,T) |
| U153 | 74-93-1 | Methanethiol (I,T) |
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| U155 | 91-80-5 | Methapyrilene |
| U156 | 79-22-1 | Carbonochloridic acid, methyl ester (I,T) |
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| U160 | 1338-23-4 | 2-Butanone, peroxide (R,T) |
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¹CAS Number given for parent compound only.



Advanced
Environmental Laboratories, Inc.

QUALITY MANUAL

Revision Number 10.4

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Last Revised By: Robert Bartolo

Quality Systems Manual

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Quality Manual Revisions

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| Revision 2 | November-05 | Addition - DEP Field SOP's FA1000, FM 1000 | 4.0 Page 1-33, 4.3 Page 1-23 |
| Revision 3 | November-03 | Procedure addition -Client questions/reanalysis reviewed by Technical Director | 2.2.2, Page 1-2 |
| Revision 3 | November-03 | Addition - Noted as highlighted and underlined | 5.1.1.1 Page 1, 5.4 Page 9 |
| Revision 3 | November-03 | Addition - Jacksonville ICP, Turbovaps, Muffle Furnaces; Gainesville Infrared Therm, Chlorine Meter, pH probe | 7.0 (Table 7.1),Page 2, 7.0(Table 7.3), Page 4 |
| Revision 3 | November-03 | Reference - DEP Field SOP's noted by underline | 8.0, Page 5 |
| Revision 3 | July-03 | SOP reference - ADMIN-021, Addition 12.2.3.1 h. NELAC Quality Systems Review checklist | 12.2.3.1, Page 4 |
| Revision 3 | November-03 | Added reference to SOP ADMIN-025, Added reference to local storage facility | Addendum Documentation |
| Revision 3 | November-03 | Update Corrective Action Reports, Figure 11.1, 11.2, 11.3 | 11.0, Page 4-6 of 15 |
| Revision 4 | November-03 | Clarification - New Methodologies and accepting new work | 1.5 - 1.6, Page 3-4 |
| Revision 4 | December-03 | Organizational Chart Update | 3.0, Page 7 of 7 |
| Revision 4 | November-03 | Addition - Methods for Jacksonville E82574 December 2003 Application | Appendix IX, Page 1-39 |
| Revision 4 | November-03 | Addition - storage facility noted by underline | 10.0, Page 11 |
| Revision 5 | April-04 | Full revision. New QA Officer redoing entire manual and renaming many sections and creating new sections to reference NELAC 2002 Standards All sections now listed as Revision 5 . Archived Manual on file. | ALL SECTIONS |
| Revision 5.1 | October-04 | Made adjustments and additions in relation to correcting deficiencies listed in the 2004 internal audits | Sects. 1.0, 2.0, 3.0, 4.0, 5.0, 6.0, 8.0, 11.0, 14.0 and 15.0 |
| Revision 5.2 | January-05 | Made adjustments and corrections in response to FDOH assessment of Orlando and Tampa Facilities. | Sects. 1.0, 2.0, 3.0, 6.0, 14.0 |
| Revision 5.3 | March-05 and April -05 | Made adjustments and corrections in response to FDOH assessment of Jax facility | Sects. 1.0, 2.0, 3.0, 7.0, 8.0, 9.0, 12.0, 14.0, TOC, & Title Page |
| Revision 6 | June-05 through August -05 | Full revision reference NELAC 2003 Standards. All sections now listed as Revision 6 . Archived Manual on file. | All Sections |
| Revision 6.1 | December-06 | Organizational Chart Update, Addition-Orlando 2 Refrigerators, BOD Incubator, Vaccum pump | Sect. 2.0, page 13 Sect. 7.0, page 31 |
| Revision 7.0 | July-06 | New year revision, All sections now listed as Revision 7.0 | All Sections |
| Revision 7.1 | August-06 | Gainesville new location, Organizational Chart Update , Minimum requirements for employment, SOP Tables update, Offsite storage visits, Gainesville Equipment and Methods Update, For calibration when using correlation coefficient to read 0.990 or greater required. | Sect. 2.0, pages 9,10,13 Sect. 3.0 SOP Tables pages 15-21,Sec 4.0 pages 1,2 Sect 7.0 all, Sec 8.1.6, Sect 14.0 all |
| Revision 7.2 | January 05-2007 | All section headers changed. Revision date now to be the date at which revisions to 7.2 began. Effective date to be date QM 7.2 placed in service. Fixed for grammar and typos throughout. The use of PQLs section 12. Gainesville change of address. Update of analyte list. Update of equipment list. Internal audit schedule set for 2007. | All sections |
| Revision 7.3 | February 23-2007 | Make listing of methods that use alternate procedures for iDOCs, update SOP tables. | Section 3, TOC |
| Revision 7.3 | August 28-2007 | Update Corporate Organizational Structure. | Section 2, Page 13 |
| Revision 7.3 | September 4-2007 | Update Equipment Lists. Section 7.0 pages 2-23 | Section 7 |
| Revision 7.3 | 5/30/2008 | Update management permit of departures-section 10.1.1 | Section 10 |
| Revision 7.3 | 6/19/2008 | Annual review dates updated for 2008-2009 | Section 11 |
| Revision 7.3 | 6/20/2008 | Update wording to specifically address each lab's sample acceptance policy. Add section 6.19. | Section 6, all |
| Revision 7.4 | 6/20/2008 | Update Equipment Lists. Section 7.0 pages 2-23 | Section 7 |
| Revision 7.3 | 4/6/2009 | Change wording to state that signed documentation needed for having read latest version of Quality Manual | Section 1, page 2 |
| Revision 7.4 | 4/6/2009 | Update Corporate Organizational Structure. | Section 2, Page 13 |
| Revision 7.5 | 4/6/2009 | Update listing and latest revisions of SOPs in tables | Section 3 |

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| Revision # | Revision Date | Reason for Revision | Section/Page # |
|--------------|---------------|--|-------------------------------------|
| Revision 7.5 | 4/6/2009 | Update preservation tables, and state policy for sample handling during power loss, hurricane, or unforeseen event | Section 6, page 21, Appendix A1-A23 |
| Revision 7.5 | 4/6/2009 | Update Equipment Lists. Section 7.0 pages 2-23 | Section 7 |
| Revision 7.3 | 4/6/2009 | Update and add methods to tables | Section 14 |

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|--------------|------------|---|--|
| Revision 8.0 | 10/30/2009 | Repaginate entire QM, with edits to many sections. Section 1, 2, 4 wording changed and additions made, Section 3 SOP list updated, Sec 6 edits to sub-sampling and representative sampling in soils, Sec 7 equipment list updated, Sec 8 calibration rules and peak identification rules expanded. Sec. 9 new tables inserted. Sec. 10 audit schedule updated, Sec. 12 mdl section expanded to include MDL acceptance criteria, Section 14 calibrations for new methods added. | ALL SECTIONS |
| Revision 8.1 | 5/5/2010 | Cover and Signatory pages plus Section 2 expanded to include new Miami facility. Section 3 edited to add/ update training SOPs, Section 5 and 8 add reference to TECH SOPs. Section 6 Sample procedures for multiphase samples expanded. Section 7 add Miami equipment list. Section update FDEP preservation tables. Section 11 new date for annual Internal Audit. Section 12 MDL, LOD, PQL, and LOQ definitions expanded, updated qualifier tables. Section 13 added language to better define sampling plan. Section 15 updated backup procedures. | Sections 2,3,5, 6, 7,8,11, 12, 13, 15 |
| Revision 9.0 | 7/1/2011 | New full revision to update language and to comply with TNI 2009 Standards. All sections affected with revision now listed as Revision 9.0. Cover and Signatory pages plus Section 2 updated to new Lab Managers and to add Tallahassee. Section 1.1.1 AEL stated to continually improve Quality System. Language added to Managements Systems. Section 3 update list of non-conventional iDOCS. Section 7 update to equipment list, all labs. 9.8.4 reference is now made to section 10.0. Section 10.3 expansion to NCF format. Section 14, table 14 updated method with letter designations. References to TNI instead of NELAC made throughout. Section 16 references added. | Sections 1, 2, 3, 7, 9, 10, 14, 16. All sections to update language from NELAC to TNI. |
| Revision 9.1 | 7/1/2012 | Minor revision to update, add language. Section 2 updated to more fully define responsibilities to ensure compliance with TNI standards, edit technical director qualifications to match TNI Standards, and new chart for management structure. Section 5 states that master calibration thermometers will now be sent out for NIST traceable calibration every 3 years. Section 7 update to equipment list, all labs. Section 12 definition for PQL rewritten to say that PQL is greater than MDL, changed from PQL is equal to or greater than MDL. Section 14, table 14 updated all labs, methods with letter designations. Acronym of DoD defined as Department of Defense at first use in each section where it appears. | Sections 2, 5, 7, 12, 14 |
| Revision 9.2 | 3/14/2014 | Minor revision to update, add language. Sec 2 Add Tim Preston as new Lab Manager Tallahassee. Add position of Field Services Supervisor Section Update SOP list, 3.4.1 Change language for Microbiology iDOCS to better reflect 2009 TNI Standards. Module 5. Update references in Sections 1,16. Sec 7, update equipment list. Sec 10 Update NCF procedures. Preventative Action Procedures added. Sec 11 Biennial Audits with Private Contractors. Sec 12 Expand on definition for hold time and analysis start. Sec 14 Update methods listed. All sections for grammar and spelling. | All Sections |

Quality Manual Revisions

| Revision # | Revision Date | Reason for Revision | Section/Page # |
|--------------|---------------|---|--|
| Revision 9.3 | 3/25/2015 | Minor revision to update, add language. Section 1.2 SOP access and approval process updated. Section 2.2.2.2 added that QA is independent of operations and free from managerial influences. Section 3.4.2.4, 3.9 The analyst documentation for reading the SOP (and QM) is done through e-mail. SOP 3.5 Alternate IDOC criteria updated for Odor and added for TCLP and SPLP. Section 4.5 "Q" drive now designated as Quality Assurance drive with controlled access and control documents. Restated in 4.10.2 as common directory for all labs. 4.11 Addition of cyber-security and cyber security training schedule. Section 5.2 added annual balance verification reference to Adel SOP Tech-008. 5.3.2 States Master NIST thermometers calibrated every 3 years. Section 6.20 Expansion to define lab's responsibility to client in regards to subcontracting labs. Section 7.0 Routine equipment inventory updates. Section 8.2.8 Addition of special condition for accepting CCV when high bias with non-detects. Section 9.1.4 Performance testing language (under DoD work) for testing by other measures. | See specific references to sections in reason for revision column. |

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| | | Section 10.1.1.4 Any suspended testing resumed only after QA approval. 10.1.1.5, .6 All audit findings and PT failures tracked through the NCF Program. Section 10.8 Preventative action section added. Section 11.5.2 Time set for notifying client (10 days or sooner) if a condition discovered during an internal will impact reported data. Figure 11.1 Internal audit schedule reset for the year. Section 13 FDEP Field SOPs now reference 7/30/14 SOPs. Section 14 Methods updated to add GRO, MADEP-EPH, MADEP-VPH, RSK-175 for Jacksonville and Enterococci by SM9320C for Tampa. 15.8 Some LIMS permissions are assigned by department (rather than individuals) in non-critical areas such as extractions where log traceability is clearly evident. For DoD work, user login shall also be through the EISC reporting software, always with individual password. Section 16 References updated to most recent dates. | |
| Revision 9.4 | 3/25/2016 | Sec 2 Minor revision to update, add language. Add Todd Romero as the new Lab Manager for Gainesville and Adolfo Fernandez as the new Lab Manager for Miami. Sec 3 Update language to emphasize electronic records and electronically saved Quality Manual and SOPs. Add to list of SOPs for the lab. Sec 4 Co-location for AEL servers now housed in Tampa. Annual cybersecurity training shall be held at each lab and no longer just at Jacksonville. Added instructions for the use of electronic logbooks. Sec 6 Expand sample rejection policy to include contacting client. Sec 7 updates to equipment list at all labs. Sec 9 update to accrediting bodies. Sec 11 All labs will now track internal audit findings, root cause, and corrections using the NCF forms. Internal audit dates set for all labs on 2016 - 2017 schedule. | See specific references to sections in "reason for revision" column. |
| Revision 9.5 | 6/25/2017 | Sec 1 update reference to DoD QSM 5.1. Sec 2 Add Fort Myers as 7th AEL Lab and update personnel. Sec 3 update list of SOPs. IDOC procedure listed for Ignitability. Sec 4 Clarify the labs process for saving original observations, data, and calculations. 4.15.6 Require that for any changes made to data (either hardcopy or electronic) include the identification of the person who made the change and the date of change. Sec 6.17 Clarify use of modified or developed methods. Sec 6.20 Clarify reporting requirements of sub-contracted lab data. Sec 7 updates to equipment list at all labs. Sec 10.7 Add requirements for making notifications of non-conforming work to client and AB. Sec 11.3 State that for DoD audit findings, willful avoidance of corrective action may result in loss of accreditation and suspension of work. Sec 12 Updated references to DoD QSM based on rev 5.1. Sec 12.7 Add statement that report designed to prevent misunderstanding or misuse. Sec 12.1 Clarify reporting of detection limits. 12.12 Add sec to address amended reporting. Section 14 Update lab methods and add Fort Myers. Update references in section 16. | See specific references to sections in "reason for revision" column. |
| Revision 10.0 | 11/9/2018 | Major revision to update to new language/requirements of TNI 2016. State ID for the Orlando lab changed to E83076. Employee training records now saved electronically and reviewed semi-annually rather than annually. Update listed SOPs section 3. Section 4: Tampa is listed as main AEL servers location with access by IT personnel only. Section 5.3 Thermometer calibrations can be performed at a single point when range of use is less than 10°C. 6.19 Expanded to specifically state that if a project has samples ran in another AEL location, that lab is responsible for entering data into LIMS, but the managing lab is responsible for generating the report with all of the data included. Section 7: Update to equipment list. Addition of new methods. Section 9: 2016 TNI requirement to test to PRTL level listed by TNI tables. Multiple PTs for new scope or makeup must be performed at least 7 days after close of previous study. Section 11: Audit schedule updated. | ALL SECTIONS |
| Revision 10.1 | 2/19/2019 | The inventory was updated to identify DO meter probes, pH meter probes, conductivity probes and nitrification inhibitor dispensers. Section 7. Language was added to define internal audit procedures and update-expand internal audit schedule. section 11. Updates to reference QSM 5.2 an ISO 17025:2017. sections 1, 16 | Section 1, 7, 11, 16 |
| Revision 10.2 | 3/31/2020 | Minor language, grammar, and spelling edits throughout. References updated to QSM 5.3 in section 1.1. Update company structure (personnel), figure 2.1. SOP lists updated, PFAS added, section 3. Where no CofA available for a consumable, a test of the consumable should be made and documented to show appropriate for lab use, section 5.5. The inventory was updated to identify new equipment, all labs in section 7. Updates procedures for case narratives through AEL intranet, section 10.8. Update procedures for calculating MDLs to EPA CFR 40 Part 136 Appendix B and TNI 2016 Standards, section 12.1. Removal of language referring to paper project folders, section 12.8. | Section 1, 2, 3, 5, 7, 10, 12, 14 |

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|---------------|-----------|--|---------------------------------|
| Revision 10.3 | 3/31/2021 | 1.2.15 Instruction to Identify risks to impartiality. 1.2.8 Assign edit responsibility of Policies and Procedures to Corporate TD and Operations VP 2.3-2.5 Update job descriptions, 2.17 Appendix update company structure. 3.0 tables -Update SOP lists. 5.10.1 Syringes to only require annual calibration. Figure 6.2 Update login checklist, figure 6.7 update Data Quality Objectives form. 10.3 Intranet web application is now used to generate NCFs. 11.1.3-11.1.4 Clarify procedure for internal auditing, figure 11.1 update auditing schedule. 13.5 Addition of AEL specific Field Services Protocols. | Sections 1, 2, 3, 6, 10, 11, 13 |
| Revision 10.3 | 5/6/2021 | Added Miami Organics and Microbiology Technical Directors and signatures | Title Page |
| Revision 10.4 | 3/31/2022 | Minor language, grammar, section reference updates and spelling edits throughout. References updated to QSM 5.4 throughout. Updated Titles in section 1.1.2. Updated reference from email sop concurrence system to Intranet SOP Review and Acknowledgement System throughout. Added updated Facilities and Equipment List. Updated conflicting language in section 8.2.6. Added relative error criteria and changed curve criteria in section 8.3.6. Updated number of calibrations points required by TNI to section 8.3.12. Updated RPD calculation default to Concentration from %R in Section 12. | Sections 1,7,8,12 |

1.0 AEL Corporate Policy

1.1 Quality Policy Statement

- 1.1.1 The Quality System of AEL is designed to accomplish the following goals: generate quality data by providing sampling and analyses that comply with The NELAC Institute (TNI) standards as well as all state and federal regulations, provide timely reporting of sampling and analysis results in compliance with the methods, standard operating procedures, and this Quality Manual, and maintain all documentation pertaining to sampling and analysis according to defined protocols in this Quality Manual.

In addition, the Quality System of AEL is designed with procedures to ascertain and meet the customer's requirements while operating within AEL's documented ethics policy. AEL management is committed to ethical laboratory practices while complying with and upholding the requirements of the TNI 2016 Standards and for the Jacksonville Laboratory also the Department of Defense (DoD) Quality Systems Manual (QSM) version 5.4.

Finally, Management's commitment is to not only to comply with but also to continually improve upon AEL's Quality System.

- 1.1.2 AEL Management is defined as the Company President, Vice President of Operations, Corporate Technical Director, Director of Client Services., IT Director, QA Officers, and the Lab Managers of the individual laboratories within the AEL network.

- 1.2 AEL Management will establish, implement, and maintain a management system appropriate to the scope of its activities. The laboratory will document its policies, systems, programs, procedures, and instructions to the extent necessary to assure the quality of the test and/or calibration results. Laboratory activities are undertaken impartially and structured and managed so as to safeguard impartiality. AEL Managements shall accomplish these goals by completing the following tasks:

- 1.2.1 In accordance with the TNI Standards, AEL management and its employees will adhere to this Quality Manual (QM).

1.2.1.1 The QM will be maintained current by the Corporate Technical Director to the applicable version of the TNI standards and to the most current version of the DoD QSM (as needed for the Jacksonville laboratory).

- 1.2.2 AEL Management will provide the necessary employees, instrumentation, and equipment to perform sampling and analyses referenced herein to meet all regulations and requirements as dictated by TNI, State or Federal Regulation guidelines.
- 1.2.3 AEL Management will review the capabilities of the laboratory, the instrumentation required and the current workload before accepting any new work that could affect the adherence to the Quality System. The Director of Client Services works closely with the Vice President of Operations, Corporate Technical Director, and Lab Managers before obtaining new work to ensure it can be completed as promised to the client while still adhering to the Quality System.
 - 1.2.3.1 New work is defined as either a large-scale project that would put a strain on the current workload already in house, a large new client, or a new method that is not yet being analyzed within the AEL network.
- 1.2.4 To ensure that all the policies defined in the Quality Manual and SOPs are followed to satisfaction, Management will utilize hiring practices to obtain the highest quality individuals for employment within AEL.
- 1.2.5 SOPs shall be approved and maintained by the Corporate Technical Director with the aide of all Quality Assurance Officers. It shall be the responsibility of the Corporate Technical Director and the Vice President of Operations to act as the approving authority and to review and approve all edits, revisions, or newly created SOPs.
 - 1.2.5.1 In the editing, revision, and updating of existing SOPs, or creation of new SOPs, the Corporate Technical Director and/or the Vice President of Operations shall either perform these tasks themselves or delegate these tasks to those individuals they feel will best review, revise, or create the SOPs.
 - 1.2.5.2 The Corporate Technical Director and/or the Vice President of Operations must review and approve all SOPs revisions prior to the effective date. Only approved SOPs shall be found on the laboratory's internal web system accessed by an icon on each analyst's desktop.
- 1.2.6 All employees, once hired will be required to read the Quality Manual and acknowledge through Intranet SOP Review and Acknowledgement System that they have read, understood, and agree to follow the guidelines set forth in the Quality Manual.

- 1.2.7 A revision to a section or a revision of the entire Quality Manual will require all employees and management to read those new portions (or the entire QM if revised in whole) and acknowledge through Intranet SOP Review and Acknowledgement System that they have read, understood, and agree to follow the guidelines set forth in the Quality Manual.
- 1.2.8 AEL Management will provide all employees with an employee handbook currently titled "Policies and Procedures Manual" which details all aspects of employment with AEL as well as all disciplinary actions that may be taken in instances of violations of those policies. The Corporate Technical Director and/or the Vice President of Operations keeps this handbook current.
- 1.2.9 Through the course of training, AEL employees shall be made aware of the relevance and importance of their activities and how they contribute to the achievements of the objectives of the management system.
- 1.2.10 AEL Management will ensure that appropriate communication processes are established within the laboratory and that communication takes place regarding the effectiveness of the management system.
- 1.2.11 AEL Management will ensure that the integrity of the management system is maintained when changes to the management system are planned and implemented.
- 1.2.12 AEL shall actively seek feedback from clients, both positive and negative, to improve the management system, the testing activities, and customer services of the laboratories.
- 1.2.13 AEL Management will continually improve the effectiveness of its management system through the use of quality policy, quality objectives, audit results, analysis of data, corrective and preventative actions, and management review.
- 1.2.14 AEL Management as well as all AEL personnel shall analyze quality control data and where they are found to be outside predefined criteria, take action to correct the problem and to prevent incorrect results from being reported. If circumstances require that results with quality control outside criteria are to be reported, those results are to be qualified and an explanation is to be included with the results in an attached case narrative.
- 1.2.15 Ethical requirements of Laboratory Employees. All employees are required to sign the Code of Ethics form, attached as Figure 1.1 upon

hire. This signed form will be maintained in the employee's training file.

- 1.2.15.1 Employees Commitment – all employees must take responsibility for his or her conduct and performance of their duties as outlined in AEL's employee handbook, quality manual, and any procedures (SOP's) as set forth by the company and or any state or environmental regulatory agency.
- 1.2.15.2 All new hires shall receive ethics and data integrity training through the QA Department within the first two weeks of employment (full time) or one month (part time). Thereafter, all employees shall undergo ethics and data integrity training on a yearly basis. (January)
- 1.2.15.3 Maintenance of property – No employee shall use AEL property for any unethical use. All AEL property shall be used in accordance with the employee's job description and duties.
- 1.2.15.4 Impartiality & conflicts of interests – AEL management is committed to impartiality. All employee personal business, financial affairs, and any other endeavors shall be conducted in a manner, which does not conflict with their duties, responsibilities, or ethical standards at AEL. The analysts and lab personnel shall be free of commercial, financial, or other pressures in regard to the reporting of impartial results. Each lab shall identify any risks to its impartiality on an ongoing basis and shall demonstrate how it eliminates or minimizes such risks.
 - 1.2.15.4.1 To ensure impartiality, management shall be informed by the employee if they have any employment outside of the company. Also, the employee shall inform management if they have any personnel stake in the outcome of the result, such as an association with the client or entity for which the results are being performed. Management will then schedule the analysis in such a way so that the analyst does not come in contact with those samples.
 - 1.2.15.4.2 Any risks to impartiality shall be identified and documented in the Managerial Review along with documentation of how that risk has been minimized or eliminated. (See SOP Admin-021)

1.2.15.5 Analyst shall be free from any undue internal or external pressure and influences that may adversely affect the quality of the work.

1.2.15.6 Confidentiality – All AEL employees will maintain confidentiality of all AEL proprietary information and or procedures when dealing with any outside agency or public. Federal, state, and local government agencies requesting proprietary or confidential AEL information must utilize appropriate procedures for requests through the appropriate chains of command at AEL (i.e., Laboratory Director, Laboratory Manager, and or Project Manager).

1.2.15.6.1 All employees are required to sign the Confidentiality Statement upon hiring, see Figure 1.2. This signed document will be maintained in the employee's training file.

1.2.15.6.2 For transmission of reports and correspondence with clients, AEL utilizes the common practices to ensure these communications are kept confidential and only for the intended recipient of the information. This is accomplished by utilizing cover pages for facsimile and confidentiality statements on all electronic communication, such as email.

1.2.15.6.3 Here is an example of the confidentiality statement required for all email correspondence within the AEL network: *“NOTICE: This communication may contain privileged or other confidential information. If you are not the intended recipient, or believe that you have received this communication in error, please do not print, copy, retransmit, disseminate, or otherwise use the information. Also, please indicate to the sender that you have received this communication in error, and delete the copy you received.”*

1.2.15.7 If a third-party requests to review the data, and they are not specifically listed on the chain-of-custody, then written permission must be obtained from the client listed on the chain-of-custody before any information can be shared with third parties.

1.2.15.8 Truthfulness – All employees will be accurate, truthful, and complete in their interactions with other employees, supervisors,

QA officer, laboratory manager, state agencies, federal agencies, local government agencies, and the public. All employees will respond to job inquiries with due diligence accurately, honestly and in compliance with all AEL policies and or SOP's.

1.2.15.9 Analysis and reporting – Employees must analyze and report data that is valid and meets the requirements of 40 CFR, the Standard Methods, and the EPA methods under which analysis takes place and assure that all quality control measures have been adhered to as set forth by the 40 CFR, Standard Methods, EPA methods, AEL Quality Manual and AEL SOP's. Any intentional misrepresentation of analysis or reporting of data will subject the employee to AEL's auditing and reprimand processes.

1.2.15.10 Bidding and price quoting – Any employee authorized to submit bids for AEL analytical services will ensure that no misrepresentations or omissions have occurred in the submittal and that all submitted information is true, accurate and abides by all state, federal, or local governmental rules and regulations.

1.2.15.11 Requests for payment – Any employee authorized to submit invoices, claims, or other requests for payment to AEL will ensure that the invoices, claims or supporting documentation reflect truthful and accurate information.

1.2.15.12 Charges for labor or services – any employee required to submit charges or expense reports to AEL will report such charges accurately and truthfully. The president of the lab, lab director, or lab manager will approve all charges.

1.2.16 Ethics Violations

1.2.16.1 Internal investigations – A Lab Director, Lab Manager, and/or QA Officer may periodically conduct internal investigations regarding unethical behavior or analytical practices. Employees will cooperate fully and honestly regarding analytical practices and or supervisory instruction. The QA Officer will retain records of investigations and employee files will be updated accordingly. All audit findings will be reported to the Lab Director, Lab Manager, and QA Officer for review.

1.2.16.2 Reporting Misconduct – All employees are to report any activity regarded as unethical or deemed as general misconduct that might affect the quality and integrity of analytical data. All reports are to be submitted to the QA Officer. Any employee

desiring to remain anonymous in their reporting of misconduct may submit in writing to the QA Officer via anonymous note.

1.2.16.3 Sanctions - Any employee exhibiting unethical behavior that may affect the quality and or validity of analytical data will be subjected to reprimand procedure and or termination from AEL. Any employee supervisor or the Lab Manager may implement disciplinary action. The Lab Director or Lab Manager may implement termination of employment or suspension from AEL with the Corporate Technical Director's and Vice President of Operation's approval. Any employee obstructing, misrepresenting, or omitting any facts during an investigation shall be terminated immediately.

1.2.16.4 AEL Management will provide all employees access to the "AEL Chemical Hygiene Plan and Safety Manual". The employee will verify they have read the document with a signature stating they have read and understand all sections encompassed. Safety Data Sheets (SDS) shall be located in a common area for easy access.

1.2.16.5 Whenever an incident of Data integrity or Ethics is reported, suspected, or discovered documented mandatory Data Integrity and Ethics Training is required to be repeated by of all laboratory employees.

1.3 Transfer of Ownership/ Change of Location/ Change of Status

1.3.1 Transfer of Ownership of AEL

1.3.1.1 In the event AEL is purchased or ownership transferred, the transfer of all archived records generated by AEL will be negotiated with the new owner as to who is to maintain those records for the required 5 years. If it is decided that the new owner is to take possession and responsibility for proper keeping of those records for the minimum 5 years, then those records are transferred to the new owner. If not taken by the new owner, then the records will be transferred to a central storage unit and left there available for retrieval. The storage facility will remain the responsibility of the Vice President of Operations and Corporate Technical Director of AEL, or their appointee, until the records are disposed of. They will be maintained in storage for a minimum of 5 years, (maximum of 7), past the end of any project before being destroyed.

1.3.1.2 The purchasing authority will be responsible for the notification of this transfer of ownership to the accrediting authority.

1.3.1.3 In any other the case or circumstance involving change of ownership not listed here, then the state regulatory agency shall be notified of this change and accommodations for the transfer of records such be designed.

1.3.1.4 All current records (within the most recent year of AEL's existence) will be transferred to the purchasing laboratory for archival purposes.

1.3.2 In the event AEL purchases another laboratory

1.3.2.1 The original laboratory will maintain all records unless AEL is directed to acquire them by the lab being purchased or through the purchased laboratory's Quality Manual.

1.3.2.2 AEL will assume responsibility for notification to all accrediting authorities that are involved with the transfer of ownership.

1.3.2.3 AEL will assume the accreditation number of the lab being purchased and become responsible for incorporating the Quality System of AEL into the newly acquired facility, including performance testing, and maintaining accreditation for certified analytes.

1.3.2.4 The purchased laboratory is responsible for notifying all regulatory agencies about storage of archived records and how to obtain them, unless explicitly explained to AEL how they will be handled and the necessary contacts, upon which time, AEL will assume this responsibility.

1.4 AEL changes location of one of its laboratories

1.4.1 The QA Officer will be responsible for all necessary written correspondence with the regulatory and accrediting agencies alerting them of the change and the new address.

1.4.1.1 The new facility will meet all necessary requirements and comply with the procedures outlined in this Quality Manual before the analysis of samples will begin.

1.5 Going out of business – Records Transfer

- 1.5.1 In the event AEL goes out of business, all records will be transferred to a centralized storage facility. These records will be retained for a minimum of 5 years past the end date of any projects, upon which they will be destroyed.
- 1.5.2 The Corporate Technical Director and Vice President of Operations or their appointee will remain responsible for retrieval and maintenance of the storage facility during the 5-year time span.
 - 1.5.2.1 All regulatory agencies will be notified, in writing, who is responsible for the storage facility, how to contact that person, and where the records are being stored.
 - 1.5.2.2 In any other case or circumstance involving change of ownership not listed here, the state regulatory agency shall be notified of this change and accommodations for the transfer of records shall be discussed.

1.6 Quality Manual Revision Process

- 1.6.1 The Quality Manual will undergo routine review as necessary to maintain consistency with the TNI Standards.
 - 1.6.1.1 As minor errors are seen and small non-procedural changes are needed, these edits shall be written into an amendment page(s) that will be attached to the Quality Manual preceding the table of contents. At the next full annual review and or if the number of edits reaches a level deemed necessary by the Corporate Technical Director, these amendments shall be fully incorporated into the Quality Manual and the revision number for minor revisions will be indicated by changing the suffix of the revision number, such as 10.2 to 10.3. Major revisions will move to the next full revision number, such as revision 10.2 moving to revision 11.0.
- 1.6.2 Minor and Major revisions will be saved with the date of the revision as part of the filename and retained for archival purposes electronically on the designated Quality Assurance (Q) drive of the AEL networked servers.
- 1.6.3 On an annual basis, the Quality Manual will undergo review, and if necessary, be given a full, formal revision, upon which the revision number will change.

- 1.6.4 When reviews have revisions that are minor, this will be indicated by changing the suffix of the revision number, such as 10.2 to 10.3.
- 1.6.5 When reviews have major revisions, the Revision Number will be changed to the next whole number for all sections, and all suffixes will return to 0. Example: 10.4 will go to 11.0, 11.3 will go to 12.0 etc.
 - 1.6.5.1 A full revision will be made whenever adoption of a new set of TNI Standards is introduced. This will ensure the Quality Manual always references the most recent and correct version of the TNI Standards.
 - 1.6.5.2 A full, formal revision requires submittal of the revised QM to the Florida Department of Health (FDOH) or be made available to the accrediting body at the next scheduled assessment.
- 1.6.6 Once a revision is complete and reviewed, the new electronic revision will replace the older electronic version of the Quality Manual in the web-based document library to ensure that all personnel have access to the correct version of the QM.

Figure 1.1

ADVANCED ENVIRONMENTAL LABORATORIES, INC.
CODE OF ETHICS

I, _____, state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at Advanced Environmental Laboratories, Inc. as explained to me by AEL management.

I agree to:

- 1) Provide accountability for the quality and integrity of the laboratory services I provide.
- 2) Strive to maintain and improve my technical knowledge and professional competence.
- 3) Maintain cooperative, professional working relationships with colleagues and laboratory clients.

Furthermore, I will notify management of any unethical or fraudulent conduct, by any employee. I also understand that any employee intentionally committing fraud will be terminated.

Signature

Figure 1.2

ADVANCED ENVIRONMENTAL LABORATORIES, INC.

CONFIDENTIALITY STATEMENT

I, _____, state that I will not discuss any information pertaining to or contained in clients' files with non-laboratory personnel. I understand that failure to adhere to this policy will result in disciplinary action.

Signature

Date

2.0 Organization and Management Structure

Advanced Environmental Laboratories, Inc. is an environmental laboratory network consisting of seven laboratories throughout the state of Florida. AEL is a privately owned company with the legal authority and responsibility held by the owner. The laboratory names and certification numbers are as follows:

Advanced Environmental Laboratories (Jacksonville), FL NELAP certification #E82574
Advanced Environmental Laboratories -Tampa, FL NELAP certification #E84589
Advanced Environmental Laboratories -Gainesville, FL NELAP certification #E82001
Advanced Environmental Laboratories -Orlando, FL NELAP certification #E53076 (E83076)
Advanced Environmental Laboratories -Miami, FL NELAP certification #E82535
Advanced Environmental Laboratories -Tallahassee, FL NELAP certification #E811095
Advanced Environmental Laboratories –Fort Myers, FL NELAP certification #E84492

AEL's Management structure is designed to have a few key employees as corporate employees and then others are designated to specific laboratories. **The attachment** at the end of this section is an organizational chart showing the organization's structure and defines the authority chain of command.

Listed below are the qualifications and Job Description for key personnel.

2.1 Laboratory Director (Technical Director)

2.1.1 Qualifications

2.1.1.1 A bachelor's degree in the chemical, environmental, biological sciences, physical sciences, or engineering, with at least 24 college semester credit hours in chemistry.

2.1.1.2 Minimum of 2 years experience in the environmental field. A Master's degree can substitute for one year of experience.

2.1.1.3 Minimum of 2 years experience in laboratory management.

2.1.2 Job Description

2.1.2.1 Provide operational and administrative leadership through planning and management of personnel and equipment resources.

2.1.2.2 Responsible for overall laboratory efficiency and financial performance of the laboratory.

2.1.2.3 Provide support for business development by identifying and developing new markets and through continuing support of the management of existing client activities.

2.1.2.4 Responsible for all functions of Laboratory functions and marketing.

2.1.2.5 Responsible for ensuring compliance with the TNI standards.

2.2 Quality Assurance Officer (see also AEL Tech SOP-014 QA Duties)

2.2.1 Qualifications

2.2.1.1 A 4-year college degree.

2.2.1.2 In lieu of a 4-year degree, 5 years as a supervisor responsible for data review will be acceptable.

2.2.1.3 Minimum 2 years experience dealing with environmental data review.

2.2.2 Job Description

2.2.2.1 Responsible for the AEL Quality System and its implementation. Serves as the focal point for all QA/QC issues.

2.2.2.2 Is independent from laboratory operations for which they have oversight. Has the ability to evaluate data objectively without outside (managerial) influence.

2.2.2.3 Oversee all quality control data with the assistance of the QA Deputies.

2.2.2.4 Responsible for maintaining the Quality Manual and ensuring its compliance with the current NELAC Standards.

2.2.2.5 Responsible for identifying and responding to QA needs, problems, and other requests.

2.2.2.6 Responsible for summarizing and reporting overall laboratory performance via internal audits, proficiency test programs, certification/accreditation activities, and blind and reference sample analysis.

- 2.2.2.7 Work with QA Deputies to ensure all labs in the AEL network understand and are following the Quality System.
- 2.2.2.8 Responsible for resolving all quality assurance issues that arise in any of the labs.
 - 2.2.2.8.1 The QA Officer has final authority to settle any disputes that may arise concerning QA issues in the laboratory network.
- 2.2.2.9 Responsible for approving any non-conformance, i.e., a deviation from the Quality System, that may occur in any part of the laboratory network.
- 2.2.2.10 Responsible for direct oversight of the QA Deputies. Indirect responsibility for Lab Managers and Project Managers with regard to quality assurance related issues.
- 2.2.2.11 Responsible for all documentation required as part of the procedures they perform, in accordance with the requirements of the Administrative Sops and the Quality Manual.
- 2.2.2.12 Follow policies outlined in Employee Handbook.
- 2.2.2.13 **Responsible for ensuring compliance with the TNI standards.**

2.3 Corporate Technical Director

- 2.3.1 Qualifications (Same as 2.2.1 above) as well as:
 - 2.3.1.1 At least BS in Chemistry, Environmental Science with 3 years environmental lab experience or 5 years environmental lab experience.
 - 2.3.1.2 Documented training and/or experience in QA/QC procedures, general knowledge of the analytical methods, highly organized and able to be objective of influence.
- 2.3.2 Job Description
 - 2.3.2.1 Serves as the supervisor of all lab QA officers and ensures they comply with all their duties and responsibilities.
 - 2.3.2.2 Oversight and maintenance of internal and external document control.

- 2.3.2.3 Scheduling and oversight of internal and external assessments.
- 2.3.2.4 Maintenance of in state and out of state regulatory requirements.
- 2.3.2.5 Interacts regularly with the labs and Managers on compliance and efficiency. Shares knowledge and experience with Managers and Analysts performing new and current methods and advises on equipment.
- 2.3.2.6 Report directly to the Vice President Operations.
- 2.3.2.7 Performs the daily on-call service role for lab users for all Horizon LIMS issues and troubleshooting via Help Desk ticket system.
- 2.3.2.8 Responsible for maintenance of Horizon LIMS and helps facilitate upgrades.
- 2.3.2.9 Assists and coordinates the completion of AEL paperless package initiative.
- 2.3.2.10 Establishes and helps enforce usage of primary/secondary data review in EISC.
- 2.3.2.11 Automates and streamlines Tier IV packages in Jacksonville for Federal work.
- 2.3.2.12 Assists lab QAs with control limits, detection limits and other QA LIMS responsibilities.
- 2.3.2.13 Responsible for the overall design and implementation of Organics and Metals lab automation.
- 2.3.2.14 Assists IT as needed on custom EDD and reports from Horizon LIMS and other related assignments.
- 2.3.2.15 Responsible for ensuring compliance with the TNI standards.**

2.4 Vice President Operations

2.4.1 Qualifications

- 2.4.1.1 Minimum of 5 years experience in the environmental industry.
- 2.4.1.2 Minimum of 2 years experience as a supervisor of personnel.

2.4.2 Job Description

- 2.4.2.1 Responsible for overall operation of laboratory network.
- 2.4.2.2 Responsible for handling routine tasks of daily business such as vendor negotiations and building maintenance/upkeep.
- 2.4.2.3 Responsible for maintaining employee handbook
- 2.4.2.4 Work closely and collaborate with the Lab Managers, Technical Directors, QA Officers, and QA Deputies to assist in the resolution of any personnel problems arising from either technical, QA, or unacceptable employee behavior issues that may arise.
- 2.4.2.5 Reports directly to the Company President/Owner. Directly responsible for oversight of the Lab Managers and indirectly responsible for everyone under the lab managers.
- 2.4.2.6 Follow policies outlined in Employee Handbook.
- 2.4.2.7 **Responsible for ensuring compliance with the TNI standards.**

2.5 Director of Client Services

2.5.1 Qualifications

- 2.5.1.1 Minimum of 5 years experience in the sales field for environmental chemistry.

2.5.2 Job Description

- 2.5.2.1 Responsible for overseeing the Sales Department
- 2.5.2.2 Responsible for developing all new marketing plans to bring in new work or to change sales directions
- 2.5.2.3 Work closely with the V.P. Operations, Technical Directors, and Lab Managers to ensure new work can be accomplished
- 2.5.2.4 Reports directly to the Company President/Owner.
- 2.5.2.5 Responsible for maintaining current customer relations to ensure continued flow of work into the laboratory.

2.5.2.6 Responsible for all documentation required as part of the procedures they perform, in accordance with the requirements of the Administrative Sops and the Quality Manual.

2.5.2.7 Follow policies outlined in Employee Handbook.

2.6 Deputy QA Officer (see also AEL Tech SOP-014 QA Duties)

2.6.1 Qualifications

2.6.1.1 At least 2 years of College and 1 year experience as a supervisor responsible for data review.

2.6.1.2 In lieu of College, 3 years as a supervisor responsible for data review will be acceptable.

2.6.2 Job Description (see also AEL Tech SOP-014 QA Duties)

2.6.2.1 Assist the QA Officer with implementation of the Quality System

2.6.2.2 Work more closely with the larger laboratories to ensure compliance while allowing the QA Officer to oversee corporate QA Policy.

2.6.2.3 Responsible for overseeing the Document Control System, as mentioned in this Quality Manual.

2.6.2.4 Work closely with the supervisors to ensure the employee training files are complete and accurate.

2.6.2.5 Reports directly to the QA Officer.

2.6.2.6 Responsible for all documentation required as part of the procedures they perform, in accordance with the requirements of the Administrative Sops and the Quality Manual.

2.6.2.7 Follow policies outlined in Employee Handbook.

2.6.2.8 **Responsible for ensuring compliance with the TNI standards**

2.7 Deputy Technical Director

2.7.1 Qualifications

2.7.1.1 At least 2 years of College and 1-year experience as a supervisor responsible for data review.

2.7.1.2 In lieu of College, 3 years as a supervisor responsible for data review will be acceptable.

2.7.1.3 This position can be filled by Department Managers when required. When the technical director is not onsite for 15 days which does not exceed 35 days.

2.7.2 Job Description (see also AEL Tech SOP-014 QA Duties)

2.7.2.1 Assist the Technical Manager with the daily operational and administrative leadership of personnel and equipment resources.

2.7.2.2 Assist in the efficiency and performance of the laboratory operations when Technical Director is off-site.

2.7.2.3 Responsible for all functions of laboratory functions and support of management of existing client activities.

2.7.2.4 Reports directly to Laboratory Manager and Technical Director.

2.7.2.5 Follow policies outlined in Employee Handbook.

2.7.2.6 **Responsible for ensuring compliance with the TNI standards**

2.8 Laboratory Manager / Laboratory Supervisor

2.8.1 Qualifications

2.8.1.1 4-years of college with a science background.

2.8.1.2 Minimum of 5 years experience in the environmental field.

2.8.1.3 Minimum of 2 years supervisory experience.

2.8.2 Job Description

2.8.2.1 Exercises day-to-day supervision of laboratory procedures.

2.8.2.2 Responsible for all data produced in the lab to be technically correct

- 2.8.2.3 Work with the QA Officer or QA Deputy to ensure quality control criteria, as defined in the analytical SOPs and the Quality Manual, are followed, and adhered to.
- 2.8.2.4 Monitor the validity of the analyses performed and data generated in the laboratory to assure reliable data.
- 2.8.2.5 Ensure that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- 2.8.2.6 Provide educational direction and mentorship to the laboratory staff.
- 2.8.2.7 Responsible for handling all instrumentation problems/malfunctions and working with the Lab Director to ensure all instrumentation is functioning properly and efficiently.
- 2.8.2.8 Responsible for ensuring data is processed through the lab and reported in a timely manner to meet the client deadlines while ensuring quality control is maintained.
- 2.8.2.9 Responsible for direct oversight of the Department Supervisors, Project Managers, Sample Receipt Personnel, and Office Personnel.
- 2.8.2.10 Responsible for all documentation required as part of the procedures they perform, in accordance with the requirements of the Administrative Sops and the Quality Manual.
- 2.8.2.11 Follow policies outlined in Employee Handbook.
- 2.8.2.12 **Responsible for ensuring compliance with the TNI standards.**

2.9 Department Supervisors

2.9.1 Qualifications

- 2.9.1.1 Minimum of 2 years as an analyst in their respective departments.
- 2.9.1.2 A college degree with a science background or a minimum of 5 years experience in their analytical department.

2.9.2 Job Description

- 2.9.2.1 Assure that all activities are performed according to methods and protocols specified in the Quality Manual and SOPs.

- 2.9.2.2 Work closely with the QA Officer or Deputy to verify adherence to the policies in this Quality manual are maintained for all analytical results produced by the laboratory.
- 2.9.2.3 Review analytical data by
 - 2.9.2.3.1 Checking documentation for completeness and proper sample identification.
 - 2.9.2.3.2 Checking raw data for calculation, interpretation, or clerical errors.
 - 2.9.2.3.3 Assuring that produced quality control data is acceptable.
- 2.9.2.4 Coordinate analytical work or field activities to assure completion of all tasks within established time frames.
- 2.9.2.5 Responsible for the training of all new analysts
- 2.9.2.6 Responsible for maintaining the employee-training file for completeness and accuracy.
- 2.9.2.7 Oversee preventative maintenance activities.
- 2.9.2.8 Evaluate and implement changes in methodology and quality control measures, as prescribed by the QA officer.
- 2.9.2.9 Identify quality control problems and takes measures to correct or eliminate the problem source.
- 2.9.2.10 Assume the responsibility for validating all data and assuring that final reports are accurate before final review by management.
- 2.9.2.11 Responsible for direct oversight and mentorship of all employees in their respective departments.
- 2.9.2.12 Responsible for all documentation required as part of the procedures they perform, in accordance with the requirements of the Administrative Sops and the Quality Manual.
- 2.9.2.13 Follow policies outlined in Employee Handbook.
- 2.9.2.14 Assumes the responsibilities of Deputy Technical Director and/or Deputy QA Officer when necessary.

2.9.2.15 **Responsible for ensuring compliance with the TNI standards**

2.10 IT Manager

2.10.1 Qualifications

2.10.1.1 A 4-year college degree in a computer related field or a certified MCSE.

2.10.2 Job Description

2.10.2.1 Responsible for maintaining the network of computers in each lab.

2.10.2.2 Responsible for maintenance and archival of LIMS database used for sample tracking and reporting of results.

2.10.2.3 Responsible for verifying functionality of all custom designed portions of the LIMS.

2.10.2.4 Responsible for Hardware and Software validation.

2.10.2.5 Responsible for training new employees learning to use the LIMS to ensure compliance with GALP.

2.10.2.6 Responsible for all documentation required as part of the procedures they perform, in accordance with the requirements of the Administrative Sops and the Quality Manual.

2.10.2.7 Follow policies outlines in Employee Handbook.

2.11 Project Manager

2.11.1 Act as a liaison between the client and the organization.

2.11.2 Oversee and coordinates project activities including work plans, quality assurance project plans and scheduling.

2.11.3 Review project data prior to final report to assure that all data (field and laboratory) are acceptable and within specified project objectives.

2.11.4 Work closely with the sample receipt personnel to ensure the sample acceptance policy is adhered to.

- 2.11.5 Responsible for indirect supervision of the sample receipt personnel.
- 2.11.6 Responsible for all documentation required as part of the procedures they perform, in accordance with the requirements of the Administrative Sops and the Quality Manual.
- 2.11.7 Follow policies outlined in Employee Handbook.

2.12 Project Manager Assistant

- 2.12.1 Assist Project Manager as needed on routine tasks while being supervised by the Project Manager. Those actions can include Act as a liaison between the client and the organization.
- 2.12.2 Oversee and coordinates project activities including work plans, quality assurance project plans and scheduling.
- 2.12.3 Review project data prior to final report to assure that all data (field and laboratory) are acceptable and within specified project objectives.
- 2.12.4 Work closely with the sample receipt personnel to ensure the sample acceptance policy is adhered to.
- 2.12.5 Responsible for indirect supervision of the sample receipt personnel.
- 2.12.6 Responsible for all documentation required as part of the procedures they perform, in accordance with the requirements of the Administrative Sops and the Quality Manual.
- 2.12.7 Follow policies outlined in Employee Handbook.

2.13 Analyst

- 2.13.1 Perform required analyses according to test methods specified by rule, permit, or Quality Assurance Project Plans
- 2.13.2 Assure that all analytical equipment has been properly calibrated before beginning tests.
- 2.13.3 Assure that all identifying information (including sample ID numbers) have been accurately transcribed into records or computer databases.
- 2.13.4 Assure that all calculations are correct.

- 2.13.5 Assure that appropriate confirmatory tests or procedures have been completed.
 - 2.13.6 Identify, document, and begin corrective actions on any quality control problem that relates to the analytical test.
 - 2.13.7 Maintain equipment in proper working condition and document all preventative maintenance and repairs.
 - 2.13.8 Responsible for all documentation required as part of the procedures they perform, in accordance with the requirements of the Administrative SOPs and the Quality Manual.
 - 2.13.9 Follow policies outlines in Employee Handbook.
 - 2.13.10 Must follow the TNI standards.
 - 2.13.11 Must have a High School Diploma or equivalency.
- 2.14 Field Services Supervisor
- To perform all duties as described under Field Technician, plus the following:
- 2.14.1 Perform a field audit of all AEL sampling personnel on a yearly basis.
 - 2.14.2 Set policy and performance standards for conducting field sampling activities in accordance with FDEP Sampling SOPs.
 - 2.14.3 Outline which forms and formats are to be used corporate wide. Select the platforms (hardcopy versus electronic) for collecting and distributing collected field parameters.
 - 2.14.4 Set the frequency for scheduled field equipment checks against in-house equipment. Establish which equipment requires routine maintenance and their schedules for maintenance.
 - 2.14.5 Suggest equipment purchases.
 - 2.14.6 Coordinate training for all sampling personnel. This to include HAZWOPPER, TREEO, and any train the trainer type training.
- 2.15 Field Technician
- 2.15.1 Perform field measurement tests according to specified methodology.

- 2.15.2 Collect samples using approved techniques and appropriate equipment.
 - 2.15.3 Assure that sample containers are properly and accurately labeled.
 - 2.15.4 Assure that appropriate preservatives are added and that appropriate sample containers are used to collect required fractions.
 - 2.15.5 Legibly document all activities in field logs or field data sheets.
 - 2.15.6 Assure that all identifying information is accurately recorded.
 - 2.15.7 Identifies and/or documents potential quality control problems (ex. unacceptable calibrations, environmental conditions, etc.)
 - 2.15.8 Responsible for maintaining equipment in working condition and documenting all preventative maintenance and repairs.
 - 2.15.9 Must have a High School Diploma or equivalency
- 2.16 Sample Technicians
- 2.16.1 Responsible for preparatory procedures, such as organic extractions and metals digestions.
 - 2.16.2 Follow the appropriate SOPs to perform their assigned tasks
 - 2.16.3 Work closely under supervision of senior analysts and supervisors.
 - 2.16.4 Follow all appropriate safety policies defined in the Safety Manual.
 - 2.16.5 Responsible for all documentation required as part of the procedures they perform, in accordance with the requirements of the Administrative Sops and the Quality Manual.
 - 2.16.6 Follow policies outlined in Employee Handbook.
 - 2.16.7 Must have a High School Diploma or equivalency
- 2.17 Sample Receipt Personnel
- 2.17.1 Responsible for direct contact with clients delivering samples to the laboratory.
 - 2.17.2 Must follow the sample acceptance policy and verify all criteria of the policy are met before taking custody of the samples.

- 2.17.3 Responsible for logging samples into the LIMS system used to track samples throughout the laboratory.
 - 2.17.4 Responsible for maintaining the storage locations for samples and disposal of samples throughout the lab.
 - 2.17.5 Under direct supervision of the project managers.
 - 2.17.6 Responsible for all documentation required as part of the procedures they perform, in accordance with the requirements of the Administrative Sops and the Quality Manual.
 - 2.17.7 Follow policies outlined in Employee Handbook.
 - 2.17.8 Must have a High School Diploma or equivalency.
- 2.18 Deputy Nomination
- 2.18.1 In the event of the absence of either the QA Officer, QA Deputy or Technical Director from the lab, a temporary deputy will be named.
 - 2.18.2 Notice of absence is to be given via email by the person who will be absent , sent to all affected constituents in the laboratory or laboratories, identifying when they will be out of the office and who will be responsible for dealing with their technical or QA issues in their absence.
 - 2.18.3 For AEL Jax
 - 2.18.3.1 Technical Director's absence will be filled by the QA Officer.
 - 2.18.3.2 The QA Officer's absence will be filled by the Technical Director or Laboratory Supervisor.
 - 2.18.4 For AEL Tampa
 - 2.18.4.1 The Technical Director's absence will be filled by the QA Officer.
 - 2.18.4.2 The QA Officer's absence will be filled by the Technical Director or Laboratory Supervisor.
 - 2.18.5 For AEL Gainesville

2.18.5.1 The Technical Director's absence will be filled by the Deputy QA Officer.

2.18.5.2 The Deputy QA Officer's absence will be filled by the Technical Director or Laboratory Supervisor.

2.18.6 AEL Orlando

2.18.6.1 Since there is limited personnel in Orlando, the Technical Director's absence will be covered by the Tampa Technical Director.

2.18.6.2 QA issues are the responsibility of the Tampa QA Officer in conjunction with the Orlando Deputy QA Officer. In the event of both their absences, QA responsibilities will be assigned by the Corporate Technical Director.

2.18.7 AEL Tallahassee

2.18.7.1 Since there is limited personnel in Tallahassee, the Technical Director's absence will be covered by the Jacksonville Technical Director.

2.18.7.2 QA issues are the responsibility of the Tallahassee Technical Director in conjunction with the Corporate Technical Director.

2.18.7.3 In the absence of the Corporate Technical Director, the Jacksonville QA Officer will be responsible for the QA issues.

2.18.8 For AEL Miami

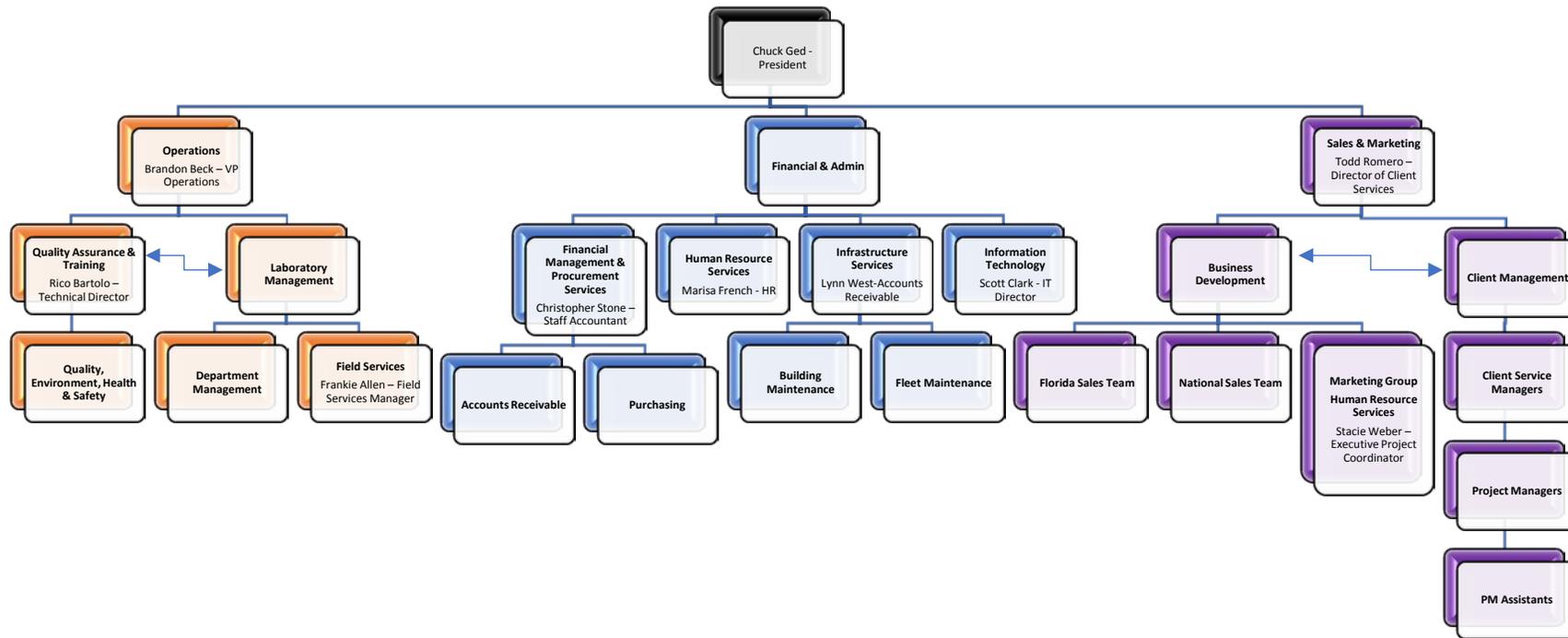
2.18.8.1 The Technical Director's absence will be filled by the QA Officer or Deputy Technical Director, which could be a Department Manager.

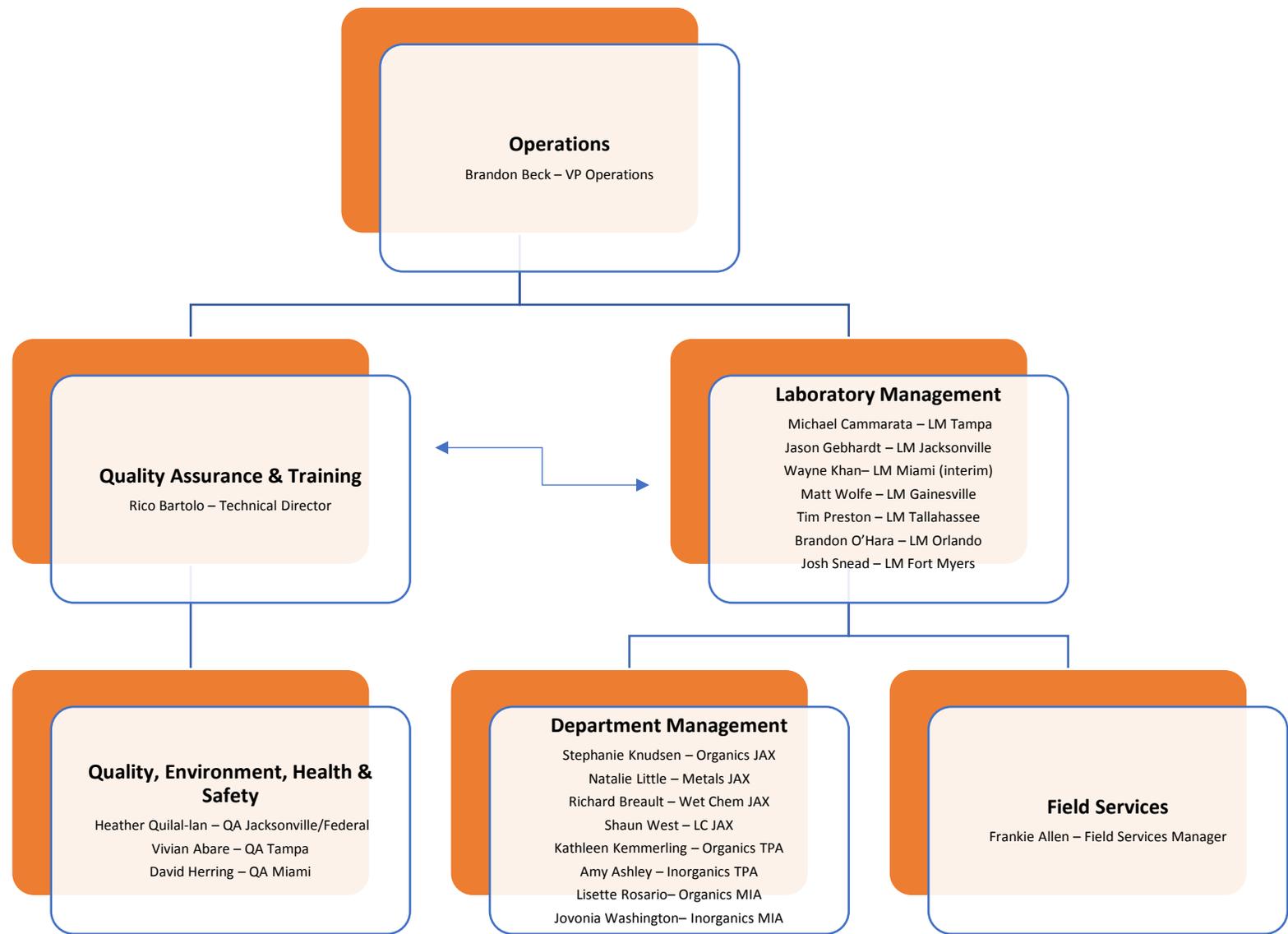
2.18.8.2 The QA Officer's absence will be filled by the Technical Director or Deputy QA Officer, which could be a Department Manager.

2.18.9 AEL Fort Myers

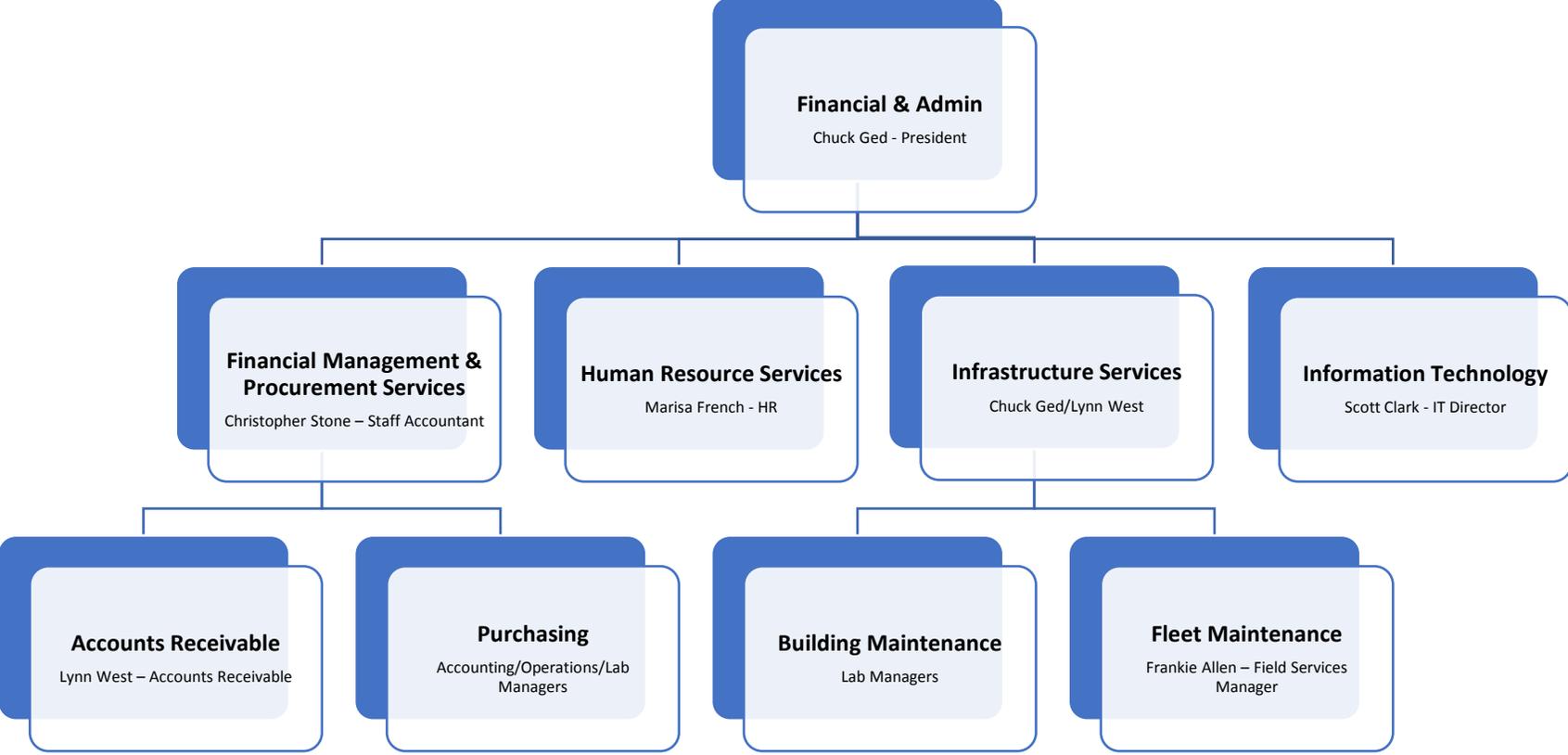
2.18.9.1 Since there is limited personnel in Fort Myers, the Technical Director's absence will be covered by the Inorganics Manager who is the appointed Deputy Technical Director. In the event of both their absences, technical managerial responsibilities will be assigned by the Corporate Technical Director.

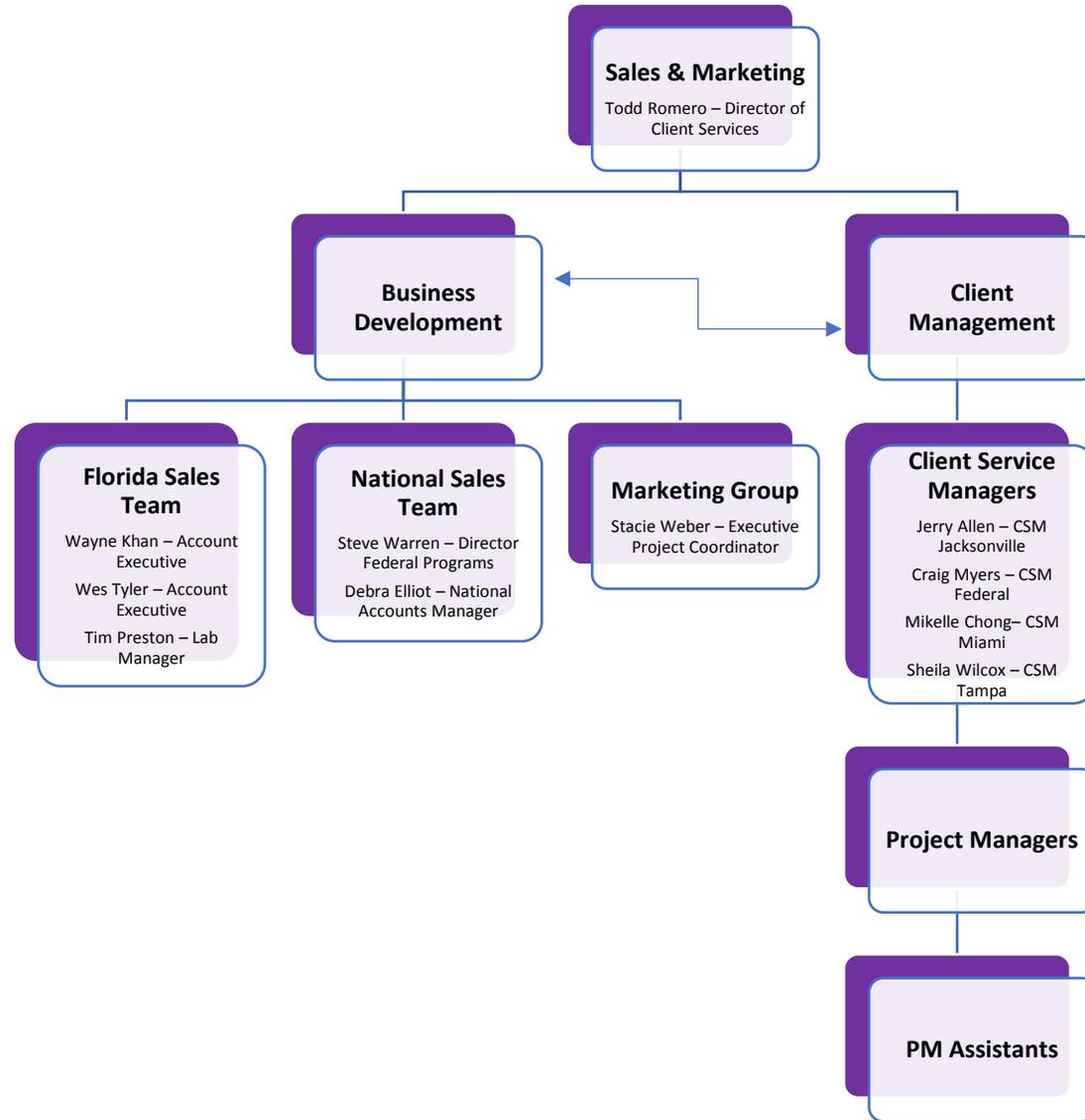
2.18.9.2 QA issues are the responsibility of the Miami QA Officer in conjunction with the Fort Myers Inorganics Manager who is the appointed Deputy QA Officer. In the event of both their absences, QA responsibilities will be assigned by the Corporate Technical Director.





Operations Department





Work Groups in Organizations

- Work teams and groups: (a) are composed of two or more individuals, (b) who exist to perform organizationally relevant tasks, (c) share one or more common goals, (d) interact socially, (e) exhibit task interdependencies (i.e., workflow, goals, outcomes), (f) maintain and manage boundaries, and (g) are embedded in an organizational context that sets boundaries, constrains the team, and influences exchanges with other units in the broader entity (Alderfer, 1977; Hackman, 1987; Hollenbeck, Ilgen, Sego, Hedlund, Major, & Phillips, 1995; Kozlowski, Gully, McHugh, Salas, & Cannon-Bowers, 1996a; Kozlowski, Gully, Nason, & Smith, 1999; Salas, Dickinson, Converse, & Tannenbaum, 1992).
- Groups cut across the company structure and can be composed of members from all levels of the organizational hierarchy.
- Strategic selection of group typologies provide strong structural support to the organizational hierarchy, encourage socialization and involve members in the decision making process.
- Groups go through the developmental stages of forming, storming, norming, and performing.

| Executive | Management & Leadership | Production & Development | Strategic Planning & Advisory | Sales Leads & Opportunities | Bids & Proposals | Quality |
|---|--|--|--|--|---|---|
| <ul style="list-style-type: none"> •President (Lead) •VP-Operations •Director of Client Services | <ul style="list-style-type: none"> •VP-Operations (Lead) •Director of Client Services (Lead) •Technical Director •Lab Managers •Client Service Managers •Department Managers | <ul style="list-style-type: none"> •VP-Operations (Lead) •Technical Director •Lab Managers •Department Managers •IT | <ul style="list-style-type: none"> •President (Lead) •VP •Director of Client Services •Lab Managers •Technical Director •Human Resources | <ul style="list-style-type: none"> •Director of Client Services (Lead) •Sales Teams •Client Service Managers •Lab Managers | <ul style="list-style-type: none"> •Director of Client Services (Lead) •Sales Teams •Marketing | <ul style="list-style-type: none"> •Technical Director (Lead) •QAs •Lab Managers |

Work Groups

3.0 Employee Training

- 3.1 Employee Training Files. Designated folders on the Q Drive will be used to collect training records for each employee. Some signed statements will be kept as Human Resources (HR) records with the Lab Manager and/or the HR Department.
- 3.2 At the time of hiring, all employees follow the process outlined below. Each employee has his or her own employee-training file, which will contain the following information.
 - 3.2.1 The Employee Training Files are broken down as follows.
 - 3.2.1.1 Resume/Ethics & Confidentiality Statements
 - 3.2.1.2 iDOCs (initial Demonstrations of Capability)
 - 3.2.1.3 cDOCs (continuing Demonstrations of Capability)
 - 3.2.1.4 Miscellaneous Training Documents
 - 3.2.1.5 Introduction to the Department Binder (Specific to the Department)
 - 3.2.2 The training files are maintained current as part of the responsibility of the department supervisor, with oversight coming from the QA Department.
 - 3.2.2.1 It is the analyst's responsibility to keep up with the training documents as completed, but the supervisor is responsible for the accuracy and completeness of the file.
 - 3.2.2.2 These files are stored in the custody of the QA officer and are reviewed periodically by the QA Officer or QA Deputy to ensure they are being maintained current. Any deficiencies will be immediately reported to the supervisor for resolution and these findings will be documented.
 - 3.2.2.3 A QA representative will review the files on a semi-annual basis, where one review can coincide with the internal audit. The review can be more frequent if there are routine errors or omissions.
 - 3.2.3 All documents maintained in the training files will need to be re-signed with each revision of the pertaining document.
 - 3.2.4 The signature of the analyst on all documents maintained in the training files signify the analyst/employee has read, understood, and agreed to abide the policy/procedure in place.
- 3.3 Resume/Ethics & Confidentiality Statements originals will be kept with the Lab Managers and/or the Company President. Electronic copies of the signed Ethics & Confidentiality Statements shall be kept on the Q drive.
 - 3.3.1 Resume. A condensed version of each employee's resume will be included. This resume is specifically formatted to ensure the education

and qualification requirements listed in section 2.0 are fulfilled for each employee.

- 3.3.2 Code of Ethics Statement. Each employee is required to sign this document at the time of employment, as seen in Figure 1.1, Section 1.0 of this Quality Manual, after reading sections 1.1 through 1.2 of this Quality Manual.
- 3.3.3 Confidentiality Statement. Each employee is asked to sign this document, as seen in Figure 1.2, Section 1.0 of this Quality Manual, after reading section 1.2 of this Quality Manual.

3.4 Analyst Statements for SOPs/QM

SOPs and the Quality Manual are accessed electronically through a desktop icon or intranet menu located on each analyst's computer.

- 3.4.1 Each analyst upon opening the Quality Manual and/or SOP must respond must agree to read and follow the document as written. Those agreement records are electronically stored.
- 3.4.2 Standard Operating Procedures (SOPs). There are three types of SOPs used by AEL: administrative, technical, and analytical. See SOP ADMIN-025 for instruction on writing SOPs.
 - 3.4.2.1.1 As each SOP is created or revised, a global e-mail to all employees is issued by the Corporate QA. All old versions are removed and access to the newly created or revised SOP must be acknowledged through Intranet SOP Review and Acknowledgement System. Personnel shall read those SOP that are pertinent to the duties they perform.
 - 3.4.2.2 Administrative
 - 3.4.2.2.1 Administrative SOPs cover all administrative aspects of how the laboratory functions and sets the policies of the laboratory.
 - 3.4.2.2.2 These cover everything from data entry to significant figures to non-conformities, etc.
 - 3.4.2.2.3 See Table 3.1 at the end of this section for a listing of all current Administrative SOPs.
 - 3.4.2.2.4 It is the responsibility of AEL Management to ensure that all administrative SOPs are current, accurate, and up to date with the Quality System.

3.4.2.3 Technical

- 3.4.2.3.1 Technical SOPs cover technical aspects and/or basic laboratory procedures and sets the policies of the laboratory.
- 3.4.2.3.2 Technical SOPs cover a broad range of subjects from glassware cleaning and phone usage to choosing the peaks for organics on multiplex chromatograms.
- 3.4.2.3.3 See Table 3.2 at the end of this section for a listing of all current Administrative SOPs.

3.4.2.4 Analytical

- 3.4.2.4.1 Analytical SOPs are based on the method being analyzed. These methods are EPA Methods for Chemical Analysis of Waters and Wastewaters, EPA SW-846 Methods, or Standard Methods.
- 3.4.2.4.2 These SOPs are the basis for performing every method of analysis within AEL. They are based off of the base EPA or Standard Method method and define exactly how AEL performs the analysis.
- 3.4.2.4.3 These methods are identified according to the department performing the analysis. Listings of the different methods are listed as Tables 3.3 through 3.7.
- 3.4.2.4.4 It is the supervisor's responsibility to ensure the analytical SOPs are accurate and up to date with any revisions or changes that are necessary.

3.4.2.5 Initial training with SOPs

- 3.4.2.5.1 Once a new employee is hired or switches to a different department, they will be required to read all applicable SOPs before beginning analysis training. Documentation of the analyst reading the SOP is documented through Intranet SOP Review and Acknowledgement System.
- 3.4.2.5.2 The supervisor is responsible for ensuring that all new analysts are properly trained and familiar with the SOPs before allowing any analysis to be completed by that analyst.

3.4.2.5.3 Once the training is complete and the analyst feels comfortable with the method and the supervisor is comfortable with the analyst's training, the initial demonstration of capability process is begun. This process is defined in Section 3.5.

3.4.2.6 Additional training

3.4.2.6.1 Once the analyst is sufficiently trained, their performance will be monitored by the proficiency-testing program as mentioned in Section 3.10 and by the continuing demonstration of capability process as explained in Section 3.6.

3.4.2.6.2 Additional training is accomplished by any of the following;

3.4.2.6.2.1 Trade seminars

3.4.2.6.2.2 Instrument Manufacturer workshops

3.4.2.6.2.3 On the job training

3.4.2.6.2.4 Training meetings held by management

3.4.2.6.3 Any additional training is documented, and a record is stored in the employee-training file.

3.5 iDOCs (initial Demonstrations of Capability) training records are tracked on a spreadsheet kept with the pdf copies of each analyst's iDOC by department and lab are saved on the Q drive by lab. The DOC process is outlined in SOP ADMIN-030.

3.5.1 Initial Demonstration of Capability (iDOC)

3.5.1.1 Every new analyst performs these before they begin any analysis on their own and report results.

3.5.1.2 The iDOC is completed according to TNI 2016 Standards Modules 4 for Chemical Testing and Module 5 for Microbiology. (See attached Figure 3.1 at the end of this section.)

3.5.1.3 In most cases, the iDOC will include the spiking of 4 aliquots of clean quality system matrix and comparing their recoveries to acceptance criteria for the method and matrix used. However, there are some methods and matrices that do not lend themselves to spiking, so an alternate procedure to prove an analyst's capability to perform the test properly is required. That criteria will be fully listed in the SOP of the following methods and summarized here:

Alternate iDOC criteria

Microbiology Methods:

Most Microbiology procedures can now be referenced through 2016 TNI Standards Volume 1 Module 5.

For all qualitative analysis such as SM9223B (Colilert), a blind study is to be performed, with a minimum of a blank, a negative culture, and a positive culture.

General Chemistry Methods:

Paint Filter Liquids Test SW9095, Odor EPA 140.1 and SM2150B, Total Solids (Total Residue) EPA 160.3/SM 2540B, Settleable Solids EPA 160.5, SM 2540F, SW1311, and SW1312, total nitrogen by calculation as well as hardness by calculation method SM2340B.

For the **paint filter test**, the Demonstration of Capability will consist of repeating the performance of an experienced analysis on 4 client samples within 10% on all samples. If unable to match the performance of the experienced analyst, then further training will be required.

The demonstrations of capability **for odor** are to be performed for two areas of performance. The first area is preparing the samples for analysis, the second is for successfully identifying positive/negative presence for odor by smelling the prepared samples. To demonstrate the ability to prepare samples correctly, the individual(s) must be shown under the direction of an experienced analyst how to set up a bank of positive and negative controls for testing by those to smell the samples. Those preparing the samples cannot be included in the group to smell samples. If those individuals who are preparing the bank of samples wish to also smell, a separate bank of samples should be prepared for them independently from those prepared for the initial group. The smell testers should not have knowledge of the number of positive samples or contents of the test samples. The preparer must also demonstrate capability by preparing actual client samples side by side with an experienced analyst and computing the results such that they match those of the experienced analyst. The test samples shall consist of two blanks and five DI water samples spiked with a food extract (Vanilla). Three DOC samples are to be spiked at the high range (100uL of pure vanilla extract in 200mL volumetric) and two DOC samples at the low range (50uL of pure vanilla extract in 200mL volumetric).

To demonstrate the ability to adequately smell test for the presences of odors, the individual(s) will need to be able to identify correctly at least 6 out of 7 samples and must positively identify the 3 samples at the high standard.

For **Total Solids (Total Residue) under EPA 160.3/SM 2540B**, the Demonstration of Capability will consist of repeating the performance of an experienced analysis on 4 client samples within 10% on all samples. If unable to match the performance of the experienced analyst, then further training will be required. If performing the test for the first time without the aide of an experienced analyst, perform 4 replicates on a representative soil sample (such as a soil sample from the lab's lake edge or landscaping area) and analyze with a %RSD of 10% or less.

For **Settleable Solids EPA 160.5 and SM2540F**, to perform the DOC, the analyst must perform a parallel study. Two analysts will set up the same sample and compare results. The RPD must be \leq 20%.

For **total nitrogen calculation**, the iDOC consists of taking the results of the iDOCs for methods EPA 353.2 Nitrate/nitrite and EPA 351.2 TKN and adding them together for total nitrogen.

For **total hardness calculation by SM2340B**, the iDOC consists of taking the results of the iDOCs for either methods EPA 200.7 or 6010 for calcium and magnesium and carrying them through the total hardness calculation.

For **Chlorophylls and Pheophytin by SM 10200H**, initial and continuing demonstrations of capability are performed by duplicate analysis. The analyst performing the DOC must run duplicates of 4 separate samples, which have also been run by an experienced analyst. The DOC is successful if the duplicate recovery is within 35% of the value obtained by the experienced analyst for each of the samples analyzed. If any samples fail this requirement the DOC must be re-performed in its entirety.

For **TCLP and SPLP** a sample with a suspected high concentration of target analytes (one that should produce results above the PQL of the analytes with the extraction process) shall be run in duplicate by the experienced analyst and the average of those recoveries are to be compared to the duplicate average of the trainee. The RPD of the average recoveries between the experienced analyst and the trainee shall be $<30\%$ for any analyte recovered above the PQL.

For **Ignitability** by EPA 1030 two substances will be tested in duplicate for ignitability. One substance shall be chosen that would not normally ignite, such as sand, and the other substance should ignite, such as a

paper product. Documenting this process using the correct procedure and forms constitutes the IDOC for method.

3.5.1.4 For department with multiple analysts performing the same tasks, the DOC process will be completed in the following manner.

3.5.1.4.1 Each extractionist doing the extraction will demonstrate that they can perform the extraction method.

3.5.1.4.2 Each analyst performing the analytical method will demonstrate that they can acceptably perform each method they are analyzing.

3.5.1.4.3 This does not mean there will be an iDOC for each extractionist/analyst combination in the department. Every analyst will have an iDOC for each analytical method and each extractionist will have an iDOC for each preparation method, but not every possible analyst/extractionist combination therein.

3.5.1.5 Analyst Training Documentation

3.5.1.5.1 This document contains the initials and dates of the analyst and supervisor/experienced analyst responsible for the training.

3.5.1.5.2 This document has different aspects of the training process that must be documented as each step of the training is completed and signed by all responsible parties.

3.6 cDOCs (continuing Demonstrations of Capability) are tracked on the iDOC spreadsheets for each lab. The spreadsheet spells out what method was used for obtaining the cDOC, as described below, most commonly by successfully performing a PT, by passing four consecutive separate source LCS, or by a re-performance of the iDOC.

3.6.1 Continued Demonstration of Capability (cDOC)

3.6.1.1 These are required on an annual basis as a continued monitoring that the analyst is continually able to perform the method satisfactorily.

3.6.1.2 These can be accomplished by a number of methods as well and are again mentioned in Figure 1.0. Some examples are:

3.6.1.2.1 Proficiency testing studies performed

3.6.1.2.2 Re-performance of the iDOC.

3.6.1.2.3 For methods that require laboratory control spikes (LCS) to be analyzed, the analyst can choose 4 recovery sets to process as a cDOC to verify acceptable performance within the acceptance limits established by that method.

3.6.1.2.3.1 Those limits can either be in-house limits or TNI/method defined limits.

3.7 Employee Training Files can also consist of the following.

3.7.1 Specific action based on an Internal audit finding.

3.7.2 Annual training classes involving data integrity or basic laboratory skills.

3.7.3 Documentation for any seminars or training classes that have been attended.

3.7.4 Any other training related document received or acquired by the employee during their tenure.

3.8 Employee Handbook. Upon hiring all employees are provided with or given access to a copy of the handbook titled "Policies and Procedures Manual". Once they have read this document, they are required to sign a document stating they have read, understand, and agree to abide by the policies listed out in this document.

3.9 AEL Quality Manual. The Quality Manual is accessed electronically through the intralab network available on each analyst's computer. Each analyst upon opening the Quality Manual must respond must agree to read and follow the document as written in order to access. Those agreement records are electronically stored.

3.10 Proficiency Testing (PT) Samples document another aspect of training that is utilized and provides a basis for determining an analyst's accuracy with a given method.

3.10.1 As defined in TNI Standards Volume 1, Module 1, all environmental testing laboratories are required to participate in these studies on a routine basis.

3.10.2 The laboratory must analyze and pass two PT samples per fiscal year for each technology/method combination that the lab is certified for and which has the compound listed in the TNI PT required Fields of Testing. These PTs are performed in compliance with the TNI 2016 Standards, Volume 1, Module 1.

3.10.3 These standards are ordered from NVLAP/TNI approved providers.

3.10.4 The PT Program in place for AEL is managed by the QA Officer and is explained in detail in Section 9 of this Quality Manual.

3.10.5 PT Failures

3.10.5.1 All PT Failures require a corrective action as to the suspected cause of the error.

3.10.5.2 The corrective action is submitted to the QA officer who will generate an NCF and track the corrective process through the NCF program.

3.10.5.3 It is a requirement of TNI Standards that the lab must have acceptable results on 2 out of the past 3 PT results reported to show continued proficiency and maintain certification. If the laboratory misses two successive or two out of 3 PT results, a quick response PT will be ordered as soon as the non-compliance is noted. Once the quick response PT is completed acceptably, the result is submitted to the accrediting authority as a makeup to replace the most recent failure and prove proficiency is re-established.

3.10.5.4 The corrective actions are the responsibility of the supervisor for completeness and accuracy. The Lab Manager has final review of all corrective actions

3.10.5.5 Repeated PT failures or careless mistakes will involve potential disciplinary action or a transfer of an employee as part of a corrective action.

3.11 Hiring of Employees

3.11.1 When AEL determines it is time to add personnel to the staff of AEL, it will use normal methods of gathering resumes of potential employees, such as placing ads or posting for the position.

3.11.2 Once the resumes are gathered, they are reviewed by multiple personnel, (middle management or higher), to ensure the training and experience is adequate for the position sought.

3.11.3 Once a list of potential employees is generated, AEL performs an interview process that involves multiple personnel taking part in the interview to ensure the potential employee is the right fit for the position.

3.11.4 Once the decision is made and the employee is hired, he or she undergoes the steps outlined earlier in this section to ensure all initial requirements are met before beginning analysis or performing any procedures.

3.12 Promoting from within

- 3.12.1 AEL prefers to promote from within the AEL network for filling more experienced positions. This is accomplished by recommendations from supervisors or lab managers for promotion.
- 3.12.2 The interview process is the same as listed above and all aspects of the new position are explained and understood before the position is offered.
- 3.12.3 If it is determined the employee would meet the job requirements as outlined in Section 2.0 of this Manual and the interviewing personnel ascertain that the employee would be beneficial in the new position, the promotion will be made.
- 3.12.4 If this promotion requires replacement of a less-experienced employee, then the procedure in Section 3.11 will be followed unless another promotion can be performed to fill the void.

3.13 Administrative and Technical SOP Responsibilities.

- 3.13.1 The following outline determines the training of each SOP dependant upon the position held.

3.13.1.1 Extractionists/Entry level-analysts

- 3.13.1.1.1 ADMIN-008 Data Qualifiers
- 3.13.1.1.2 ADMIN-010 Internal Data Review
- 3.13.1.1.3 ADMIN-011 Significant Figures
- 3.13.1.1.4 ADMIN-012 MDLs, LODs, PQLs, and LOQs
- 3.13.1.1.5 ADMIN-013 Ordering Supplies and Reagents
- 3.13.1.1.6 ADMIN-016 Complaints, NCFs, Corrective Action
- 3.13.1.1.7 ADMIN-018 Waste Disposal
- 3.13.1.1.8 ADMIN-022 Document Control
- 3.13.1.1.9 ADMIN-024 Estimated Levels of Uncertainty
- 3.13.1.1.10 ADMIN-025 Writing and Revising SOPs
- 3.13.1.1.11 ADMIN-027 Data Quality Objectives
- 3.13.1.1.12 ADMIN-028 Case Narratives
- 3.13.1.1.13 ADMIN-029 Backup and Restoring Data
- 3.13.1.1.14 ADMIN-030 Demonstrations of Capability
- 3.13.1.1.15 ADMIN-031 Receipt of Consumables
- 3.13.1.1.16 ADMIN-033 Acceptance Criteria and Charts
- 3.13.1.1.17 ADMIN-035 Ethics and Data Integrity
- 3.13.1.1.18 ADMIN-036 Defining the Default Date Format
- 3.13.1.1.19 ADMIN-037 Foreign Soils
- 3.13.1.1.20 ADMIN-038 Calibration, Integration, Chromatography
- 3.13.1.1.21 ADMIN-039 Lab Definitions
- 3.13.1.1.22 ADMIN-040 Analysis under DoD QSM
- 3.13.1.1.23 ADMIN-046 Chemical Spill

- 3.13.1.1.24 TECH-001 Glassware Cleaning
- 3.13.1.1.25 TECH-002 De-ionized Water Maintenance
- 3.13.1.1.26 TECH-004 Balance Operation
- 3.13.1.1.27 TECH-005 Pipette Use and Calibration
- 3.13.1.1.28 TECH-006 Thermometer Use and Calibration
- 3.13.1.1.29 TECH-007 Syringe Use and Calibration
- 3.13.1.1.30 TECH-008 Volumetric Glassware Use and Calibration
- 3.13.1.1.31 TECH-009 Multiple Peak Compound Identification
- 3.13.1.1.32 TECH-010 Retention Time Windows
- 3.13.1.1.33 TECH-013 Measuring pH in Di water

3.13.1.2 Senior Analysts/Supervisors. The SOPs for required reading will be those listed for the extractionists/entry-level analysts in addition to the following:

- 3.13.1.2.1 ADMIN-005 & 5a Sample Receipt and Log-in
- 3.13.1.2.2 ADMIN-014 Certification Flags
- 3.13.1.2.3 ADMIN-020 Annual Internal Audit
- 3.13.1.2.4 ADMIN-021 Annual Managerial Review
- 3.13.1.2.5 ADMIN-026 Electronic Signatures
- 3.13.1.2.6 ADMIN-041 Client Services Objectives

- 3.13.1.2.7 TECH-009 Multipeak Compound Identification (Organics)
- 3.13.1.2.8 TECH-010 Retention Time Windows
- 3.13.1.2.9 TECH-011 Organics for Project Managers
- 3.13.1.2.10 TECH-012 Metals for Project Managers
- 3.13.1.2.11 TECH-015 Wet Chemistry for Project Managers

3.13.1.3 Project Managers. The SOPs for required reading will be those listed for the extractionists/entry-level analysts in addition to the following:

- 3.13.1.3.1 ADMIN-001 Telephone Use
- 3.13.1.3.2 ADMIN-005 & 5a Sample Receipt and Log-in
- 3.13.1.3.3 ADMIN-014 Certification Flags
- 3.13.1.3.4 ADMIN-020 Annual Internal Audit
- 3.13.1.3.5 ADMIN-021 Annual Managerial Review
- 3.13.1.3.6 ADMIN-026 Electronic Signatures
- 3.13.1.3.7 ADMIN-041 Client Service Objectives

- 3.13.1.3.8 TECH-011 Organics for Project Managers
- 3.13.1.3.9 TECH-012 Metals for Project Managers
- 3.13.1.3.10 TECH-015 Wet Chemistry for Project Managers

3.13.1.4 Sample Receipt/Front Office

- 3.13.1.4.1 ADMIN-001 Telephone Use
- 3.13.1.4.2 ADMIN-005 & 5a Sample Receipt and Log-in
- 3.13.1.4.3 ADMIN-014 Certification Flags
- 3.13.1.4.4 ADMIN-014 Manual Log-in
- 3.13.1.4.5 ADMIN-020 Annual Internal Audit
- 3.13.1.4.6 ADMIN-021 Annual Managerial Review
- 3.13.1.4.7 ADMIN-023 Sample Kit Preparation
- 3.13.1.4.8 ADMIN-026 Electronic Signatures

3.13.1.5 Upper/Senior Management (Lab Managers, Lab Supervisors, QA Department, Operations Manager, Lab Director)

- 3.13.1.5.1 All ADMIN SOPs
- 3.13.1.5.2 All TECH SOPs

3.13.1.6 Field Personnel

- 3.13.1.6.1 ADMIN-005 & 5a Sample Receipt and Log-in
- 3.13.1.6.2 ADMIN-023 Sample Kit Preparation
- 3.13.1.6.3 ADMIN-035 Ethics and Data Integrity
- 3.13.1.6.4 ADMIN-039 Lab Definitions

3.13.1.7 Sales Staff

- 3.13.1.7.1 ADMIN-001 Telephone Use
- 3.13.1.7.2 ADMIN-026 Electronic Signatures
- 3.13.1.7.3 ADMIN-035 Ethics and Data Integrity
- 3.13.1.7.4 ADMIN-039 Lab Definitions

- 3.13.1.7.5 TECH-011 Organics for Project Managers
- 3.13.1.7.6 TECH-012 Metals for Project Managers

Figure 3.1

From TNI Standard EL-V1M4-2016: Chemical Testing

Initial DOC

1.6.2 An individual must successfully perform an initial IDOC prior to using any method, and any time there is a change in instrument type, method, or any time that a method has not been performed by an analyst in a twelve (12) month period.

1.6.2.1 The laboratory shall document each initial DOC in a manner such that the following information is readily available for each affected employee:

- a) analyst(s) involved in preparation and/or analysis;
- b) matrix;
- c) analyte(s), class of analyte(s), or measured parameter(s);
- d) identification of test method(s) performed;
- e) identification of laboratory-specific SOP used for analysis, including revision number;
- f) date(s) of analysis; and
- g) summary of analyses, including information outlined in Section 1.6.2.2.c.

1.6.2.2 If the method or regulation does not specify a DOC, the following procedure is acceptable. It is the responsibility of the laboratory to document that other approaches to DOC are adequate.

a) The analyte(s) shall be diluted in a volume of clean quality system matrix (a sample in which no target analytes or interferences are present at concentrations that will impact the results of a specific test method) sufficient to prepare four (4) aliquots at the concentration specified, or if unspecified, to a concentration of one (1) to four (4) times the limit of quantitation.

b) At least four (4) aliquots shall be prepared and analyzed according to the test method(s) either concurrently or over a period of days.

c) Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations of the sample (in the same units) for each parameter of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence and logarithmic values, the laboratory shall assess performance against established and documented criteria.

d) Compare the information from (c) above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if there are not established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters does not meet the acceptance criteria, the performance is unacceptable for that parameter.

e) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst shall proceed according to i) or ii) below.

- i) Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with b) above.
- ii) Beginning with b) above, repeat the test for all parameters that failed to meet criteria.

f) Repeated failure, however, confirms a general problem with the measurement system. If this occurs, locate, and correct the source of the problem and repeat the test for all compounds of interest beginning with b).

g) When an analyte not currently found on the laboratory's list of accredited analytes is added to an existing accredited test method, an initial demonstration shall be performed for that analyte.

From TNI Standard EL-V1M5-2016: Microbiological Testing

1.6.2 Initial DOC

An initial DOC shall be made prior to using any method, and at any time there is a change in instrument type, personnel or method or any time that a method has not been performed by the laboratory or analyst in a twelve (12) month period.

1.6.2.1 The laboratory shall document each initial DOC in a manner such that the following information is readily available for each affected employee:

- a) analyst(s) involved in preparation and/or analysis;
- b) matrix;
- c) organism(s);
- d) identification of method(s) performed;
- e) identification of laboratory-specific SOP used for analysis, including revision number;
- f) date(s) of analysis;
- g) summary of analyses, including information outlined in Section 1.6.2.2.c.

1.6.2.2 If the method or regulation does not specify an initial DOC, the following procedure is acceptable. It is the responsibility of the laboratory to document that other approaches to initial DOC are adequate.

a) The target organism(s) shall be diluted in a volume of clean quality system matrix (a sample in which no target organisms or interferences are present at concentrations that will impact the results of a specific method). This matrix shall be sterile phosphate or sterile peptone solution unless specified by the manufacturer. Prepare at least four (4) aliquots at the concentration specified, or if unspecified, to the countable range for plate methods or working range for most probable number (MPN) type methods.

b) At least four (4) aliquots shall be prepared and analyzed according to the method either concurrently or over a period of days.

c) Using all of the results, convert these results to logarithmic values, then calculate the mean recovery and standard deviation of the log converted results in the appropriate reporting units for each organism of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence, the laboratory shall assess performance against established and documented criteria.

d) For qualitative tests, acceptable performance in a blind study, either internally or externally generated, may be used to meet this Standard, provided that the study consists of a minimum of a blank, a negative culture, and a positive culture for each target organism.

e) Compare the information from c) above to the corresponding acceptance criteria for precision and accuracy in the method (if applicable) or in laboratory-generated acceptance criteria (if there are not established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters does not meet the acceptance criteria, the performance is unacceptable for that parameter.

f) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst shall proceed according to i) or ii) below.

- i) Locate and correct the source of the problem and repeat the initial DOC for all parameters of interest beginning with b) above.
- ii) Repeat the initial DOC for all parameters that failed to meet criteria.

g) Repeated failure, however, confirms a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with b).

Table 3.1:

Table of Administrative SOPs

All SOPs will be assigned a number in the form of Admin-### and a listing will be kept in the table below. The listed SOP dates and revisions are the most current at the time of this QM revision. As the SOPs are reviewed and revised on a routine basis, the most current listings of SOPs are available on each analyst's desktop under the SOP Icon and on the designated Quality Assurance (Q) drive of the AEL networked servers.

| SOP # | Title |
|--------------|---|
| Admin-001 | Telephone Use |
| Admin-005 | Sample Receipt and Log-in |
| Admin-005a | Sample Login Policies |
| Admin-008 | Data Qualifiers |
| Admin-010 | Internal Data Review |
| Admin-011 | Significant Figures |
| Admin-012 | MDLs, LODs, PQLs, and LOQs |
| Admin-013 | Ordering of Supplies and Reagents |
| Admin-014 | Certification Flags |
| Admin-016 | Complaints Tracking, NCFs, and Corrective Action |
| Admin-018 | Waste Disposal and Pollution Prevention |
| Admin-019 | Manual Log-in |
| Admin-020 | Annual Internal Audit |
| Admin-021 | Annual Management Review |
| Admin-022 | Document Control |
| Admin-023 | Sample Kit Preparation |
| Admin-024 | Estimated Levels of Uncertainty |
| Admin-025 | Writing Administrative, Analytical, and Technical SOPs |
| Admin-026 | Electronic Signatures |
| Admin-027 | Data Quality Objectives |
| Admin-028 | Case Narratives |
| Admin-029 | Daily Backup and Restoring of Electronic Data |
| Admin-030 | Demonstrations of Capability |
| Admin-031 | Receipt of Consumable Items |
| Admin-033 | Acceptance Criteria and Control Charts |
| Admin-035 | Ethics & Data Integrity, Proactive Fraud Prevention & Detection |
| Admin-036 | Defining the Default Date Format |
| Admin-037 | Receipt, Handling, and Disposal of Foreign Soils |
| Admin-038 | Calibration, Manual Integration, and Rules for Chromatography |
| Admin-039 | Laboratory Definitions |
| Admin-040 | Analysis under DoD QSM |
| Admin-041 | Client Service Objectives |
| Admin-042 | EDD Reports |
| Admin-043 | Legal Chains of Custody |
| Admin-044 | Software Development and Testing |
| Admin-045 | Receipt of DOD Samples |
| Admin-046 | Documenting a Chemical Spill or Sample Spill |
| Admin-047 | Reporting DoD Sample Analysis |

Table 3.2: **Table of Technical SOPs**

All SOPs will be assigned a number in the form of TECH-### and a listing will be kept in the table below. The listed SOP dates and revisions are the most current at the time of this QM revision. As the SOPs are reviewed and revised on a routine basis, the most current listings of SOPs are available on each analyst's desktop under the SOP Icon and on the designated Quality Assurance (Q) drive of the AEL networked servers.

| SOP # | Title |
|----------|---|
| Tech-001 | Glassware Cleaning |
| Tech-002 | De-ionized Water Maintenance |
| Tech-003 | Annual Balance Calibration Verification |
| Tech-004 | Balance Operation |
| Tech-005 | Pipette Use and Calibration |
| Tech-006 | Thermometer Use and Calibration |
| Tech-007 | Syringe Use and Calibration |
| Tech-008 | Volumetric Glassware Use and Calibration |
| Tech-009 | Multi-Peak Compound Identification for Organics |
| Tech-010 | Establishing and Maintaining Retention Time Windows |
| Tech-011 | Organics for Project Managers |
| Tech-012 | Metals for Project Managers |
| Tech-013 | Measuring pH in Di Water |
| Tech-014 | QA Duties and Responsibilities |
| Tech-015 | Wet Chemistry for Project Managers |

Table 3.3: **Table of METALS SOPs**

All SOPs will be assigned a number in the form of MET-### and a listing will be kept in the table below. The listed SOP dates and revisions are the most current at the time of this QM revision. As the SOPs are reviewed and revised on a routine basis, the most current listings of SOPs are available on each analysts desktop under the SOP Icon and on the designated Quality Assurance (Q) drive of the AEL networked servers.

| SOP # | Title |
|---------|--|
| MET-001 | EPA SW3005A |
| MET-002 | EPA SW3010A |
| MET-003 | EPA SW3020A |
| MET-004 | EPA SW3050B |
| MET-005 | EPA SW7470A |
| MET-006 | EPA E245.1 |
| MET-008 | EPA E200.7 |
| MET-009 | EPA SW6010B |
| MET-010 | EPA SM3113B |
| MET-013 | EPA 200.9 Graphite Furnace GFAA |
| MET-015 | EPA 200.2 Digestion for Total & Recoverable Metals |
| MET-016 | EPA SW6020 |
| MET-017 | EPA E200.8 |
| MET-018 | SM 2340B |
| MET-024 | EPA SW7471A for Hg Soils |
| MET-025 | EPA 1631-E for Low Level Mercury |
| MET-026 | EPA 3060A Alkaline Digestion for Hexavalent Chromium |

Table 3.4:

Table of Microbiological SOPs

All SOPs will be assigned a number in the form of MICRO-### and a listing will be kept in the table below. The listed SOP dates and revisions are the most current at the time of this QM revision. As the SOPs are reviewed and revised on a routine basis, the most current listings of SOPs are available on each analyst's desktop under the SOP Icon and on the designated Quality Assurance (Q) drive of the AEL networked servers.

| SOP # | Title |
|--------------|--|
| MICRO-003 | HPC by SM9215B |
| MICRO-008 | Total Coliform Membrane Filter Analysis SM9222B |
| MICRO-009 | Fecal Coliform Membrane Filter Analysis SM9222D |
| MICRO-010 | SM9223B (MMO-MUG) & Colitag |
| MICRO-012 | EPA 1603: E Coli in Water Modified Membrane-Agar |
| MICRO-013 | Total Coliform by 9221B by MPN |
| MICRO-014 | Fecal Coliform by 9221E by MPN |
| MICRO-017 | Enterococci by EPA 1600 |
| MICRO-018 | Fecal Strep by SM9230 |
| MICRO-019 | Microbiology Quality Assurance Procedures |
| MICRO-020 | SM9230C Enterococci |
| MICRO-021 | E. Coli MPN by SM9223B |
| MICRO-022 | Colilert-18 Quanti -Tray |
| MICRO-023 | Enterolert Quantit-Tray |
| MICRO-025 | E. Coli by SM9223B Quanti-Tray |
| MICRO-026 | HPC by SM9215E (Simplate) |

Table 3.5:

Table of Semi-Volatiles Department SOPs

All SOPs will be assigned a number in the form of SVOC-### and a listing will be kept in the table below. The listed SOP dates and revisions are the most current at the time of this QM revision. As the SOPs are reviewed and revised on a routine basis, the most current listings of SOPs are available on each analyst's desktop under the SOP Icon and on the designated Quality Assurance (Q) drive of the AEL networked servers.

| SOP Number | Title |
|------------|--|
| SVOC-001 | Method 3510C, Separatory Funnel Liquid-Liquid Extraction |
| SVOC-002 | Method 3550B-C, Sonication extraction |
| SVOC-003 | Method 3580A, Waste Dilution |
| SVOC-004 | Method FL-PRO, Florida Petroleum Range Organics by GC/FID |
| SVOC-005 | Method 8310, Polynuclear Aromatic Hydrocarbons by HPLC |
| SVOC-006 | Method 8270C&D, Semi-Volatile Organics Compounds by GC/MS |
| SVOC-007 | Method 8100, Polynuclear Aromatic Hydrocarbons by GC/FID |
| SVOC-008 | Method 8081B, Organochlorine Pesticides by GC/ECD |
| SVOC-009 | Method 8082A, PCBs by GC/ECD |
| SVOC-012 | Method 3630C, Silica Gel cleanup |
| SVOC-013 | Method 3620C, Florisil Cleanup |
| SVOC-014 | Method 3665A, Sulfuric Acid Cleanup |
| SVOC-015 | Method 504.1, EDB and DBCP in Water by Micro-extraction and Gas Chromatography |
| SVOC-016 | Method 8141B, ORGANOPHOSPHORUS COMPOUNDS |
| SVOC-017 | Method 8151A, EXTRACTION AND ANALYSIS OF HERBICIDES |
| SVOC-018 | Method 3540C, Soxhlet Extraction |
| SVOC-019 | Method 552.2, Determination of Haloacetic Acids and Dalapon in Drinking Water by Liquid-Liquid Extraction, Derivatization and Gas Chromatography with Electron Capture Detection |
| SVOC-020 | Method 515.3, Extraction and Analysis of herbicides in Drinking Water by Gas Chromatography |
| SVOC-021 | Method 525.2 Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column GC/MS |
| SVOC-022 | Method FLO-PRO Modified Diesel Range Organics by GC/FID |
| SVOC-023 | Method 547, Determination of Glyphosphate in Drinking Water by Direct Aqueous Injection HPLC, Post Column Derivatization, and Fluorescence Detection |
| SVOC-024 | Method 531.1, Measurement of N-methylcarbamoyloximes and N-methylcarbamates in Water by Direct Aqueous Injection HPLC With Post Column Derivatization |
| SVOC-025 | 508, Drinking Water Pesticides |
| SVOC-026 | 548.1 endothall by GC/MS |
| SVOC-027 | 549.2 diquat by HPLC |
| SVOC-028 | 8270-SIM, Semi-Volatile Organics by GC/MS, utilizing SIM mode |
| SVOC-029 | Method 8011, EDB and DBCP in Water by Micro-extraction and Gas Chromatography |
| SVOC-030 | Wipe sampling for 2, 4, 6 Tribromophenol |
| SVOC-032 | Method 3535A Solid Phase Extraction (SPE) for Explosives |
| SVOC-034 | Method 3660B, Sulfur cleanup |
| SVOC-036 | EXTRACTABLE PETROLEUM HYDROCARBONS (EPH) |
| SVOC-037 | Wipe Sampling for PCBs |
| SVOC-038 | EPA 625.1 |
| SVOC-039 | EPA 608.3 |
| SVOC-040 | 8015C DRO |
| SVOC-041 | PFAS by EPA 533, 533 Mod, DoD Table B-15 |

Table 3.6:

Table of Volatiles Department SOPs

All SOPs will be assigned a number in the form of VOC-### and a listing will be kept in the table below. The listed SOP dates and revisions are the most current at the time of this QM revision. As the SOPs are reviewed and revised on a routine basis, the most current listings of SOPs are available on each analyst's desktop under the SOP Icon and on the designated Quality Assurance (Q) drive of the AEL networked servers.

| SOP Number | Title |
|------------|---|
| VOC-001 | Method 5030, Sampling/Prep procedure for aqueous samples and high level soils |
| VOC-002 | Method 5035, Sampling/Prep procedure for low-level soils |
| VOC-003 | Method 8260, Volatile Organics Compounds by GC/MS |
| VOC-009 | Method 8015, VOCs by GC/FID |
| VOC-010 | 524.2 Purgeable Organic Compounds in Water by Capillary Column GC/MS |
| VOC-012 | Storage Blanks |
| VOC-013 | RSK by EPA175 |
| VOC-014 | GRO by EPA8015 |
| VOC-015 | VOLATILE PETROLEUM HYDRO CARBONS (VPH) |
| VOC-016 | THM and HAA Formation Potential by SM5710B & SM5710D |
| VOC-017 | EPA 624.1 |

Table 3.7:

Table of Wet Chemistry Department SOPs

All SOPs will be assigned a number in the form of WC-### and a listing will be kept in the table below. The listed SOP dates and revisions are the most current at the time of this QM revision. As the SOPs are reviewed and revised on a routine basis, the most current listings of SOPs are available on each analyst's desktop under the SOP Icon and on the designated Quality Assurance (Q) drive of the AEL networked servers.

| SOP # | Title |
|--------|--|
| WC-001 | Non-Filterable Residue (TSS), E160.2, SM 2540D |
| WC-002 | PH, E150.1/9040B/9045C/SM 4500H+B |
| WC-003 | Flashpoint/Ignitability, SW1010 |
| WC-004 | Oil & Grease, TPH, E1664A -SM5520B |
| WC-005 | TCLP, SW1311 |
| WC-006 | TS (%), SM2540G |
| WC-007 | Filterable Residue (TDS) E160.1 / SM 2540C |
| WC-008 | Conductivity E120.1 / SM 2510B |
| WC-009 | SPLP, SW1312 |
| WC-010 | Paint Filter Liquids Test, SW9095 |
| WC-011 | Threshold Odor (T.O.N.), E140.1, SM 2150B |
| WC-012 | Nitrate/Nitrite Autoanalyzer, E353.2/SM4500NO3-F |
| WC-013 | Ortho-Phosphate by Autoanalyzer, E365.1 |
| WC-014 | TS (Total Residue), E160.3, SM2540B |
| WC-015 | Color, E110.2, SM 2120B, 2120C |
| WC-016 | Chloride by Autoanalyzer, E325.1, 325.2/SM4500CL-E |
| WC-017 | Chemical Oxygen Demand, E410.4 |
| WC-019 | Ammonia by E350.1 |
| WC-021 | TOC by SW9060 |
| WC-022 | TOC by 415.1 / SM5310B |
| WC-023 | Sulfide by SM4500S-D / 376.1 |
| WC-024 | Cyanide by SM4500CN-E / 335.2 / 9010 /9014 |
| WC-030 | Total and Free Chlorine by SM4500CI-G |
| WC-031 | Phenolics by E420.2 |

| | |
|--------|---|
| WC-034 | Fluoride by SM4500F-C |
| WC-035 | Hardness by Titration by SM2340C / 130.2 |
| WC-036 | Turbidity by Nephelometry, E180.1 |
| WC-038 | CBOD-BOD, E405.1/SM5210B |
| WC-040 | Unionized Ammonia by DEP SOP |
| WC-041 | TKN by E351.2 |
| WC-042 | MBAS, E425.1, sm5540C |
| WC-043 | Alkalinity by Titration, E310.1/SM2320B |
| WC-044 | Total Phosphorus by E365.4 |
| WC-046 | SOUR by SM2710B |
| WC-047 | Dissolved Oxygen, SM4500O-G |
| WC-049 | Orthophosphate (UV SPEC), E365.2/SM4500P-E |
| WC-051 | Volatile Solids by E160.4 |
| WC-052 | Settleable Solids, E160.5, Imhoff Cone |
| WC-054 | Inorganic Anions by IC, E300.0 |
| WC-055 | Bromate by IC, E300.1 |
| WC-056 | Total Solids by Moisture Analyzer |
| WC-057 | PH Soils by EPA 9045D |
| WC-058 | Chlorophylls by SM10200H |
| WC-059 | Total Phosphorus 365.3 |
| WC-060 | Total Phosphorus 365.1 |
| WC-061 | Silica by Spec 370.1 |
| WC-063 | Calibration of Gels and Meters |
| WC-064 | Chromium VI by SM 3500Cr-D |
| WC-065 | UV-254 by SM 5910B |
| WC-067 | Corrosivity (Langelier Index) SM2330B |
| WC-068 | Calculation of Organic and Total Nitrogen |
| WC-069 | Ferrous Iron by SM 3500Fe-D |
| WC-070 | Color by Spec Analyzer |
| WC-071 | BOD and CBOD by auto SM5210B |
| WC-072 | Salinity SM2520B |
| WC-073 | CaCO3 Equivalency ASTM C25 11 33 |
| WC-074 | Flashpoint by EPA 1020 |
| WC-075 | Ignitability by EPA 1030 |
| WC-076 | EPA 9023 EOX |
| WC-077 | EPA 9020B TOX & 1650C AOX |
| WC-078 | TDS SM2540 StableWeigh |
| WC-079 | TDS SM2540G Percent Organic Matter, Reduction of Organic Matter, and Percent Foreign Matter Content |
| WC-080 | SM 2710F Specific Gravity and Density |
| WC-081 | SM 2310B Acidity |

4.0 Document Control

- 4.1 The document control system (DCS) is designed to allow for easy archival and retrieval of documents, data, and reports generated by AEL.
- 4.2 The DCS is accomplished by following the procedure outlined in SOP ADMIN-022.
- 4.3 The DCS details how and where all documentation generated by AEL is stored, who has access to archived reports, and the documentation used to track the retrieval of these documents.
- 4.4 Proper utilization of the DCS should allow easy and accurate recreation of any result produced by AEL.
- 4.5 All documents issued to personnel in the laboratory as part of the management system shall be reviewed and approved for use by authorized personnel prior to issue. A master list of all documents and their distribution shall be kept on the designated Quality Assurance (Q) drive of the AEL networked servers. In this context "document" shall include SOPs, Quality Manuals, logbooks, binders, spreadsheets, benchsheets, cover sheets, work forms, charts, or instruction manuals used in the lab.
- 4.6 Through storage of raw data either in paper records or electronically, the lab shall retain sufficient records to establish an audit trail of work for the periods listed in this SOP. The record keeping system as described in this SOP shall allow the history of the sample and associated data to be readily understood through the saved documentation. The lab saves unequivocal, accurate records that document all the lab activities such as sample receipt, sample prep, or data verification and inter-lab transfers of samples and/or extracts.
- 4.7 Documents can be kept on paper or electronically, or both.
- 4.8 Offsite Records Storage (paper)
 - 4.8.1 Each AEL Laboratory will maintain as much recent data onsite as possible. When space restrictions require the offsite storage of records, the archival process will be documented as follows.
 - 4.8.2 The system to track the shipment of records is maintained in a binder that tracks the file box number assigned to each file box.
 - 4.8.2.1 This document lists the file box number, a brief description of what is in the storage box, the date the box is archived offsite, and the person responsible for archiving the records.

4.8.2.2 These tracking documents are maintained in the custody of the QA Officer or QA Deputy.

4.8.3 Off-site storage facilities will be sites where the records are secure, free from damage caused by fire, water, theft, loss, environmental deterioration, or vermin, such as a public storage facility.

4.8.3.1 The offsite storage facilities will be visited at least on a monthly basis with a log kept of these visits to ensure the records are still intact.

4.8.3.2 A logical and sensible storage system will be implemented in the offsite storage facility to allow for easy and rapid retrieval of all documents.

4.8.4 AEL retains all hard copy and/or electronic documentation for a minimum of five but no longer than seven years past the end of a project. Those projects that are ongoing longer than a year will be marked as such on file storage boxes.

4.8.5 All documents will be disposed of in a proper manner to ensure confidentiality of proprietary information, such as sending the records to an industrial shredding facility or sending to a recycling center.

4.8.6 Archived records stored offsite will be accessible by the personnel listed in Section 4.10.

4.8.6.1 When records are taken out of the archived storage, the person retrieving the document must be authorized to do so and document what they are taking, the reason for taking the record, and the date.

4.8.6.2 Then, once finished with the document and it is ready for return to archived storage, it will be signed off that the document was returned and placed back into the appropriate storage location.

4.9 Onsite Records Storage (paper)

4.9.1 As long as there is space onsite for the storage of records, those records will remain onsite.

- 4.9.2 The onsite record storage will be tracked in a similar manner as the offsite storage process described above.
- 4.9.3 Onsite storage includes projects that are complete, raw data packs, run logs, prep logs, shot logs, logbooks, and archived documents, etc.
- 4.9.4 Once the records are placed in storage boxes, they are considered archived and are placed under restrictive access at that point.
- 4.9.5 Records that are still current or in use are accessible by all personnel.

4.10 Archived Records Access (paper)

- 4.10.1 AEL limits the access of archived records to upper and middle management personnel. This includes the following positions:

- 4.10.1.1 Technical Director
- 4.10.1.2 QA Officer
- 4.10.1.3 QA Deputy
- 4.10.1.4 Operations Manager
- 4.10.1.5 Sales Director
- 4.10.1.6 Lab Managers
- 4.10.1.7 Project Managers
- 4.10.1.8 Department Supervisors
- 4.10.1.9 Laboratory Supervisors

- 4.10.2 All personnel who retrieve records from archived storage, either onsite or offsite will accept custody of them as detailed in Section 4.8.6 above.

4.11 Electronic Records

4.11.1 Electronic Data Files

- 4.11.1.1 The IT Director is responsible for the backup of all electronic records on the network servers according to SOP ADMIN-029.

- 4.11.1.1.1 This procedure outlines the file locations that are backed up, the frequency, and the redundant process to ensure all records can be restored in the case of a system failure.

4.11.1.1.2 The backup process is done using servers at a remote locate.

4.11.1.2 The analysts may utilize a similar backup process on their instrument computers when backing up their methods and data.

4.11.1.2.1 This backup process is completed utilizing recordable compact discs or USB drives.

4.11.1.2.2 The analysts keep a record of which files are contained on the individual discs and the discs are maintained in their custody.

4.11.1.3 Instruments that are retired, but require a specific software are stored with the software available as well as any archived data, to allow for record retrieval should the need arise.

4.11.2 Electronic Document Maintenance

4.11.2.1 QA Directory. A single common directory for all AEL laboratories exists for access to all documents. The structure and details are listed in SOP ADMIN-025.

4.11.2.2 All documents, spreadsheets, forms, etc. (herein referred to as documents) that are generated using computer programs and stored electronically will be controlled and validated in the following manner.

4.11.2.2.1 All documents will be assigned a document control number (DCN).

4.11.2.2.2 A master list of these documents will be maintained by the QA department.

4.11.2.2.3 Each document will have an issue date and last revised date. (Only the last revised document will be available for use.)

4.11.2.2.4 Once approved, these documents will be placed in a controlled access folder where they cannot be modified without QA's approval.

4.11.2.2.4.1 If modified, the document will be given a revision date to track as well.

4.11.2.2.5 All original files will be retained to allow traceability to the revision performed.

4.11.2.2.6 The expired documents will be retained in an 'archived' read only folder and will not be available for use.

4.11.2.2.7 No uncontrolled versions of any documents will be allowed for use in the laboratory. This will ensure that no unapproved documents are used.

4.12 Electronic Data Security

4.12.1 All electronic data is generated and processed behind hardened electronic firewalls, is protected by anti-virus and anti-spyware systems (with real-time updates) and is protected by multiple layers of security masks to prevent unauthorized access.

4.12.2 As soon as data is acquired by each instrument, it is delivered automatically to two separate servers housed at our central data center ("Co-location") in Tampa, FL. These servers are in locked cabinets with access by IT personnel only.

4.12.3 Nightly automatic backups are performed, generating logs which are reviewed daily. Backups and archived data are stored as "read only" in a "write-protected" environment to prevent amendment, deletion, etc. by unauthorized persons.

4.12.4 Electronic records are retained and accessible for a minimum of 5 years unless a longer period is specified by contract for a particular project.

4.12.5 For any change of the LIMS, all clients shall be notified.

4.12.6 For any and all DoD work, the EISC R&R Suite software shall be utilized to collect and report data.

4.12.6.1 Each employee that accesses the system has a user ID. For a new analyst and at the start of each year, at log-in, each user will be given a prompt to change the password. If the prompt is not seen initially, there is a spot on the top of the main page that the analyst will need to click to give

them the prompt to change it. Each January, this will be reset in the system to bring the prompt back up and the analyst will go through the password change again.

4.12.6.2 Annual cyber-security training will be given at the beginning of the year. The main elements emphasized in the training are:

4.12.6.2.1 Establishing a strong password. The password should contain a combination of uppercase and lowercase letters, numbers, and symbols, and be typically a minimum of seven characters long.

4.12.6.2.2 Appropriate Internet use guidelines. Internet use is to be strictly for work purposes only and shall be to trusted sites only. Note: No streaming video is allowed. Penalties for violating company cyber-security policies shall begin with a written reprimand and continued violation will result in termination.

4.12.6.2.3 Appropriate e-mail use. Do not open any e-mail except from a known address. Do not start any executable files from an e-mail, unless first approved by the IT Department.

4.12.6.2.4 Appropriate rules of behavior to handle and protect customer information and other vital data. As discussed with annual ethics training, confidentiality with client information is again stressed. No one other than the client or the client designate is to receive electronic data for any of the testing we perform. This data is to be provided by the lab personnel designated to disseminate that data, mainly the project managers and the lab managers. Also, any of the lab proprietary information shall not be distributed without upper management approval.

4.12.6.2.5 The use of anti-virus and anti-spyware. No attempts shall be made to disable this software from any computer. No computer is to be moved to another location without IT Department permission beforehand.

4.12.6.2.6 Wi-Fi access training for mobile device security is performed. At present, any mobile device that is connected to the laboratory's network (presently only laptops) has first undergone a setup by the IT Department. This policy of all computers going first to the IT Department will be continued.

4.13 Dates of Service or Use for logbooks

4.13.1 All documents such as logbooks, will have a sticker on the front of the logbook stating the date the logbook is placed in service, the person responsible for the logbook, and the date it is completed.

4.13.2 Logbooks that are retired before they are complete will have be "Z-ed out" by drawing a Z over the entire page of each unused page to ensure that no information is entered in the logbook at a later date.

4.13.3 Archived logbooks will be removed from service once it is no longer needed. This will be soon after the replacement logbook is placed into service, but not necessarily at the same time.

4.13.3.1 Archived logbooks will be retained onsite in the possession of the QA Department for at least 3 months to allow easy retrieval or review during that time period. At any time after 3 months, the logbooks will be transferred to permanent storage according to the process outlined above.

4.13.4 Electronic logbooks are now available for several methods. Those that are used as a daily record must be saved as a PDF file once completed. Those logbooks that are continuously updated, such as a standards logbook, shall be electronically saved under backup so as to have a traceable record.

4.14 Manual Data Entry

4.14.1 Any manual entry into a logbook, data pack, bench sheet, etc. that is entered incorrectly will be corrected by drawing one line through the wrong entry and then the person responsible will enter the correct information and initial and date the correction. Additionally, the person making the entry of the correction will supply a reason correction was necessary if it is not self-evident.

4.14.2 All manual calculations will be initialed and dated.

- 4.14.2.1 If the data pack requires numerous calculations, then a statement on the cover sheet for the packet may be included stating 'All calculations were performed and verified by (initial) on (date).'
- 4.14.2.2 If it is later determined that one of the calculations is incorrect, then the procedure mentioned in Section 4.14.1 will be performed to correct the entry.
- 4.14.3 This process will be done for each correction made on a page.
 - 4.14.3.1 If there are numerous corrections or calculations to be performed on a page, then the analyst may put an * by each entry and a statement at the bottom of the page indicating 'all corrections were performed by (initial) on (date)'
- 4.14.4 No entries are to be scratched out, 'whited-out', obliterated, erased, or otherwise made undeterminable. Doing so will create an ethics violation and will be dealt with according to the policy outlined in Section 1.6 of this Manual.
- 4.14.5 All entries are to be made in permanent ink at the time they are made. There is no provision for allowing results to be written on a note pad or piece of paper and then transferred to the logbook at a later time.

4.15 Revision Process

- 4.15.1 All procedural/technical SOPs will be reviewed and/or revised on an annual basis, at a minimum. If no revision is required, the date of review shall be listed in the revision log. Administrative and non-procedural SOPs shall be reviewed and revised every three years.
- 4.15.2 All documents will be reviewed on an as needed basis.
- 4.15.3 The Quality Manual will be reviewed and revised on an annual basis at a minimum.
- 4.15.4 Hand-written amendments/revisions of documents are allowed if the following conditions are met.
 - 4.15.4.1 The document is of a type only available in a paper form. No electronic documents can be hand edited.

- 4.15.4.1.1 Any request for edits to an electronic document shall be e-mailed to the QA department.
- 4.15.4.2 The person responsible for the change includes the initials and date with the amendment/revision.
- 4.15.4.3 The change is approved by the QA department, which means a QA representative will be required to initial and date the amendment/revision as well.
- 4.15.4.4 The hand-written change is incorporated into the next regularly scheduled revision process.
- 4.15.5 Any edits to the Quality Manual shall be handled through the QA Department with those edits listed in an amendment page kept at the front of the Quality Manual pending their incorporation into the next revision.
- 4.15.6 Records for changes made to data (either hardcopy or electronic) include the identification of the person who made the change and the date of change.

5.0 Traceability of Measurements

5.1 AEL utilizes the following processes to ensure every measurement made in the AEL laboratory is traceable, either to NIST standards or approved standards from approved vendors.

5.2 See also the following Technical SOPs for the full procedures and a more in-depth discussion than that found in this section.

- 5.2.1 TECH-003 Balance Verification Annual
- 5.2.2 TECH-004 Balance Operation Daily
- 5.2.3 TECH-005 Pipette Use and Calibration
- 5.2.4 TECH-006 Thermometer Use and Calibration
- 5.2.5 TECH-007 Syringe Use and Calibration
- 5.2.6 TECH-008 Volumetric Glassware Use and Calibration
- 5.2.7 ADMIN-038 Calibration, Manual Integration, and Rules for Chromatography

5.3 Thermometers

5.3.1 All laboratory thermometers are calibrated against a NIST certified thermometer on an annual basis in accordance with the Appendix listed at the end of this section.

5.3.1.1 For digital thermometers, quarterly calibration is required, except those that are purchased with a NIST traceable calibration certificate. Those are used through to their expiration date and replaced or sent to a vendor capable of NIST calibration to recalibrate/recertify.

5.3.1.2 Per TNI 2016, thermometers shall be calibrated at a single point when the range of the normal temperature monitoring is less than 10 °C. When the range spans greater than 10°C, then the calibration is to be performed at low and high points to encompass the range of measurements.

5.3.2 The master analog NIST certified thermometers used to calibrate all in house thermometers and occasionally client thermometers are sent to the manufacturer/outside vendor for re-certification/re-calibration and returned with a certificate of NIST traceability once every 3 years at a minimum in accordance with the SOP Appendix. The digital wide scale (presently -1 to +1000°C) is repurchased new at its expiration date.

- 5.3.2.1 The documentation for the NIST thermometers is stored with the QA Officer and on the designated Quality Assurance (Q) drive of the AEL networked servers.
- 5.3.3 Thermometers are utilized in the following locations:
 - 5.3.3.1 Analytical Use Ovens
 - 5.3.3.1.1 Ovens that are used for non-analytical uses, such as glassware drying, are not required to have the temperature recorded
 - 5.3.3.2 Refrigerators
 - 5.3.3.3 Freezers
 - 5.3.3.4 Incubators
 - 5.3.3.5 Water baths
 - 5.3.3.6 Concentrators
 - 5.3.3.7 At locations where temperature readings are required for a nearby piece of equipment such as a TCLP rotator.
 - 5.3.3.8 Where room temperature requires monitoring.
- 5.3.4 There is a logbook or benchsheet for each thermometer where the daily temperature recording is stored in the vicinity of the thermometer itself.
- 5.3.5 The procedure for performing the daily temperature recordings is explained in Appendix 5.2.
 - 5.3.5.1.1 All thermometers requiring daily checks are observed and manually documented on days of business when employees are in the lab. Min-Max registering thermometers are used to monitor temperatures over weekend and holidays if employees are not present to read temperatures on those days.
- 5.3.6 Where temperatures need monitored when no analyst is present to document temperatures (such as a weekend or holiday) or where constant monitoring is required (such as CBOD incubation and

TCLP rotation), the lab shall use digital Min-Max registering thermometers.

5.3.6.1.1 The thermometers are reset at the time that monitoring will start and read at the time the monitoring period ends. The range of temperatures displayed shall be documented as the minimum and maximum temperatures for that time period.

5.3.6.1.2 Min-Max digitals are purchased with a NIST traceable calibration certificate. Those are used through to their expiration date and replaced, which is normally a two-year period or less.

5.3.7 The refrigerator/freezer temperature logs are to be reviewed by the QA Department on a regular basis. (At least monthly). The QA for each lab may elect to keep those records in their office rather than have them kept by department.

5.4 Calibrations Weights

5.4.1 AEL utilizes calibration weights to verify all balances are accurately measuring the specified amount.

5.4.2 The balances are checked each morning, or before use if used infrequently.

5.4.3 The process of checking the balances is discussed in the SOP Appendix section.

5.4.4 The weights checked will be determined by the particular use of the balance. The weights utilized should encompass the range of the typical amounts weighed on the balance.

5.4.5 The logbooks all list the weights being utilized and the acceptance range of the weight.

5.4.6 The weights are certified Class S (Class 1) or equivalent weights.

5.4.6.1 An outside certification company verifies the certification every 5 years of a master set of calibration weights. These weights are used to verify all other weights used throughout all the facilities. This verification process is coordinated

through the QA Department, in accordance with Appendix 5.6.

5.4.6.2 The records of the re-certification are kept in the QA Officers custody.

5.4.7 All AEL weights are Class S or Class 1 and are traceable to NIST certified standards.

5.5 Certificates of Analysis (C of A)

5.5.1 All Certificates of Analysis are stored in and by the department receiving the product/standard. These are documented with the following information:

5.5.1.1 Date Received

5.5.1.2 Analyst Receiving

5.5.1.3 Expiration Date (if none is provided by the manufacturer).

5.5.1.4 Lab assigned internal lot#.

5.5.2 All standards that are used for calibration purposes will have a C of A. The C of A will trace the standards purity and concentration back to proven concentrations and standards as provided by the manufacturer. These standards must be from an approved vendor. (See QA directory for most up to date approved vendor's list)

5.5.3 Consumables which are certified clean should be provided with a C of A to ensure the cleanliness meets the standards required. These C of As are tracked according to the lab assigned internal lot#.

5.5.4 Some examples of the consumable that would require a C of A are listed below. If no certificates exist or are unavailable, method blanks using the consumables should be used to verify the cleanliness of those items traceable to lot #.

5.5.4.1 Pre-cleaned Sample Bottles

5.5.4.2 Digestion Vessels

5.5.4.3 Disposable BOD Bottles

5.5.4.4 Autosampler vials

5.5.5 Where no C of A comes from the vendor, the lab should use other means to show that consumables are appropriate for use in the lab. Chlorine strips and pH strips are two examples of items that can be tested and approved for use this way. Internal documentation of this shall constitute a substitute for the C of A.

5.6 Instrument Calibrations

- 5.6.1 Instrument calibrations are covered in Section 8.0 of the Quality Manual.
- 5.6.2 Instrument calibrations are done using standards that are traceable to Certificates of Analysis referencing NIST standards.
- 5.6.3 Calibration Standards are purchased according to AEL SOP ADMIN-013 to ensure an approved vendor is supplying the standards and proper documentation is provided to verify the accuracy and traceability of the standards.
- 5.6.4 All standard preparations are documented in standard preparation logbooks. These logbooks must include the following information for each standard made.
 - 5.6.4.1 Analyst initials
 - 5.6.4.2 Date made
 - 5.6.4.3 Date Expire
 - 5.6.4.4 Lot number of parent standard
 - 5.6.4.5 How the standard was prepared and any applicable lot # of the reagents used to prepare the standard
 - 5.6.4.6 A unique identifier must be assigned to the standard.
 - 5.6.4.6.1 This is typically a combination of the logbook page and department but is defined in the individual standard logbooks in use throughout the laboratory.
- 5.6.5 All original standards and reagents from the manufacturer will be labeled with an expiration date upon receipt if there is none assigned from the manufacturer.
 - 5.6.5.1 The expiration date is set to be a maximum of 20 years from the date of receipt unless it is stipulated by method or it is common knowledge the standard will not remain valid for that long. Upon which, the analysts will use good professional judgment in determining the expiration date.

5.6.5.2 If at any time during the use of a standard it is seen that the standard is no longer accurate, it will be replaced and disposed of accordingly.

5.6.5.3 If at any time during the use of a reagent it is seen that the reagent is no longer exhibiting pure characteristics, it will be replaced and disposed of accordingly.

5.6.6 All calibrations must be traceable from the standards used to the certificates of analysis or statements of purity depending upon the type of standard being used.

5.7 Expiration Date Policy

The process of assigning expiration dates for calibration standards and reagents will be accomplished with the following policy.

5.7.1 The analytical SOPs state the policy used, and any discrepancy found between the Quality Manual and the analytical SOPs will utilize the analytical SOPs as the determining document.

5.7.2 As a general rule, all reagents, standards, and consumables that do not have an expiration date assigned by the manufacturer or stipulated by method will be assigned an expiration date of 20 years from the date opened when those items are still viable. (See AEL SOP-Admin 13 for full details and conditions)

5.7.3 All child standards will not have an expiration date that exceeds the parent standard.

5.7.3.1 Definition: Child Standard refers to any standard made from another standard. Also referred to as working or intermediate standards.

5.7.3.2 Definition: Parent Standard refers to a standard that is opened directly from the standard supplied by the manufacturer, including stock standards that are ready to use from the manufacturer. Also referred to as stock standards.

5.7.4 The expiration date clock starts with the date the standard is made.

5.7.5 For Stock standards that require opening a vial and diluting, such as for organics, the expiration date applied to the stock standard made starts with the date the vial is opened.

5.7.5.1 For Standards that require this process, the expiration date on the vial, which is assigned by the manufacturer, is treated as the last day the vial can be opened. The expiration date of the stock standard made at that point cannot exceed the expiration date on the vial.

5.7.5.1.1 Example: If the vial is opened one week before the expiration date assigned by the manufacturer on the vial, then any standard made from that vial would have an expiration date of one week as well.

5.7.6 Validation of expired standards

5.7.6.1 In those instances where an expired standard will need to be used, validation of the expired standard is required. This is done by comparing the expired standard to a separate source non-expired standard. The expiration date of the newly verified standard shall not exceed that of the validating standard.

5.7.6.1.1 To minimize the occurrences of using expired standards, all uses will require approval from the QA Department or the Technical Director of the lab. Approval will be granted by the initials of the approving individual on the calibrations page with an explanation stating the standard has been validated and approved.

5.7.6.2 This may include the analysis of a CCV and ICV. If one is expired and one is within its expiration, as long as the two standards validate each other and agree within the required QC requirements of the analytical method, then the expired standard is considered validated and acceptable for use.

5.7.6.2.1 If this validation is of a stock standard, then a new expiration date may be assigned according to the specifications described in the analytical method.

5.7.6.2.2 This validation process must be well documented and referenced in the standard prep logs. A reference to the date of the validation procedure is required to be included in the standard prep log.

5.7.6.3 The process of using expired standards is discouraged, but due to the high cost of certain standards, it is deemed acceptable by AEL if all above conditions are met.

5.7.6.3.1 Under no circumstances will two expired standards be considered validated if they agree. There must always be at least one of the standards within its expiration range.

5.8 Support Equipment

- 5.8.1 Support equipment includes items such as balances, fume hoods, autoclaves, etc.
- 5.8.2 AEL's support equipment can be serviced by an outside source when necessary to verify compliance with the requirements of the equipment being tested or appropriate regulations in accordance with the guidelines listed in this sections Appendix.
- 5.8.3 When serviced by an outside vendor, the records of the servicing are kept on file in the QA Officer's custody if a balance or hood or in the department if an oven, incubator, or autoclave.
- 5.8.4 If any problems arise with the equipment or if the equipment is suspected of not operating properly, that item is placed out of service until it is deemed acceptable by the lab or where applicable, by the appropriate outside vendor.
- 5.8.5 Hoods are checked annually using a traceable calibrated anemometer with records kept on the designated Quality Assurance (Q) drive of the AEL networked servers.

5.9 Autopipets, pipets, and glassware.

- 5.9.1 Autopipets, such as Eppendorf pipets, are checked on a quarterly basis for accuracy, at a minimum. Some projects specifications (such as for DoD work) may require daily checks.

5.9.1.1 The checking of these is documented in a logbook or by use of an electronic benchsheet. Electronic records are kept on the designated Quality Assurance (Q) drive of the AEL networked servers.

5.9.1.2 Each department is responsible for performing their own pipet verification.

5.9.1.3 This verification is accomplished by utilizing the procedures found in TECH-SOP -005.

5.9.2 Standard Pipettes and Glassware

5.9.2.1 All pipets and glassware that are used for measuring volumes are either Class A or certified TD or TC at a specified temperature.

5.9.2.2 No other class of glassware is allowed for measuring volumes unless of a disposable variety and those are checked for accuracy prior to use per lot.

5.10 Glass Microliter Syringes

5.10.1 AEL identifies each glass microliter syringe uniquely. Internal verifications for volume and proper gas tight seal are required on these syringes when first received and on an annual basis thereafter. Verification shall also be performed whenever it is suspected that the syringes are not performing properly. (A Teflon seal shall not be used for syringes used for PFAS analysis)

5.10.2 Any syringes failing these verification checks shall be removed from service if found to be outside of acceptance criteria. (The Jacksonville laboratory calibrates each syringe to comply with DoD work requirements)

5.10.3 Records of verification shall be stored electronically on the QA directory.

APPENDIX for Section 5

Appendix Section 1

Daily Balance Calibration Procedure

- Prior to use each day, all balances will be checked against the NIST traceable weight set.
- The process will include using weights to bracket the typical weights used on the specific balance each day. Each balance may utilize a specific range depending upon the type of balance or the intended use of the balance.
 - The weight will be recorded in the logbook near the vicinity of each balance and compared to the established acceptance criteria listed in the logbook. The acceptance ranges will be listed in the calibration logbook.
- The recorded weight will be verified for accuracy within the acceptance range for each weight needed.
 - If the weight is outside tolerance, that weight will be checked in duplicate again and reassessed.
 - If both rechecks are acceptable, then the process can continue.
 - If the rechecks are unacceptable, then routine maintenance is performed on the balance, and the weight will be rechecked in duplicate again.
 - Routine maintenance would include cleaning the balance thoroughly and checking the level of the balance.
 - If the weights are acceptable, then the process can continue.
 - If unacceptable, then the balance is removed from service until it can be serviced and placed back into service by an outside calibration company.
- Once all weights are recorded and verified to be acceptable, the balance is ready for use for that day.

Appendix Section 2

Daily Temperature Recording of Support Equipment

- Each piece of equipment that is used for any type of thermal activity will require daily monitoring.
 - This includes refrigerators for sample storage and reagent/standard storage.
 - This includes freezers for sample storage and reagent/standard storage.
 - This includes ovens used for testing. Ovens that are used for drying glassware only are not required to be checked daily.
 - This includes all incubators and water baths used for testing.
 - This includes all muffle furnaces.
 - This includes all digestion blocks.

- The thermometer(s) should be dispersed throughout the piece of equipment to represent all areas/zones of temperature.
 - If the piece of equipment is of sufficient size where one thermometer cannot reasonably represent all the zones of the equipment, such as a walk-in refrigerator, then multiple thermometers will be used and monitored.

- The daily or morning temperature should be done by the first employee to arrive to check the temperature before anyone can open the equipment and change the equilibrium created overnight.

- Each thermometer log will list the thermometer ID and equipment it is associated with.
 - If the temperature log is a stand-alone entity, then the log will reference the correction factor associated with the thermometer, as determined by Appendix 5.3.
 - If the temperature is to be recorded as part of another logbook, such as metals digestions, then the maintenance log for the piece of equipment will list the date of NIST calibration and the correction factor.

- All incubators and water baths used in microbiology require 2 checks each business day – morning and afternoon, the exception being for when no samples are present in the devices.

- The acceptable temperature range shall be listed either in the logbook or in the SOP, if the logbook is associated with an analysis.

- Any correction-adjusted temperature that is not within range will have the temperature rechecked within 30-60 minutes.
 - If the temperature is acceptable at that point, it will be recorded as passing and monitored accordingly the next day.
- If the temperature is still outside the range, then the temperature control will be adjusted accordingly to see if a slight adjustment can bring the temperature back in line with requirements.
 - There should be at least 2-3 hours elapsed before rechecking the temperature after adjusting.
- If the recheck is acceptable, then nothing further is required.
- If the temperature is still unacceptable, then the content of the equipment will be removed to another similar piece of equipment and service will be performed until the equipment maintains an acceptable temperature.
 - This may require an outside service agency to work on the equipment.
 - The piece of equipment will be labeled as out of service during this time period.

Appendix Section 3

Annual NIST Thermometer Calibration Process to apply Thermometer Corrections

- On an annual basis, all thermometers will be verified against a NIST certified and calibrated thermometer, except digital thermometers, which are verified quarterly.
- Documentation of this will be retained electronically or in a logbook or (either temperature log or maintenance log) associated with the equipment/thermometer.
- The NIST thermometers are kept in the possession of the QA Department and can be distributed for use when necessary.
- The thermometer is tested in an appropriate range for each thermometer using one of the following procedures.
 - Freezer thermometers will be tested by comparison with a NIST traceable thermometer, both placed in the freezer for measurement.

- Refrigerator thermometers will be tested by comparison with a NIST traceable thermometer, both placed in the refrigerator for measurement.
- Oven thermometers will be tested by using a beaker of sand placed in the oven to be used as the container from which the measurements are made.
 - The sand should coincide with the temperature of the oven.
- Incubators will utilize the same procedure as the refrigerators.
- Metals digestion blocks will use water placed in the block, via digestion tubes or vessels, and the temperature raised to the working temperature. Then the thermometers will be placed in the water and the same process as above used for checking the temperature.
- Temperature checks of devices that are greater than the range of the yearly vendor calibrated NIST certified thermometers ($> 201^{\circ}\text{C}$) shall be checked with the wide range NIST traceable digital thermometer. A container of sand shall be placed in the device and allowed to come to equilibrium. The temperature probe will then check the temperature of the sand.
- The correction factors will be included into the documentation with the thermometer in the temperature log or maintenance log. The correction factor cannot exceed 3 degrees C for regular laboratory use thermometers. If greater than 3 degrees C, a replacement thermometer must be ordered.
- The correction factor cannot exceed 1 degrees C for microbiological incubator thermometers. If greater than 1 degrees C, a replacement thermometer must be ordered.
 - For those instruments with adjustable digital outputs, such as a many makes of microbiological waterbaths, the temperature reading of each piece of equipment will be adjusted to the correct NIST true value.
 - The daily temperature readings will be direct reads from the digital thermometer readout as calibrated to NIST true value.
- The thermometers are identified by their serial number.
 - If there is no discernible serial number present on the thermometer, then an internal thermometer number is assigned.
- A list of the thermometers, serial number (or internal #), correction factor, and date of calibration will be retained by the QA Department.

Appendix Section 4

Certified NIST Thermometer Renewal Calibration Procedure

- The NIST thermometers utilized by AEL are sent to an outside agency on a basis of once every 3 years to verify the accuracy of each thermometer.
- Once verified, the thermometer is returned to AEL with a certificate attesting to its accuracy.
 - These certificates are retained by the QA Department.
- The QA Department is responsible for monitoring the renewal dates and sending the thermometer to the service company before the expiration date.
 - The thermometer will be sent out for recalibration as the expiration date approaches. No thermometers will be calibrated against an expired thermometer.
- The thermometers are sent using a certified courier agency, such as FedEx, and securely packaged to ensure breakage does not occur during shipment.
- Once the thermometer is returned after its renewal process, the thermometer will be thoroughly examined to ensure it is still in proper working order.
 - This would include checking the mercury or spirit column for any separation, that the thermometer is not broken, and testing it versus another thermometer to make sure it reads correctly.
 - If possible, another NIST thermometer will be used as the reference thermometer.
 - If the thermometers agree and there is no obvious sign of damage, the thermometer will be considered acceptable and ready for use.
- A record of this checking procedure is retained by the QA Department in the temperature logbook.
- This logbook will also be used as a sign-out process for tracking the location of the NIST certified thermometers.

Appendix Section 5

Annual Calibration Weight Renewal Procedure

- The master calibration weights utilized by AEL are sent to an outside agency every 5 years to verify the accuracy and traceability to NIST standards of each weight.
- Once recalibrated, the weight set is returned to AEL with a certificate attesting to its accuracy.
 - These certificates are retained by the QA Department.
- The QA Department is responsible for monitoring the renewal dates and sending the weights to the service company before the expiration date.
 - The weight set will be sent out one month before its expiration date to ensure the renewal is performed before the expiration date.
- The weight sets are sent using a certified courier agency, such as FedEx, or picked up by the vendor, and securely packaged to ensure breakage or loss does not occur during shipment.
- Once the weight set is returned after its renewal process, the weights will be thoroughly examined.
 - This would include checking that none of the weights are broken and checking the weights to make sure they are accurate by using an analytical balance.
 - If the weights agree and there is no obvious sign of damage, the weight set will be considered acceptable and ready for use as the master set that all other weights are compared too.
- A record of this checking procedure is retained by the QA Department in the weight set tracking logbook.
- This logbook will also be used as a record of the storage locations of the different weights sets.

Appendix Section 6

Support Equipment Certification/Testing Procedure using a Certified Service Company

- The support equipment in use by AEL will be serviced by an outside agency on a maintenance required need only. The routine checks performed by AEL personnel ensure that the equipment is in good working order prior to its use. Any equipment deemed faulty will be removed from service until it is repaired in house if within the lab's ability to properly do so, or by an outside certified vendor. (Note: for any lab performing DoD work, the balances are calibrated and certified in good working order annually by an approved vendor.)
- The support company will be vetted to ensure they are certified to service the equipment. Only vendors from the approved vendors' list shall be contacted to do the service.
- Support Equipment includes the following:
 - Autoclaves
 - Analytical Balances
 - Fume Extraction Hoods
- The service company will provide a certificate to serve as record of the testing performed and verify that the equipment is operating within specifications.
- The QA Department will retain the certificates supplied by the service company.
- Any equipment deemed faulty will be removed from service if the service company cannot repair it. A new piece of equipment will be ordered to replace the faulty equipment at that point.

6.0 Sample Acceptance

For this section, please note that all references to "AEL" equate to a reference to all the Advanced Environmental Laboratories, Inc. facilities located in Jacksonville, Orlando (Altamonte Springs), Tampa, Gainesville, Miami (Miramar), Tallahassee, and Fort Myers.

6.1 Sample Acceptance Policy

- 6.1.1 AEL will accept samples that are received if all the following factors are met:
 - 6.1.1.1 The samples are in the correct containers. See Appendix 6.1 for a listing of the required containers.
 - 6.1.1.2 The samples are submitted within the recommended hold time and there is enough time for analysis before the hold time will expire. See Appendix 6.1 for the recommended hold times.
 - 6.1.1.3 The samples are preserved properly, including thermal preservation. See Appendix 6.1 for the proper preservation.
 - 6.1.1.4 The samples are submitted with a Chain of Custody that is correctly filled out and adequately transfers custody of samples from the client/sampler/courier to the laboratory. See Figure 6.1 for an example of a Chain of Custody, with the minimum required fields clearly indicated.
 - 6.1.1.5 The samples bottles are clearly labeled, unambiguous, and correctly correspond to the Chain of Custody.
 - 6.1.1.5.1 This includes the samples bottles being correctly identified with the client sample ID, collection time, and collection date in addition to the collector's initials.
 - 6.1.1.6 The Chain of Custody correctly and completely identifies the analytical tests and methods need for each sample submitted.
 - 6.1.1.7 There is sufficient sample volume to effectively complete the required tests and the sample bottles are correctly filled, i.e. zero-headspace vials, in reference to the analysis of volatile organic compounds in water.
 - 6.1.1.8 All field sheets must be submitted and complete at the time of sample drop-off. This applies to all samplers employed or subcontracted by AEL.

- 6.1.2 Any sample that does not meet all of the conditions mentioned above will have the exception documented and depending upon the circumstances, will either be qualified accordingly or rejected entirely. These possibilities are explained in greater detail in the sections below.
- 6.1.3 Excess sample and sample not consumed during analysis may be returned to the client upon completion of analysis, dependent on the nature of the sample. If a sample is of a hazardous nature, the client may be asked to retrieve the sample or pay for the transportation costs to return the sample.
- 6.1.4 AEL reserves the right to refuse any samples that are of a known or suspected high hazard that may be at concentrations levels the laboratory(s) is not equipped to process. These may include such items as reactive chemical, explosive, radiological, or biological agents at levels deemed dangerous to handle under normal environmental laboratory conditions.
- 6.1.5 AEL reserves the right to refuse any samples that are not accompanied by written disclosure of the known or suspected presence of a known or suspected high hazard.
- 6.1.6 AEL's sample acceptance or rejection policy will be listed in the terms and conditions of our contract when AEL provides the contract to the client or contractor. When working under a client or contractor's contract, AEL shall abide by the conditions set forth in that contract, unless a condition or situation is found for which the laboratory is not equipped to handle properly or safely. Upon that discovery, the laboratory will contact the client or contractor to discuss the disposition of the sample(s).

6.2 Login Checklist

- 6.2.1 See Figure 6.2 for an example of the Login Checklist
- 6.2.2 This checklist is to be followed for every project upon receipt with the documentation to be written on the Chain of Custody. Any anomalies will be notated accordingly in the Sample Acceptance Discrepancies Log located in each sample receipt department of each AEL facility.
- 6.2.3 Any exceptions that warrant client notification or project management notification will also instigate the completion of a NCF as explained in Section 10.0 of this Manual and SOP ADMIN-016.
- 6.2.4 Samples received after hours or on the weekends shall be checked for as follows if no short hold time samples are present. The coolers are to be checked for any volatiles samples, and if present, they will be taken to the

volatiles refrigerator if waters and volatiles freezer if soils. The coolers minus any volatiles shall then be taken to inside the walk-in refrigerator, where the lids will be propped open. The regular full time staff shall then proceed with the full login checklist during normal business hours.

- 6.2.5 If a short hold sample needs to be processed and analyzed after hours or over the weekend, those handling that sample and/or the analyst shall check the preservations, labels, and all items as listed on the checklist and make note of any discrepancies in the sample discrepancy logbook and leave notification for the regular login staff so that any issues can be addressed during regular business hours.
- 6.2.6 If a discrepancy is observed on samples received after hours or over the weekend which are of such a nature that the quality of the results may be affected if an issue is not resolved quickly, then a contact number for the lab manager and/or QA officer shall be available for the after hours personnel to contact on how to proceed.
- 6.2.7 For samples and projects that contain work being analyzed under DoD ELAP certification or for a DoD ELAP client, any after hours or weekend sample receiving shall be accepted and logged in by a member of the regular full time login staff only, or someone who has been fully trained to accept samples and is so designated as member of the login staff.
- 6.2.8 All after hours and weekend received samples and projects that contain work being analyzed under DoD ELAP certification or for a DoD ELAP client shall be processed the same as those sample received during regular business hours. All checks and login procedures shall be performed at the time of receipt.

6.3 Sample Containers

- 6.3.1 The correct sample containers are listed in Appendix 6.1 for each method or test.
- 6.3.2 The types of possible sample containers are:
 - 6.3.2.1 Amber glass bottles, various sizes
 - 6.3.2.2 Clear and Amber Poly bottles, various sizes
 - 6.3.2.3 Clear glass jars, various sizes
 - 6.3.2.4 Coliform bottles, 100mL
 - 6.3.2.5 Whirl-Pak bags, 100mL
 - 6.3.2.6 Clear glass vials, various sizes and preservations
 - 6.3.2.6.1 The vials are pre-preserved from the manufacturer for many of the tests requiring these vials.
 - 6.3.2.7 Amber glass vials, various sizes

6.3.2.8 AFDW slides in cartridges.

6.3.3 AEL purchases these bottles from an environmental bottle manufacturer and receives them certified pre-clean from the manufacturer.

6.3.4 The pre-cleaned bottles are accompanied by a Certificate of Analysis document stating the concentration for various parameters that the bottles are verified clean.

6.3.4.1 These C of As are maintained by the sample receipt personnel, and are tracked by the lot number associated with the bottles received. If no certificates exist or are unavailable, method blanks using the bottles should be used to verify the cleanliness of those items traceable to lot #.

6.3.5 Any sample that is received in the wrong bottle type or bottles that are not certified clean (i.e. provided by AEL) will be documented on the Login checklist.

6.3.5.1 If the sample bottle exception could affect the results (such as a sample for semi-volatile organic analysis being received in a plastic bottle which could cause contamination with some of the target parameters for the analysis) client consultation will be required before proceeding with the analysis.

6.3.5.1.1 The circumstances for accepting this sample would require;

6.3.5.1.1.1 Written approval from the client that it is okay to proceed with the analysis and they are aware of the potential issues with the sample,

6.3.5.1.1.2 The sample results to be qualified accordingly, refer to Section 12.0 of this Manual for a listing of the qualifiers and/or SOP Admin-008, and

6.3.5.1.1.3 The exception to be fully explained in the case narrative that accompanies the report in addition to the documentation on the login checklist.

6.3.5.1.2 The client should be advised, in situations like this, to resample if at all possible, since the data is suspect and could be rejected by the regulatory agency during the review process.

6.4 Sample Chemical Preservation Check

- 6.4.1 The chemical preservation will be verified prior to analysis. See Appendix 6.1 for a listing of the preservations for each method.
 - 6.4.1.1 The preservation of each sample must be clearly notated on each bottle.
 - 6.4.1.2 If the sample kit is prepared by AEL and the bottles are properly identified with the type of chemical preservative added, only the samples that have preservatives added will be checked. For samples that only require thermal preservation, e.g. TSS, Color, etc, the pH will not be checked unless the bottle is mislabeled to indicate a chemical preservative was added.
- 6.4.2 The preservation checks will be documented in the logbooks utilized by the analysts in the different departments.
 - 6.4.2.1 This will be accomplished by using pH strips to verify acid or base preservation without compromising the sample's integrity. Either a clean glass pipet will be used for each sample bottle to remove a small aliquot to place a drop on the pH paper or a small, controlled pour onto the pH strip without any pour back into the bottle can be used.
 - 6.4.2.2 For samples that do not lend themselves to easily checking the preservation or where checking the preservation before analysis could affect the integrity of the sample, the preservation will be checked during or immediately after analysis and documented accordingly at that point.
 - 6.4.2.2.1 Examples of this would be the residual chlorine check associated with microbiological samples, or the acid preservation of volatile organic compound analysis.
 - 6.4.2.2.2 This check will be documented in the actual laboratory performing the analysis, and any sample not meeting the proper preservation will have the results qualified accordingly.
- 6.4.3 The preservation of each sample must be clearly notated on each bottle.
- 6.4.4 Any chemical preservation violation will be qualified according to SOP ADMIN-008 by the analysts during the data entry phase of the results.

6.4.4.1 Chemical preservation violations also require an NCF to be generated according to SOP ADMIN-016.

6.5 Sample Thermal Preservation Check

6.5.1 The temperature of the sample bottles will be verified upon receipt. The thermometer used for this is an infrared thermometer, so the sample will not have to be physically touched by the thermometer and the sample's integrity will not be compromised. However, if the samples are in a cooler with ice, the samples will be accepted as having the correct thermal preservation if the samples show a temperature decrease from ambient temperature and were sampled the same day the samples arrive in the laboratory.

6.5.1.1 If the samples were recently collected and have not had time to show any decrease in ambient temperature, but arrive on ice, then the temperature of the ice will be taken and the samples will be accepted as having correct thermal preservation.

6.5.2 The sample receipt personnel will document the temperature of the samples, or the cooler, on the login checklist at the time of receipt.

6.5.3 Any thermal preservation violation will be qualified according to SOP ADMIN-008 by the analysts during the data entry phase of the results.

6.5.3.1 Thermal preservation violations also require an NCF to be generated according to SOP ADMIN-016.

6.5.3.2 Since thermal preservation violations could seriously affect the integrity of the sample or the validity of the data, the client will be consulted before proceeding with the analysis. The results of the consultation will be documented on the NCF by the appropriate project manager to provide the documentation necessary to determine acceptance/rejection of the sample.

6.6 Sample Hold Times

6.6.1 Most samples for environmental analysis have recommended hold times that certain tests or preparation procedures must occur before that time limit is reached. The hold time begins once the sample is collected. Either the EPA or FDEP assigns the hold times. See Appendix 6.1 for a listing of the recommended hold times according to FDEP, which is based upon 40CFR Part 136.

6.6.2 Sample receipt personnel will verify that all samples are within hold time before signing the COC to receive custody of the samples.

6.6.3 Any sample that does not meet hold time requirements will be documented accordingly on the Login Checklist. The client will be advised of the situation and it will be recommended that the client resample if possible.

6.6.3.1 If resampling is not an option, then the sample results will be qualified accordingly, an NCF will be generated and exception will be detailed on the case narrative.

6.7 Bottle Filling Requirements

6.7.1 Certain bottles have specific requirements for the way they must be filled. Sample receipt personnel will verify these procedures are properly completed before accepting the samples.

6.7.2 One example of bottles and/or methods requiring certain filling requirements is VOC analysis via method 5030. The 40 mL VOC vial is specifically designed to have zero headspace if properly filled.

6.7.2.1 If there are air bubbles or headspace present larger than 1% of the vial volume or bubbles are greater than 5 mm (1/4") in diameter, then the samples will be considered improperly filled and will be notated as such on the Login Checklist.

6.7.2.2 If the vials are determined to be improperly filled, and it affects all the vials for a given sample, the client will be advised to resample. If that is not possible, the sample results will be qualified as improper preservation and headspace will be noted in the case narrative.

6.7.3 Another example of bottles with certain filling requirements is the VOC analyses in soil using method EPA SW-846 5035. The SOP given to clients is attached as Figure 6.3 to this section.

6.7.3.1 This method requires 2-40mL VOC vials to contain 5ml of DI water and a stir bar. During sampling, approximately 5 grams of soil is added to each of these vials.

6.7.3.2 There is a 3rd vial that contains 5 mL of methanol. 5 grams of soil is added to this vial as well.

6.7.3.3 And there is a 4th vial that does not have any preservative and is to be completely filled, void of as much headspace as possible.

- 6.7.4 Any samples that do not meet any of these requirements will be fully documented and the decision made on how to proceed before the samples are distributed to the analysts.

6.8 Sample Volume

- 6.8.1 Sample receipt personnel will verify there is sufficient volume to perform the requested analyses before accepting receipt of the samples into the laboratory.
- 6.8.2 Any sample that is determined to have insufficient volume will be documented on the login checklist and the client will be consulted on how to proceed.
- 6.8.3 Written documentation will be obtained if the decision is to proceed with the sample either by using limited volume and thus raising the detection limits or by reducing the number of tests required for the sample.
 - 6.8.3.1 If the decision is to proceed with reduced volume, the 'dilution' factors will be notated on the final report to account for not having the required amount of sample and the detection limits will be adjusted according to this 'dilution'.
- 6.8.4 The standard policy given to clients will be to completely fill all bottles that are supplied for a given sample to assure there is always sufficient volume to perform the requested analyses.

6.9 Client Sample IDs

- 6.9.1 The client is responsible for properly labeling the sample bottles and the Chain of Custody with the correct information. The sample IDs listed on the COC must correspond to the sample IDs labeled on the sample bottles.
- 6.9.2 Any discrepancies found will be resolved before proceeding with analysis. Any changes to sample IDs on the COC or on the bottles will be documented accordingly.
- 6.9.3 Any discrepancies will be documented on the Login Checklist, and the resolution, if known.

6.10 Sample Rejection

- 6.10.1 AEL reserves the right to reject any sample that does not meet its requirements for sample acceptance.

6.10.2 Any sample that is known to be sampled improperly can be rejected. For some samples it may be appropriate to analyze after contacting the client, discussing what the samples are to be used for, and then having the results reported with qualification, such as qualification for improper preservation. However, some samples will be rejected outright. This could include microbiological samples or demand samples that have an excessive amount of chlorine, and thus have no chance of biological growth, due to over-chlorination by the person performing the sampling.

6.11 Sample Login into the LIMS

6.11.1 Once the samples are accepted into the laboratory, the sample receipt personnel will enter the project and sample information into the LIMS system and assign unique identifiers to each project, sample, and sample bottle.

6.11.2 SOP ADMIN-005 provides detailed instructions for how to proceed with this process.

6.11.3 The unique project and sample identifiers are provided in Section 12.11 of this Manual.

6.11.3.1 An example of a unique project number would be J210001 or A2102445

6.11.3.2 An example of a unique sample number would be J210001-001 or A210245001.

6.11.4 Using a sequential alphabetic identification after the sample ID and placing the label containing this identifier on the sample bottles identify the individual sample bottles.

6.11.4.1 An example of this format for a project with 1 sample and four bottles would be:

6.11.4.1.1 J210001-01A for the first sample bottle

6.11.4.1.2 J210001-01B for the second sample bottle

6.11.4.1.3 J210001-01C for the third sample bottle, and

6.11.4.1.4 J210001-01D for the fourth sample bottle.

6.12 Sample Receipt and creation of Sub Samples as it relates to each Facility.

6.12.1 Sample receipt and sample handling for all Advanced Environmental Laboratories shall follow the guidelines set forth in AEL SOP Admin-005 and in this quality manual as described in this section 6. These documents will constitute the sampling plan for these facilities.

- 6.12.2 All field sampling for all facilities shall follow the most current DEP Sampling SOPS as listed on Florida DEP website:
<http://www.dep.state.fl.us/labs/qa/sops.htm>.
- 6.12.3 All field-sampling personnel shall undergo FDEP SOP Sampling Training or be trained by someone who has attended such training.
- 6.12.4 When obtaining sample aliquots from a submitted sample, the laboratory shall use the procedures and techniques as outline in section 6.21 to obtain a representative sample.
- 6.12.5 The sample acceptance policy in effect for all Advanced Environmental Laboratories facilities, as listed in section 6.12.1, are those set forth in this quality manual. A brief outline can be seen in figure 6.3, which is also to be prominently posted in each sample receipt department.

6.13 Sample Storage

- 6.13.1 Once the samples are entered into the LIMS system and properly labeled, they are dispersed throughout the laboratories for analysis.
- 6.13.2 Samples that require thermal preservation are placed in refrigerators promptly once the login process is completed.
 - 6.13.2.1 Samples for common tests or all tests except volatile organic analysis are placed into common refrigerators.
 - 6.13.2.2 Samples that are to be tested for volatile organic analysis are placed in refrigerators inside the volatiles laboratory once the login process is complete.
- 6.13.3 Samples that do not require thermal preservation are placed in the laboratory where the analysis will be performed.
 - 6.13.3.1 If the samples require multiple analyses or the laboratory to perform the analysis does not have storage capabilities, the samples will be placed in the refrigerators as well.

6.14 Project Folder Review

- 6.14.1 Once the project is logged into the LIMS system, the sample receipt personnel distribute the project folder to the appropriate project manager.
- 6.14.2 The project manager reviews the project folder to ensure accuracy between the chain of custody and the information entered into the LIMS.

6.14.2.1 The project manager accomplishes any corrections or revisions, possibly in combination with the IT Director.

6.14.2.2 All revisions are documented as to the reason for the revision. All revised paperwork is generated and supplied to the analysts with justification for the new paperwork as well.

6.14.3 This folder is then followed through report completion as described in Section 12.0 of this Manual.

6.14.4 Sample Receipt Acknowledgment

6.14.4.1 Some clients request confirmation of sample receipt. AEL accomplishes this by faxing, emailing, or electronic data deliverables the following documents after the review process:

6.14.4.1.1 The COC,

6.14.4.1.2 The Project Summary report, and

6.14.4.1.3 Any specialized spreadsheets requested for this task. This is not always included depending upon the client.

6.15 Sample Disposal

6.15.1 Sample disposal is accomplished according to SOP ADMIN-018.

6.15.2 Sample disposal refers to samples, extracts, digestates, and leachates that are associated with a given sample.

6.15.3 The standard policy for retaining samples is 30 days after completion of the analysis.

6.15.3.1 Some clients have longer retaining periods requested in the statement of work, so samples associated with those projects are handled differently to ensure the clients request is adhered to.

6.15.3.2 Samples requiring this longer retain period will be indicated with a colored label to indicate they are to be disposed of differently.

6.15.4 The sample receipt personnel work in accordance with the hazwaste manager to accomplish sample disposal.

6.15.5 In extreme circumstances, sample may be disposed of before the 30 day hold period if there is insufficient room for storage of new samples AND

the hold times for the samples being disposed of are sufficiently exceeded (>7 days past the recommended hold time.)

6.16 Kit Preparation

6.16.1 Kit preparation is accomplished by following the procedure outlined in SOP ADMIN-023.

6.16.1.1 This procedure outlines all aspects of sample kit processing including many of the following:

6.16.1.1.1 Preservative preparation

6.16.1.1.2 Bottle labeling

6.16.1.1.3 Sample grouping

6.16.1.1.4 Cooler tracking

6.16.1.1.5 Sample bottle lot # tracking

6.16.1.1.6 Documentation to be included with bottle kits

6.16.1.1.7 Kit delivery

6.16.1.1.8 Quality control samples to be included with each kit and the tracking of these, such as trip blanks.

6.16.1.1.9 The types of bottles required for certain types of tests or groups of tests.

6.16.2 The project manager, laboratory manager, or sales staff – whomever is in contact with the client, generates the kit request via a written form or written communication (email). No verbal kit requests are allowed.

6.16.3 The sample receipt personnel will complete the kit request.

6.16.4 Once complete, the bottle kit will be picked up by the client, delivered to the client by AEL courier, or shipped to the client or sampling site via a third-party shipping company.

6.16.5 An example of a kit request form is attached as Figure 6.4.

6.17 Data Quality Objectives (DQOs) and Review of Requests, Tenders (Bids), and Contracts.

See also ADMIN SOP-027 Data Quality Objectives.

6.17.1 To ensure that the final report will meet all the requirements of the client or regulatory agency, a review is conducted prior to the acceptance of new work, or if work is already being performed for an existing client who predates NELAC requirements, a review or summary outline of the work requirements shall be on file with the project managers responsible for servicing those clients.

- 6.17.2 AEL utilizes a DQO form for completing these steps and gathering the required information if not already spelled out in a Bid or Contract. The process for completing this form is detailed in SOP ADMIN-027. An example of the DQO form is provided as Figure 6.5 at the end of this section. If the client provides a contract or a permit for review by the lab, then these can substitute for the DQO form. In the case where an existing client has an established history of testing or is an established client with routine and simple tasks, at the initial review, the date of the review with the reviewer's initials shall suffice as adequate documentation.
- 6.17.3 The purpose of the DQO is to ascertain as much information before accepting the work. This allows AEL to prepare for any special requirements that may be required to accomplish the goals of the client before receiving the samples.
- 6.17.4 The goals of the DQO are essentially the following but may encompass more details for large scope projects or clients with many extra requirements.
- 6.17.4.1 Detection Limits
 - 6.17.4.2 Methods to be used.
 - 6.17.4.3 To ensure the lab has the capability and resources to meet the requirements of the project(s).
 - 6.17.4.4 The test and/or calibration method is appropriate to the client's and project's needs.
 - 6.17.4.5 Format of Report (Level of Report – as explained in Section 12.0 of this Manual).
 - 6.17.4.6 Any electronic deliverable requirements
 - 6.17.4.7 Invoice requirements, i.e., special mailing addresses
 - 6.17.4.8 Delivery of report; fax, mail, or email.
 - 6.17.4.9 Chain of command for difficulties arising during the analysis that require client consultation.
 - 6.17.4.10 Person(s) to contact if a drinking water test is performed and an MCL exceedance is seen.
 - 6.17.4.10.1 Any lab developed methods will be through planned development assigned to a qualified person equipped with the resources to perform the method.
 - 6.17.4.10.2 The plan is to be updated and communicated to all personnel involved.
 - 6.17.4.10.3 An SOP must be written as part of the method development and method validation.
 - 6.17.4.10.4 Method validation shall be on any lab-developed or non-standard methods, along with any use outside the original intended use of the method to confirm the fitness for use.

6.17.4.10.5 The range and accuracy of the developed or modified method meet the range and accuracy relevant to the client's needs.

6.17.4.10.6 The use of a developed or modified method must be communicated and approved in writing from the client.

6.17.5 The project manager, sales staff, or laboratory manager completes the DQOs.

6.17.6 Any special requirements or situations that could affect the normal procedure of analysis require consultation between the Laboratory Manager and person generating the DQO. The QA Officer may also need to be consulted. All special circumstances shall be verified that they can be completed as directed by the client while being within the confines of the AEL Quality System before accepting the work from the client.

6.18 New Work Acceptance Mechanisms (Review of Requests, Tenders (Bids), and Contracts)

6.18.1 A Request shall be defined as any request from a client, whether verbally or in written correspondence for analysis to be performed. A quote can be a direct result of the request. A Tender is a bid for new work or continuation or renewal of existing work. A contract may be any written or oral agreement to provide a customer with testing and/or calibration services.

6.18.2 The customer shall be informed of any deviation from the request, quote, bid, or contract as it is first proposed or if in the course of performing the work, any deviation from the request, bid, or contract takes place. If a request, quote, bid, or contract has to be amended after work has commenced, the same review process shall be repeated, and any amendments shall be communicated to all affected personnel.

6.18.3 Before accepting any new work, AEL will assess the impact of the new work on current workload to ensure it can be completed effectively and timely. (See also section 6.17)

6.18.3.1 The primary person responsible for completing this assessment is the laboratory manager or the project manager in conjunction with the lab manager.

6.18.3.2 The laboratory manager or project manager shall have an understanding of the amount of work, time frame of receiving and completing the work, and the testing involved to make an accurate determination.

6.18.3.3 The laboratory manager or project manager shall review the lab's capability to establish that the laboratory possesses the necessary physical, personnel and information resources, and that the laboratory's personnel have the skills and expertise necessary for the performance of the tests and/or calibrations in question. The review may also encompass results of earlier participation in inter-laboratory comparisons or proficiency testing and/or the running of trial test or calibrations using samples or items of known value in order to determine uncertainties of measurement, limits of detection, confidence limits, etc.

6.18.3.4 The laboratory manager or project manager may consult the department supervisors, other lab managers (for projects involving subcontracting work within the AEL network), and/or the QA Officer to determine the feasibility of accepting the new work based upon the current workload.

6.18.4 New work for this purpose is defined as a new client or a new scope of work from an existing client.

6.18.5 The sales staff will work closely with the lab managers to ensure that all upcoming work can be completed timely and accurately within the rules of the Quality System before accepting the new work.

6.18.6 The sales staff works closely with the Corporate Operations Manager and lab managers to forecast future incoming work so new work can be accepted or rejected readily when the situation arises.

6.18.7 Any work that will have to be subcontracted outside the AEL network will require the sales staff to coordinate the details of the new work with the subcontract laboratory and get assurances from the subcontract laboratory that the work can be completed within the guidelines given by the client for the new work.

6.18.8 AEL will not accept new work if it will put too large of a strain on any of the following:

6.18.8.1 The rules of the Quality System,

6.18.8.2 The current equipment,

6.18.8.3 The current analytical staff, or

6.18.8.4 The project management staff

6.18.9 If the new work is proposed to fit within what is considered acceptable and the QA Officer agrees the Quality System will not be jeopardized, then the new work will be accepted.

6.18.10 This implies that AEL can meet all data quality objectives, as outlined above and in SOP ADMIN-027.

6.19 Subcontracting of Analytical Samples within the AEL network.

- 6.19.1 All samples that can be analyzed within the AEL network will be subject to all the same requirements of documentation and certification when subcontracting to a sister facility as those when subcontracting to an outside lab. Current FDOH certifications are required and for all the labs a copy of the most current certifications is kept on the Jax server in the QA folder, under Q:\Reference\AEL Certs. Chains of custody are required as well.
- 6.19.2 When a separate whole volume container is not available for shipping and a split sample from a container must be made, then follow the instructions as outlined in section 6.21 "Representative Samples".
- 6.19.3 A new label with the same label information as the original (preservation, collection time, etc.) should be created by the LIM system and placed on the split container.
- 6.19.4 It may be convenient for the client to drop off samples at the AEL lab closest to the sampling location rather than at the lab that will analyze the samples. Also, some analysis is only performed at certain AEL labs, such as Low-Level Mercury at the Jacksonville location or Surfactants at the Gainesville location. In those situations, the receiving lab will still login samples into Horizon under their AEL letter designation (see section 8) but then set those tests or the entire project for shipping to the sister lab in the shipping queue in Horizon. Finally, in the event of balancing workload and to expedite testing, it may be appropriate to send samples to a sister lab for analysis on test that the receiving lab does perform.
- 6.19.5 The initial receiving lab when receiving the samples, shall follow all the steps as listed in section 6 of AEL SOP Admin-005, and accept custody of those samples on behalf of AEL.
- 6.19.6 Any anomalies and notifications/questions about the received samples, including the writing of NCFs for those anomalies, shall be handled by the receiving labs with e-mail correspondence sent to the sister lab that will be ultimately analyzing any samples not kept in house.
- 6.19.7 Once samples have been fully received and logged in (with shipping queue set for off-loading to the sister lab) those samples are stored refrigerated or frozen per method requirements until packed for shipment to the sister lab. Normally a separate area or shelf is set aside for sister-lab shipping as an easy visual indicator for samples to be shipped.

- 6.19.8 Metals samples can be stored without refrigeration; however acid preservation should be checked. If further acidification is required to reach a $\text{pH} \leq 2$, then that shall be done and documentation of that shall be listed in a logbook with the date/time and lot# of the acid used. That documentation shall accompany the samples to the sister lab.
- 6.19.9 For Volatiles samples: samples shall be stored away from any potential contaminant sources, including away from other “hot” samples.
- 6.19.10 For Volatile soil samples, if samples are to ship and be received by the sister lab with 48 hours of collection, then they can be refrigerated at 0-6°C prior to shipping. However, if to be shipped and received after 48 hours, then the samples are to be stored on their sides and frozen prior to shipping. Then those frozen samples must be shipped with sufficient dry ice to have them remain frozen at minus 10°C or lower.
- 6.19.11 All other samples shall be refrigerated until shipping at 0-6°C.
- 6.19.12 Towards the end of the day, samples that are to be shipped to sister labs are repacked on new ice (or on dry ice for frozen volatile soil samples) and an inter-lab chain is created listing all the samples to be shipped. The inter-lab chain is reviewed against what is contained in the coolers to ensure that what is in the coolers matches exactly what is on the inter-lab chain.
- 6.19.13 Samples with limited and short hold times must be marked on the outside of the coolers. Notification must be made to the sister lab that these samples are coming their way.
- 6.19.14 Samples requiring special handling or special conditions must be marked on the outside of the coolers. Notification must be made to the sister lab that these samples are coming their way.
- 6.19.15 All these conditions must be notified to the sister lab who will be receiving these sub-samples.
- 6.19.15.1 Sub-samples to a local sub-lab (also to be noted on the cooler)
 - 6.19.15.2 Rush samples (also to be noted on the cooler)
 - 6.19.15.3 Samples that are not drinking water, logged as drinking water (can use stickers on containers)
 - 6.19.15.4 Samples that are potentially unusually hot (separate cooler)
 - 6.19.15.5 Samples that require other than normal reporting
 - 6.19.15.6 Incorrect preservation or container, limited volume
 - 6.19.15.7 Headspace in a volatile sample
 - 6.19.15.8 Any anomalous condition with samples witnessed

- 6.19.15.9 Samples to arrive on other than normal Blue Streak shipping days.
- 6.19.16 Coolers are labeled and set aside for pick up by Blue Streak courier for next morning delivery. Send along with the coolers a copy of the preservation checks made upon original receipt and a copy of the inter-lab chain.
- 6.19.17 Blue Streak shall deliver the coolers overnight to the sister lab. Arrival times can vary, but all should be delivered prior to business hours or no later than 9:00am the next morning. Plan accordingly for short holds.
- 6.19.18 The samples shall be unpacked and accepted by the sister lab using the following guidelines.
- 6.19.19 Check first those cooler marked “short hold” or “rush”
- 6.19.20 Put on a pair of latex disposable powder free gloves. Place the cooler in a well-ventilated area, open it, and determine if there is ice in the cooler. The temperature measurement shall be taken using an IR gun measuring the surface temperature of the sample containers. Note temperature on the inter-lab chain of custody.
- 6.19.20.1 The target range is 0-6 degrees Celsius for samples that are thermally preserved.
- 6.19.20.2 Frozen volatile soil samples shall be received on dry ice at temperatures at or below minus 10°C.
- 6.19.20.3 Acid preservation shall not need to be checked, unless for some reason the original sister lab did not check pH of the samples.
- 6.19.21 While unpacking the coolers, the lab must verify that the samples received match those listed on the inter-lab chain of custody. Sign with time and date the inter-lab chain of custody. Any anomalies must be immediately addressed with the shipping lab.
- 6.19.22 Make a copy of the inter-lab chain of custody and save that record in the login department. Send back to the original receiving lab that original copy of the inter-lab chain using an inter-lab mailer envelope.
- 6.19.23 The lab must then mark those sent samples as received in Horizon so that the samples show properly as received and ready for analysis on the analysts’ backlogs.

- 6.19.24 Once samples are shipped to the sister lab, setup and analysis takes place normally with entry into the LIMS also proceeding normally as a single LIMS is linked to all labs. Data entered into this single database will be available for review at any of the AEL labs.
- 6.19.25 The Project Manager is assigned by client and is designated when the samples are initially logged in. Any anomalies with sample preparation or analysis shall be communicated to the Project Manager through e-mail. When the Project Manager is at a sister lab, the Lab Managers and QA Officers for those labs involved shall also be included in the e-mail.
- 6.19.26 LIMS will automatically note where the analysis took place as data entry cannot take place without assignment of the analytical instrument and location.
- 6.19.27 The Project Manager shall review the project and sample results the same as if all analysis was performed in house. Any questions or request for information shall be through e-mail. When the Project Manager is at a sister lab, the Lab Managers and QA Officers for those labs involved shall be included in those e-mails.
- 6.19.28 The Project Manager shall ensure that the final project clearly lists the proper lab location for each analysis. The Project Manager is responsible that all information on the final report is fully reviewed, correct, and compliant to TNI Standards through all normal review processes. Reporting shall be conducted as normal as listed in section 12 of this Quality Manual.

6.20 Subcontracting of Analytical Samples to outside of the AEL network.

- 6.20.1 All samples that cannot be analyzed within the AEL network will be subcontracted to another TNI certified laboratory. A copy of the lab's certificate and scope shall be on file to release to the client upon request. (If for DoD work, then a lab certified under DoD ELAP certification will be required for subcontracting along with the requirement that their cert and scope to be on file as well).
- 6.20.1.1 The final report of the sub-contracted lab is attached to the AEL generated report, unless it is requested by the client or required by the type of work to integrate that data into the AEL report. In that case the sub-lab data shall be clearly indicated in the AEL report.
- 6.20.2 The laboratory is responsible to the customer for the subcontractor's work, except in the case where the customer or regulatory authority specifies which subcontractor is to be used.

6.20.2.1 The subcontractor's work shall be reviewed by the project manager when received in the same manner as is done for in-house results. Any issues with the quality shall be immediately communicated to the QA Officer and the Lab Manager.

6.20.2.2 Any delays in receiving results in the agreed upon timeframe shall constitute a quality issue and shall also be communicated to the QA Officer and the Lab Manager.

6.20.2.3 In the event of a question in the data, raw data and quality control documents will be requested from the subcontracted lab. As deemed necessary, samples will be split between subcontracting labs to confirm results and/or select the best subcontracting lab.

6.20.3 The sales staff is responsible for determining the subcontract laboratory to be used for certain tests and negotiates pricing with the subcontract laboratories.

6.20.4 Subcontractor laboratories' certified analyte sheets shall be maintained on the designated Quality Assurance (Q) drive of the AEL networked servers.

6.20.5 AEL will notify clients, in writing, when their samples are going to be subcontracted to another laboratory. This is accomplished by any of the following processes.

6.20.5.1 A letter is sent to all clients that submit routine samples on a regular basis that details which tests will be sent to which laboratory as a routine. The client is asked to sign this document and return it. The client will be notified if samples are shipped to another laboratory other than what is listed on the letter they signed, but otherwise, samples will be sent according to these instructions and not require written notification for every project submitted.

6.20.5.1.1 These letters are sent to all clients on a routine basis or whenever the normal subcontract laboratories change. The signed document is placed in the clients file (which is in the custody of the project manager) and can also be found on the Jax server, in the ael_qa folder, under the project manager's folder.

6.20.5.2 Or the client will be emailed or faxed where the samples they submitted will be subcontracted. They will be given a certain amount of time to respond if this is unacceptable, otherwise the samples will be sent there for analysis.

6.20.6 AEL will not submit any sample to a non-TNI certified laboratory that requires certification for analysis or is for regulatory purposes.

6.21 Representative Samples

6.21.1 AEL utilizes the best possible means to ensure that samples are homogenized before analysis and any aliquot used by the laboratory for analysis is representative of the overall sample. The procedure for obtaining representative samples is detailed below.

6.21.2 Liquids

6.21.2.1 When possible, AEL will provide sufficient sample bottles to allow for the entire sample to be delivered in separate bottle so no aliquots will need to be split off by the laboratory.

6.21.2.2 When sufficient bottles are not received, AEL will mix the sample thoroughly before splitting off any aliquots.

6.21.2.2.1 If a special chemical preservation is required for any of the aliquots, it will be added AFTER the aliquots are split off.

6.21.2.2.2 Aliquots will not be taken from chemically preserved bottles unless there is no other sample available. If this is required, the results may be qualified and explained in the case narrative, as necessary.

6.21.2.3 If there is insufficient sample to split off, the client will be contacted for instructions on how to proceed before any aliquots are split off.

6.21.2.4 Once all aliquots and bottles are prepared, the analysts will thoroughly shake each bottle before beginning the analysis and pouring out any aliquots needed for actual analysis. Samples with heavy sediment are shaken vigorously, and then the aliquot is rapidly transferred to the container to be used for analysis or preparation.

6.21.3 Time-Composite Samples

6.21.3.1 Any time-composited sample collected by AEL will be thoroughly mixed together before filling any sample bottles. This is accomplished by either thoroughly shaking the container or using a glass stirring rod (pre-cleaned).

- 6.21.3.2 Once the sample bottles are filled, the bottles are preserved according to the guidelines listed in Appendix 6.1 of this Manual.
- 6.21.3.3 Any time-composited sample delivered by the client that is not already split into the proper bottles is treated similarly.
- 6.21.3.4 The collection time of the sample for composite samples is treated as the time the last aliquot of the composite is collected for all 24-hour collection time or less time-composited samples.

6.21.4 Soil samples

- 6.21.4.1 It is the policy of AEL to report all soil samples on a dry weight basis.
- 6.21.4.2 All AEL reports state that results having units of mg/kg are reported on a dry-weight basis unless otherwise noted.
- 6.21.4.3 This is the first process completed upon receipt of the sample. The sample is thoroughly composited and mixed together by the analyst performing the % moisture test, or if to be split between labs, the sample is thoroughly mixed prior to separating into separate containers. This is to always occur before any other analysis is begun.

- 6.21.4.3.1 Empty the contents of the jar (or jars if more than one per site, excluding volatiles samples) into a clean stainless steel or Pyrex container and thoroughly mix the sample to complete the compositing process. Then the entire sample is either transferred back to its original container(s) or split to separate containers by taking the mixed sample, dividing into quarters, and taking equal increments from each quarter to fill the sub-samples. The mixing container is then cleaned before the next sample is composited.

NOTE: Aluminum foil may be used in place of the stainless-steel bowl when metals are not being tested.

- 6.21.4.3.2 Rocks, leaves, twigs, sticks, and other vegetation shall be removed (unless actually determined to be part of the sample to be tested) and their removal shall be documented. This is to prepare a homogenous sample. If unsure when removal is appropriate, the department supervisor and/or lab manager shall be consulted.

6.21.4.3.3 Once the sample is composited thoroughly, a 'C' is written on top of the jar(s) to indicate the sample has been thoroughly composited and is now ready for use.

6.21.4.3.4 The analyst performing the % moisture test will composite every sample jar for each sample, even if there are multiple jars, to ensure that every jar is treated the same and as representative as possible.

6.21.4.4 The percent moisture values are entered into the LIMS system after being calculated, and every soil result will be corrected by the % moisture value. This is accomplished automatically by the LIMS system.

6.21.4.5 To avoid preparing non-representative samples, the laboratory shall not "target" a specific weight (i.e., the laboratory shall not manipulate the sample material so that sample aliquot weigh exactly a precise weight such as 1.00 +/- 0.01 grams. More reasonable will be weights such as 6.1, 5.9, 6.23 grams etc.)

6.21.5 Waste and Chemical samples

6.21.5.1 For any waste, chemical matrix, or multi-phasic samples, AEL will report them on an as-received basis. If the sample matrix does not lend itself to become a homogenous mixture, the percent moisture requirement is not necessary, and the compositing process is skipped.

6.21.5.1.1 The report will be notated to indicate that these samples are reported on an as-received basis and not corrected for dry-weight content.

6.21.5.2 For any **multi-phase** sample containing layers of differing miscibility, i.e., solvent versus aqueous, AEL will first determine the percentage of each layer.

6.21.5.2.1 If the amount of each layer is over 10% by volume, then each layer is treated as an individual sample and each layer is analyzed separately. Once analyzed, the results are adjusted for the fraction amount of the total volume of each phase. This is done by multiplying each phase's final result by its percent fraction. These final results are then added together to achieve the final results. In other words, the contribution of each phase

to the final result should be proportional to the size of each layer.

6.21.5.2.2 The mathematical combining of each phase's results is done for all samples, unless otherwise requested by the client. The process chosen and percentages determined will be identified in the case narrative that accompanies the report. See Section 10.6 of this Manual for instructions on generating case narratives.

6.21.5.2.3 If one of the phases makes up less than 10% of the total by volume, then the sample is shaken in a best effort to homogenize the sample with any aliquot used for analysis immediately removed prior to the phases again separating. This is to avoid the potential for having to deal with limited volume on the less than 10% phase of the sample.

6.21.5.2.4 If multiple jars are provided, all the jars (except those used for volatile analysis) will be combined into a larger jar before determining the percentage of each layer, or if there is one large bottle (1L or larger) that is most representative of the entire sample, it may be used to determine the percentages of each layer. This is allowed due to the difficulty of filling some of the smaller bottles with narrow openings for some of the wastes being sampled.

6.21.5.3 Any sample that is submitted in many small jars that vary in physical characteristics will be combined in a larger jar, thoroughly mixed together and then transferred back to the smaller containers, if necessary. (Except those sample volumes used for volatile analysis. Any discrepancy in uniformity will be noted in the case narrative.) This will ensure the most representative sample possible for the sample throughout all departments that will be analyzing the sample.

6.21.6 Non-traditional sample matrices

6.21.6.1 This could include building or construction materials, filters (oil or air), or wood debris.

6.21.6.2 These materials will be broken into smaller pieces using whatever means are necessary, such as a hammer, drill, saw, etc., until a usable sample size is obtained.

6.21.6.3 These matrices are typically supplied for TCLP procedures and may not allow for them to be broken down into the method required size but will be broken down to the smallest pieces the matrix will allow. Any samples that cannot be taken to the appropriate smallest size (such as 3/8" or smaller for TCLP analysis) shall have that explained in the case narrative.

6.22 Sub-sample Labeling and Identification

6.22.1 For all samples that are received, and the individual bottle is only used for one test, the letter identification process will be carried out to any subsequent containers used in the analytical process and no further identification will be needed. The labeling and identification process for the individual bottles associated with a given sample is explained in section 6.11.3 – 6.11.4 of this Quality Manual.

6.22.1.1 Example of this type of situation would be VOC vials and the amber bottles for semi-volatiles.

6.22.2 In addition to the sample ID assigned by LIMS in section 6.11 of the Quality Manual, an additional identifier will be included on each individual container that is used in the analytical process.

6.22.2.1 The types of containers can be defined as:

- 6.22.2.1.1 Flasks
- 6.22.2.1.2 Vials
- 6.22.2.1.3 Disposable sample cups
- 6.22.2.1.4 Digestion Tubes
- 6.22.2.1.5 Weighing boats
- 6.22.2.1.6 Crucibles
- 6.22.2.1.7 Petri dishes
- 6.22.2.1.8 Separatory funnels
- 6.22.2.1.9 Zymark tubes

NOTE: This list may not be all-inclusive, and if another type of container is used, it must be uniquely identified and traceable to the sample number and test involved.

6.22.3 The ID with identifier will be entered into any digestion, analytical or prep logbook to ensure traceability from the container to the logbook.

6.22.4 The process of labeling sample aliquots will only encompass intermediate containers being used for all automated analyses. If the analysis utilizes an autosampler, the autosampler vials, cups, or tubes will not be uniquely identified, primarily due to space constraints.

6.22.4.1 The autosampler container will be uniquely identified by use of the autosampler location and the accompanying sample info file, however named, which will directly relate back to the original assigned number and have a reference to the test being performed. The sample info file will be required to reference the sample number in its entirety and include an unambiguous reference to the analysis being performed. The analysis reference will either be the method/parameters/test name or instrument, if the instrument uniquely identifies the type of analysis.

6.22.4.1.1 Sample info files are referred to as shot logs, run logs, autosampler lists, etc. by the different departments.

- 6.22.5 Non-automated analyses will have the ID and identifier applied to all sample containers in use throughout the analytical process, once the aliquot is taken from the original container.
- 6.22.6 This identifier will be linked to the test by an acronym as explained below, such as J1102201-01A-OP, for the portion of Sample number J1102201-01 that was in bottle A and then a subsample was taken out for the orthophosphate analysis.
- 6.22.7 Acceptable acronyms. Any not included in this list will utilize a similar form of abbreviation and will be explained on the cover sheet or logbook that accompanies each analysis and will be listed in the definitions section of the analytical SOP. Typical abbreviations are listed below and usually defined by either creating an acronym from the first letter of each word in the test or the chemical symbol.

- 6.22.7.1 Chemical Oxygen Demand = COD
- 6.22.7.2 Orthophosphate = OP
- 6.22.7.3 Carbonaceous Biochemical Oxygen Demand = CBOD
- 6.22.7.4 Biochemical Oxygen Demand = BOD
- 6.22.7.5 Total Suspended Solids = TSS
- 6.22.7.6 Total Dissolved Solids = TDS
- 6.22.7.7 Fluoride = F
- 6.22.7.8 Chloride = Cl
- 6.22.7.9 Nitrate = NO₃
- 6.22.7.10 Nitrite = NO₂
- 6.22.7.11 Nitrate + Nitrite = NO_x
- 6.22.7.12 Total Phosphorus = TP
- 6.22.7.13 Total Kjeldahl Nitrogen = TKN
- 6.22.7.14 Ammonia = NH₃
- 6.22.7.15 Alkalinity = Alk
- 6.22.7.16 Bromate = BrO₃

| | |
|-----------|---------------------------|
| 6.22.7.17 | Sulfate = SO ₄ |
| 6.22.7.18 | Mercury = Hg |
| 6.22.7.19 | ICP Metals = ICP |
| 6.22.7.20 | GFAA Metals = GFAA |
| 6.22.7.21 | Total Coliform = TC |
| 6.22.7.22 | Fecal Coliform = FC |
| 6.22.7.23 | Turbidity = Turb |
| 6.22.7.24 | pH = pH |
| 6.22.7.25 | Color = color |
| 6.22.7.26 | PAH = PAH |
| 6.22.7.27 | FL-PRO = FL-PRO |
| 6.22.7.28 | BNAs or SVOCs = BNAs |
| 6.22.7.29 | Pesticides = Pest |
| 6.22.7.30 | PCBs = PCB |
| 6.22.7.31 | DRO = DRO |
| 6.22.7.32 | Haloacetic Acids = HAA |
| 6.22.7.33 | Endothall = 548 |
| 6.22.7.34 | Glyphosate = 547 |
| 6.22.7.35 | Diquat = 549 |
| 6.22.7.36 | Ethylene Dibromide = EDB |
| 6.22.7.37 | TCLP Metals = TM |
| 6.22.7.38 | TCLP SVOCs = TS |
| 6.22.7.39 | TCLP VOCs = TV |
| 6.22.7.40 | TCLP Herbicides = TH |
| 6.22.7.41 | TCLP Pesticides = TPest |
| 6.22.7.42 | SPLP Metals = SM |
| 6.22.7.43 | SPLP SVOCs = SS |
| 6.22.7.44 | SPLP VOCs = SPV |
| 6.22.7.45 | SPLP Pesticides = SPest |
| 6.22.7.46 | SPLP Herbicides = SH |
| 6.22.7.47 | SPLP PAH = SPAH |
| 6.22.7.48 | SPLP BTEX = SB |
| 6.22.7.49 | SPLP PCBs = SPCB |
| 6.22.7.50 | SPLP FL-PRO = SF |

6.23 Reviews of Contracts, Requests, or Tenders

See also ADMIN SOP-027 Data Quality Objectives.

6.23.1 Reference Section 6.17 for the Data Quality Objectives that are completed before any new contracts are attempted.

6.23.2 Reference Section 6.18 for New Work Acceptance mechanisms once the work is attained. This section details how new work is obtained.

- 6.23.3 All bids and contracts are completed in direct correlation to the Business Development Plan utilized by the Sales Force of AEL. The Sales Force includes the General Manager, the Laboratory Director, and the Laboratory Managers.
 - 6.23.4 All contracts are reviewed for laboratory capabilities and compliance with methods and/or regulatory standards before bids or contracts are processed. The DQO from Section 6.17 is a helpful tool for accomplishing this aspect of the bidding process.
 - 6.23.5 The sales staff maintains all contracts. Any discrepancies between the laboratory and the contract are worked out before the receipt of samples, if at all possible.
 - 6.23.5.1 In the event there is a discrepancy that arises after samples have been received and/or analyzed, the contract may be revised per the client's instructions. If a revision is completed, this revision will replace the original contract.
 - 6.23.6 All original and revised contracts are maintained in the custody of the sales staff, archived by the sales staff and destroyed in accordance with the processes outlined in Section 4.0 of this Manual.
 - 6.23.7 Any deviation from the contract that is discovered throughout the analytical process requires written notification to the client of the deviation. If this deviation causes a revision of the contract, then see Section 6.23.5.
 - 6.23.8 Any change in the certification status or capabilities of the laboratory will be communicated to the client and the contract will be revised accordingly.
 - 6.23.9 Any revisions to the contract will go through the same review progress as the original contract.
- 6.24 Contingencies for handling samples in the event of a power loss, natural disaster, terrorist strike, or other unforeseeable occurrence.
- 6.24.1 Most actions to be taken will be heavily dependent upon the severity and duration of the event. It will be the responsibility of the technical directors to determine the best course of action in relation to the event. As always, personnel safety will come first. Second to that, an attempt to preserve the samples to such a time that they can be analyzed should be made. If samples that require thermal preservation are noted to rise above acceptable limits, the client should be notified to determine whether to analyze and qualify or to resample. It should also be noted that the

regulatory agencies may make allowances to hold time and thermal preservation during such an event and should be contacted as to the acceptability of qualified results. Please see also the AEL Chemical Hygiene Plan and Health and Safety Manual for greater detail of AEL's emergency planning.

- 6.24.2 **In the event of a power loss.** Limit access to the walk-in coolers and refrigerators to conserve the cold. If events necessitate, use ice if available and ice down samples in coolers while still in the walk-in. If the duration of the power loss looks to be beyond the ability to maintain thermal preservation limits, and if the AEL sister labs can take those samples, then arrangements should be made to move the samples to one of those locations.
- 6.24.3 **In the event of a hurricane.** Avoid scheduling sampling and discourage clients from sampling just prior to the event if predictions are being made for landfall locally. Secure the samples and clear the area as directed by any hurricane forecast alerts. Personnel safety becomes the priority at this point. If time allows and if the AEL sister labs can take those samples, then arrangements should be made to move the samples to one of those locations. The lab's Technical Director will make the determination as to the feasibility and necessity to move samples.
- 6.24.4 **In the event of any unforeseeable occurrence.** Personnel safety again comes first. The technical directors will determine the best course of action in relation to the event to best preserve sample integrity.

Figure 6.2

Log-in Checklist

1. Were custody seals on shipping container(s) intact?
2. Were custody papers properly included with samples?
3. Were custody papers properly filled out (ink, signed, match labels)?
4. Did all bottles arrive in good condition (unbroken)?
5. Were all bottle labels complete (sample #, date, signed, analysis, preservatives)?
6. Did the sample labels agree with the chain of custody?
7. Were correct bottles used for the tests indicated?
8. Were proper sample preservation techniques indicated on the label?
9. Were samples received within holding times?
10. Were all VOA vials free of the presence of air bubbles?
11. Have all Soil VOA Vials and Encores been placed in a freezer within 48 hours of collection?
12. Were samples in direct contact with wet ice? If "No," check one: NO ICE BLUE ICE
13. Was the cooler temperature less than 6°C?
14. Where pH preservation is required, are sample pHs checked and any anomalies recorded by Sample control? Are all <2 or >10? Note: VOA samples are checked by laboratory analysts.
15. Was sufficient sample volume provided to perform all tests?
16. If for Bacteriological testing, were containers supplied by AEL? (See QA officer if answer is no)
17. Were all sample containers provided by AEL? (Other than Bacteriological)
18. Were samples accepted into the laboratory?
19. When necessary to split samples into other bottles, is it noted in the comments?
20. Where Encores received and if so, how many?

If any of the above items from the checklist are not met, it is to be noted in the "Sample Acceptance Discrepancies Log".

Also use the "Sample Acceptance Discrepancies Log" to note any other anomalous events or issues arising in sample log-in.

Figure 6.3 (For posting in Log-in)

Sample Acceptance Policy

AEL will accept samples that are received if all the following factors are met:

- 1 The samples are in the correct containers. See AEL Quality Manual appendices for a listing of the required containers and holding times.
- 2 The samples are submitted within the recommended hold time and there is sufficient time for analysis before the hold time will expire.
- 3 The samples are preserved properly, including thermal preservation.
- 4 The samples are submitted with a Chain of Custody that is correctly filled out and adequately transfers custody of samples from the client/sampler/courier to the laboratory.
- 5 The samples bottles are clearly labeled, unambiguous, and correctly correspond to the Chain of Custody.
 - 5.1 This includes the samples bottles being correctly identified with the client sample ID, collection time, and collection date in addition to the collector's initials.
- 6 The Chain of Custody correctly and completely identifies the analytical tests and methods need for each sample submitted.
- 7 There is sufficient sample volume to effectively complete the required tests and the sample bottles are correctly filled, i.e. zero-headspace vials, in reference the analysis of volatile organic compounds in water.
- 8 All field sheets must be submitted and complete at the time of sample drop-off. This applies to all samplers employed or subcontracted by AEL.
- 9 Any sample that does not meet all of the conditions mentioned above will have the exception documented and depending upon the circumstances, will either be qualified accordingly or rejected entirely.
- 10 Labels should be durable and water resistant. Use an indelible ink pen or Sharpie when writing on sample labels.
- 11 There should be adequate sample volume to perform the necessary tests (Including a matrix spike and spike duplicate if this sample is randomly selected from the test batch for this purpose. For table of volumes-see fig. 6.6 in QM.)
- 12 See the AEL Quality Manual section 6 for greater detail on Sample Acceptance.
- 13 AEL reserves the right to refuse any samples that are of a known or suspected high hazard that may be at concentrations levels the laboratory(s) is not equipped to process.
- 14 AEL reserves the right to refuse any samples that are not accompanied by written disclosure of the known or suspected presence of a known or suspected high hazard.

Figure 6.4

Recommended Number of Sample Containers and Sample Volume to Ensure that Adequate Sample is Available for the Analysis of Matrix Spike and Spike Duplicates When Randomly Selected.

| Test Group | Methods | References | Container | Number of Containers Required |
|-----------------------|--|--|---------------------------------|---|
| Volatile Organics | All Water Matrices-Purge and Trap GC and GC/MS | 502.2, 524.2, 602, 624, 8021, 8260 | 40 ml Vial | 3 Vials for all samples |
| Semivolatile Organics | Non-potable Water Matrix GC-GC/MS | 608, 608.2, 625, Flo-Pro, Dro, 8081, 8082, 8270 | 1 Liter Amber | 3 Liters for at least one sample per every 10 |
| Semivolatile Organics | Non-potable Water Matrix GC-GC/MS micro extraction | 504.1, 8011 | 40 ml Vial | 3 Vials for all samples |
| Semivolatile Organics | Drinking Water Matrix GC, HPLC | 504.1, 515.3, 552.2, 547, 531.1 | 40 ml Vial | 3 Vials for all samples |
| Semivolatile Organics | Drinking Water Matrix GC-GC/MS | 508, 525.2, 548.1 | 1 Liter Amber | 3 Liters for at least one sample per every 10 |
| Semivolatile Organics | Drinking Water Matrix HPLC | 549.2 | 1 Liter Amber Plastic | 1 Liter for every sample |
| Volatile Organics | Soil Matrix--Purge and Trap GC and GC/MS | 8260 | 40 ml Vials Soil Vial or Jar | 2 DI Vials for each sample plus 1 MEOH Vial and 1 4 oz soil jar or vial |
| Semivolatile Organics | Soil Matrix GC-GC/MS | Flo-Pro, Dro, 8081, 8082 8270, PAH-8270 Sims | 8 oz Soil Jar | One 8 oz soil Jar |
| All Metals | Drinking Water Matrix | All Test Methods | 1 Liter Plastic | One 1 Liter Plastic |
| All Metals | Non-potable Water Matrix | All Test Methods | 500ml Plastic | One 500 ml Plastic |
| All Metals | Soil Matrix | All Test Methods | 4 oz Soil Jar | One 4 oz soil Jar |
| Microbiology | DW and Non-potable Water | All Test Methods | 100ml Thio Cup | One 100ml Thio Cup |
| Wet Chemistry | Water Matrices | Alkalinity, TDS, Surfactants TKN, Bromide, Bromate, Chlorate, Chlorite, Ammonia, Total Phosphorus, Flashpoint | 250ml Plastic | One 250 ml per Test |
| Wet Chemistry | Water Matrices | Cyanide | 500ml Plastic | One 500 ml per Test |
| Wet Chemistry | Water Matrices | BOD, CBOD, Odor, TSS, Chlorophylls, Phenol | 1 Liter | One 1-liter Bottle per Test |
| Wet Chemistry | Water Matrices | Cl, FI, Sulfate, COD, Color, pH, Conductivity, Nitrate, Nitrite, ortho Phosphorus, Turbidity | 125ml Plastic | One 125ml Plastic per Test |
| Wet Chemistry | Water Matrices | Oil & Grease, TPH | 1 Liter | One extra Liter per sampling site for every 20 collected at that site |
| Wet Chemistry | Soil and Sediment | All Test Methods | 8 oz Jar | One 8 oz Jar |

Please note: For preservation and bottle type, please see the tables listed in the appendix of section 6.

Figure 6.5

STANDARD OPERATING PROCEDURES For EPA Method 5035 (Low Level Soil Sampling)

METHOD SUMMARY

This method describes a closed system for the analysis of volatile organic compounds (VOCs) in solid materials (e.g. soils, sediments, and solid waste). The applicable concentration range of the low soil is approximately between 0.5 to 200 µg/kg.

PROCEDURE USING A DISPOSABLE SYRINGE

1. Prepare soil samples in the field as outlined in your FDEP QA manual. Be sure not to disturb the soil, which could volatilize the low concentration of volatile compounds.
2. Position the bottom of the plunger of the 10-cc disposable polypropylene syringe to the 3-cc mark.
3. Force the syringe into the soil sample so that it compresses into the entire space.
4. Remove any excess from the end of the opening and outside of the syringe.
5. Open a 40-ml vial containing preservative (DI water or Methanol) and force the contents of the syringe into the pre-weighed vial, remove any soil around the threads of the vial and replace the top.
6. Gently shake the vial so that the soil is in the liquid.
7. Repeat steps 3 through 7 with the same syringe for the second and third vials.
8. Additionally, collect the same soil sample in a non-preserved 40mL vial with no headspace and close the container as quickly as possible to minimize the loss of volatile compounds. This is used for possible high-level analysis when necessary.
9. Place the vials back in the foam holder.
10. Store samples on ice at 4⁰C. Sample holding time is 48 hours from time of sampling for either analysis or freezing for the DI preserved vials. If the vials are frozen by the client, the DI vials must be laid on their side to avoid breakage of the glass.

Figure 6.6

| | |
|---|--|
|  | Advanced Environmental Labs 6681 Southpoint Parkway Jacksonville, FL 32216 |
|---|--|

Kit Request Form

Client: _____

Special Instructions: _____

Kit Request Date: _____

Date Kit Needed By: _____

| Qty. | Bottle Type | Tests Needed |
|-------|--|--------------|
| _____ | 125 mL plastic, unpreserved | _____ |
| _____ | 125 mL plastic, w/ HCl | _____ |
| _____ | 125 mL plastic, w/ HNO ₃ | _____ |
| _____ | 125 mL plastic, w/ H ₂ SO ₄ | _____ |
| _____ | 250 mL plastic, unpreserved | _____ |
| _____ | 250 mL plastic, w/ HCl | _____ |
| _____ | 250 mL plastic, w/ HNO ₃ | _____ |
| _____ | 250 mL plastic, w/ H ₂ SO ₄ | _____ |
| _____ | 500 mL plastic, unpreserved | _____ |
| _____ | 500 mL plastic, w/ HCl | _____ |
| _____ | 500 mL plastic, w/ HNO ₃ | _____ |
| _____ | 500 mL plastic, w/ H ₂ SO ₄ | _____ |
| _____ | 1 L plastic, unpreserved | _____ |
| _____ | 1 L plastic, w/ HCl | _____ |
| _____ | 1 L plastic, w/ HNO ₃ | _____ |
| _____ | 1 L plastic, w/ H ₂ SO ₄ | _____ |
| _____ | 1 L amber, unpreserved | _____ |
| _____ | 1 L amber, w/ H ₂ SO ₄ | _____ |
| _____ | 4 oz jar | _____ |
| _____ | 8 oz jar | _____ |
| _____ | 3-40 mL vials, unpreserved | _____ |
| _____ | 3-40 mL vials, w/HCl | _____ |
| _____ | 3-40 mL vials, w/NaThio | _____ |
| _____ | Soil Syringe Kit | _____ |
| _____ | Cyanide preserved w/ NaOH _____ mL bottle | _____ |
| _____ | Sulfide preserved w/ NaOH O& ZnAcetate _____ mL bottle | _____ |
| _____ | Coliform cups, w/NaThio | _____ |
| _____ | Tevlar Bag | _____ |
| _____ | Other _____ | _____ |

Kit Built By: _____ Date: _____ Kit Delivered By: _____ Date: _____

Figure 6.7



Data Quality Objectives Form (For setting up all new clients / unique projects)

All new clients are Pre-Pay / CASH in advance
unless approved for credit by Corporate.

Check One:

- Prepayment Required (MUST Note in LIMS using the PO Field in Profile)
- Credit Approved by Corporate

Client Name: _____ Federal Client: Yes / No
Client Address: _____
Client Contact: _____ Contact Email: _____
Project Name: _____ Start Date: _____
Sampling Required? Yes / No (Attached Sampling Form if Yes) Required TAT: _____
AEL Primary Lab _____ AEL PM: _____
LIMS ID (Approved by Lab Mngr): _____ AEL Sales Rep: _____
Must have complete contact info for client Accounting Dept: POC Name, address, email, and phone.

1. What target levels are required? Circle Rule and level. (Get list of MCLs or old lab report if analysis not tied to a rule)
62-550 Drinking Water 62-770 Petroleum TCLP, SPLP NPDES Permit Levels
62-777 Contaminant CTLs (GW, FSW, MSW, GWLY) (Soil-DE Commercial or Residential) Client Will Provide Specific Target Levels

2. What will the data be used for-compliance, monitoring, non-regulatory, etc.? _____

3. How would the client like the report delivered? (Circle one): E-mailed Faxed Hard Copy

4. Is specific or custom EDD required (name it)? _____

5. Do We Provide Pre-labeled Bottles or Printed COC? _____

6. Do we send invoice to someone other than the PM? **Yes / No**

7. If Yes to #6, to whom and how are invoices sent to others? Include the name, address, email, phone of other recipients)

8. Are there any special project requirements or report deliverable level? (warnings/sample history such as "hot" samples, always been clean, expected clean, etc.; special analyte lists). {Note: Test groups are specific analytes [RCRA / TCLP / Priority Pollutants], but most organic methods have no specific list (i.e. 8260, 8270, 8141, 8151)}. If client asks for a method – you MUST ask for an analyte list or provide the client with AEL's official list. Add sheets if needed.

Person Filling Out Form: _____ Date DQO Completed _____

AEL Bottle Kit Preparation Guidelines with Cross Comparison Chart of Analytical Methods and FDEP SOP Tables

Making acid and base mixes of 1:1 to be performed by trained personnel in the lab. Measure water first in a Pyrex beaker, then in a hood while wearing gloves, lab coat, and safety glasses, add concentrated acid slowly to the water. Never mix directly in glass jars or bottles as the bottoms can break out with heat stress. Sulfuric acid mixes will become very hot, so go slow in 50ml increments and pause for 20mins between each increment. Take safety precautions against hot containers. Allow 5 hours to cool before handling and/or use ice bath. Sodium Hydroxide when mixing and when made from pellets will also become very hot. Use similar precautions as those used for acid mixing.

| | | |
|-----------------------------|------------------------|----------------------------|
| NH4Cl=Ammonium Chloride | ZnAc= Zinc Acetate | Na2S03=Sodium Sulfite |
| HNO3= Nitric Acid | H2SO4=Sulfuric Acid | MCAA=Monochloroacetic Acid |
| Na2S2O3= Sodium Thiosulfate | NaOH= Sodium Hydroxide | HCl= Hydrochloric Acid |

| Waters / Aqueous | Comments | Preservation makeup | Acid and Reagent Mix | EPA (or SM) Method | FDEP FS1000 |
|--|--|---|---|--|---|
| Volatile Organics by GC/MS 624 2-40 ml Vials Unpreserved, 1 HCL Preserved 624 | Samples are to be run within 7 days. Preserved vial used only if 7 days cannot be met. Running in 7 days gives best recoveries for 2-chlorovinyl ethyl ether and MTBE. Acrolien must be run in 3 days if unpreserved, otherwise sample preserved at 4-5 pH | 2 unpreserved, 1 preserved vial. Preserved vial purchased from vendor pre-preserved and in foil pack to prevent contamination. If inventory low, prep acid vials in volatiles department by adding 500ul of 1:1 HCL to 40ml vial. | Pesticide grad or better HCL mixed with ultrapure DI water from volatiles department at a ratio of 1:1. | 624-if residual chlorine present add 10mg/40mls sodium thiosulfate prior to shipping. If aromatics run-collect separate vial-pH <2 with 1:1 HCL | 624-Purgeable Aromatics, sodium thiosulfate, ph <2. Purgeable halocarbons sodium thiosulfate, no pH adjustment. Acrolein + acrylonitrile ph 4-5, all 14 days |
| Volatile Organics by GC/MS 8260/624 2-40 ml Vials Unpreserved, 1 HCL Preserved 8260 | Samples are to be run within 7 days. Preserved vial used only if 7 days cannot be met. Running in 7 days gives best recoveries for 2-chlorovinyl ethyl ether. Acrolien must be run in 3 days if unpreserved, otherwise preserved at 4-5 pH | 2 unpreserved, 1 preserved vial. Preserved vial purchased from vendor pre-preserved and in foil pack to prevent contamination. If inventory low, prep acid vials in volatiles department by adding 500ul of 1:1 HCL to 40ml vial. | Pesticide grad or better HCL mixed with ultrapure DI water from volatiles department at a ratio of 1:1. | 8260-If residual chlorine present, add 4 drops (AEL-100ul) of 10% sodium thiosulfate solution. If MTBE is to be analyzed-do not acidify samples, analyze in 7 days. Acrolein, acrylonitrile adjust pH to 4-5, 7 days | 8260-Purgeable Aromatics, sodium thiosulfate, ph <2. Purgeable halocarbons sodium thiosulfate, no pH adjustment. 14 days. Acrolein + acrylonitrile ph 4-5, 7 days |
| Semi-volatile Extractable Organics by GC/MS 625 1 L Amber Glass | grab enough sample volume for QC to be performed. If a single sample, must have duplicate, if multiple samples, a duplicate sample every set and/or every 10 samples. | 1 one liter amber unpreserved , plus QC. No acid, but method asks for Testing Cl to ensure not present | Weight 40 grams reagent grade sodium thiosulfate and add to 500mls of DI water. Then add 1ml of this solution to 1 liter of sample. | If residual chlorine present, add 80mg sodium sulfate per liter | Sodium Thiosulfate 0.008%, store in dark, 6 degrees, 7 days |
| Semi-volatile Extractable Organics by GC/MS 1 L Amber Glass, 8270 | grab enough sample volume for QC to be performed. If a single sample, must have duplicate, if multiple samples, a duplicate sample every set and/or every 10 samples. | 1 one liter amber unpreserved , plus QC. No acid, but method asks for Testing Cl to ensure not present | Weight 40 grams reagent grade sodium thiosulfate and add to 500mls of DI water. Then add 1ml of this solution to 1 liter of sample. | If residual chlorine present, add 3 mls of 10% sodium thiosulfate solution per gallon (or 0.008%) | Sodium Thiosulfate 0.008%, store in dark, 6 degrees, 7 days |
| Polynuclear Aromatic Hydrocarbons 1 L Amber Glass, 8270-SIM | if sample is stand alone test, unpreserved 1 ltr amber okay, (plus QC volume) if sampling also for FL-PRO, then preserved | 1 unpreserved amber liter , unless to be run with FL-PRO, then 2mls of 1:1 H2SO4 (HCL if from client is acceptable also) | Weight 40 grams reagent grade sodium thiosulfate and add to 500mls of DI water. Then add 1ml of this solution to 1 liter of sample. | If residual chlorine present, add 3 mls of 10% sodium thiosulfate solution per gallon (or 0.008%) | Sodium Thiosulfate 0.008%, store in dark, 6 degrees, 7 days |

| FL-PRO / TRPH / DRO 1 L Amber Glass (H2SO4) | FL-PRO samples must be acid preserved. One amber liter (plus QC volume) preserved with H2SO4 to a pH < 2 | 1 one liter amber with 2mls of 1:1 H2SO4 1:1 (HCL if from client is acceptable also) Grab one duplicate per batch, batch consisting of 10 samples or less. | 2mls of 1:1 H2SO4 to each amber liter | N/A | Screw cap 1 liter glass bottles. screw cap lids with Teflon liners. Sulfuric or hydrochloric acid preservation <2, stored at 4 degrees C. Extract in 7 days. |
|---|---|--|--|--|--|
| Waters / Aqueous | Comments | Preservation makeup | Acid and Reagent Mix | EPA (or SM) Method | FDEP FS1000 |
| Chlorinated Pesticides 1 L Amber Glass, 608 | DO NOT acid preserve. Can damage recoveries. pH should be neutral. If pH is not within 5.0-9.0, adjust to neutral pH of 7 with 1:1 sulfuric acid or 1:1 sodium hydroxide as needed. This shall be done at the lab upon receipt. | 1 one liter amber, unpreserved. Method asks for <u>Testing Cl</u> to ensure not present. Grab one duplicate per batch, batch consisting of 10 samples or less. | Normally unpreserved in bottle kits. pH adjustment at lab | All samples iced at 4 degrees C. If not extracted within 72 hours, adjust to pH of 5.0 to 9.0 with either sodium hydroxide or sulfuric acid. Record the type and amount of preservation used. Add sodium thiosulfate when residual chlorine present. | Cool to 6 degrees C, adjust ph to between 5.0 - 9.0. pH adjustment can be made upon receipt at the lab or omitted if extracted within 72 hours. If testing for Aldrin, add 0.008% Sodium thiosulfate. Extract in 7 days. |
| Chlorinated Pesticides 1 L Amber Glass, 8081 | Grab enough sample volume for QC to be performed. If a single sample, must have duplicate, if multiple samples, a duplicate sample every set and/or every 10 samples. | 1 one liter amber, unpreserved. Method asks for Testing Cl to ensure not present. Grab one duplicate per batch, batch consisting of 10 samples or less. | Normally unpreserved in bottle kits. pH adjustment at lab | If residual chlorine present, add 3 mls of 10% sodium thiosulfate solution per gallon (or 0.008%) | Cool to 6 degrees C, adjust ph to between 5.0 - 9.0. pH adjustment can be made upon receipt at the lab or omitted if extracted within 72 hours. If testing for Aldrin, add 0.008% Sodium thiosulfate. Extract in 7 days. |
| PCB's only 1 L Amber Glass, 8082/608 | Grab enough sample volume for QC to be performed. If a single sample, must have duplicate, if multiple samples, a duplicate sample every set and/or every 10 samples. If running with 8081 analysis, collect for 8081 and 8082 samples can be run from 8081 volume. | 1 Liter amber unpreserved. When collected to be analyzed with 8081 samples, use 8081 instructions. Grab one duplicate per batch, a batch consisting of 10 or less samples. | N/A. When collected to be analyzed with 8081 samples, use 8081 instructions. | Cool to 6 degrees, hold time, none listed | Hold times 1 year prior to extraction, 1 year after extraction. |
| Organophosphorous Pesticides 1 L Amber Glass, 8141 | DO NOT acid preserve. Can damage recoveries. pH should be neutral. If pH is not within 5.0-8.0, adjust to neutral pH of 7 with 1:1 sulfuric acid or 1:1 sodium hydroxide as needed. This shall be done at the lab upon receipt. | Adjust 1 liter amber to neutral pH , then thermal preservation only, treat with sodium thiosulfate if residual chlorine is present. Grab one duplicate per batch, batch consisting of 10 samples or less. | N/A | Collect on ice at 4 degrees C, store at <6 degrees C | pH at 5.0- 9.0 (adhere to the 5.0-8.0 as required per method), extract within 7 days |
| Chlorinated Herbicides 1 L Amber Glass, 8151 | Thermal preservation only, treat with sodium thiosulfate if residual chlorine is present. | 1 liter amber unpreserved , treat with sodium thiosulfate if residual chlorine is present. | N/A | If residual chlorine present, add 3 mls of 10% sodium thiosulfate solution per gallon (or 0.008%) | 7days to extract, 40 days after extraction |
| Explosives 8330 | Thermal preservation only, treat with sodium thiosulfate if residual chlorine is present. | 1 one liter amber unpreserved , treat with sodium thiosulfate if residual chlorine is present. Grab one duplicate per batch, batch consisting of 10 samples or less. | Weight 40 grams reagent grade sodium thiosulfate and add to 500mls of DI water. Then add 1ml of this solution to 1 liter of sample only when chlorine present. | If residual chlorine present, add 3 mls of 10% sodium thiosulfate solution per gallon (or 0.008%) | 7days to extract, 40 days after extraction |

| | | | | | |
|---|--|---|--|--|---|
| TCLP EPA 1311 SPLP EPA 1312 1 L Amber Glass | Aqueous samples in 1 liter amber glass. | 1 liter amber unpreserved. (if waste sample, see smaller volumes and hold times under soil/waste) | N/A | Thermal preservation only | Volatiles 14 days to extract, 14 days form prep to analyze. Semivolts 14 days to extract, 7 days to prep extract, 40 days to analyze. Mercury 28 days to extract, 28 days to analyze. Metals 180 days to extract, 180 days to analyze |
| Ethylene Dibromide (EDB) 3-40 ml Vials (NaS2O3), 8011/504(DW) | 40ml vials get 3mg of sodium thio crystals just prior to shipping | 3 vials each sample with sodium thiosulfate, or prepreserved from vendor with certificate of analysis stating preservation and amount. | Add 20 grams reagent grade sodium thiosulfate to 250mls of ultrapure DI water. Then add 40 ul of this solution (TV=80mg/ml) to each 40ml vial. | 75ul of 40mg/ml to each empty 40ml vial, 4 degrees C. | sodium thiosulfate, 4 degrees C, then 14 days hold |
| Waters / Aqueous | Comments | Preservation makeup | Acid and Reagent Mix | EPA (or SM) Method | FDEP FS1000 |
| 8 RCRA Metals / Priority Pollutant Metals/ Full list Metals 500 ml Plastic (HNO3) | 1 500 ml plastic poly with 1:1 nitric. pH is checked at the bench prior to analysis. If adjustment needed, wait 24 hrs to analyze. | For each 500mls sample, add 2mls of 1:1 nitric acid. pH checked at the bench prior to analysis. If adjustment needed, wait 24 hrs to analyze. | Slowly pour 500mls of concentrated nitric acid into 500mls ultra pure di water to make 1:1mix | Use 1:1 Nitric Acid and preserve to <2, good for 180 days, (except Chrome VI, Hg, 28 days) | Use 1:1 Nitric Acid and preserve to <2, good for 180 days, (except Chrome VI, Hg 28, days) |
| As/Cd/Cr/Pb 250 ml Plastic (HNO3) | 1 250 ml plastic poly with 1:1 nitric. pH is checked at the bench prior to analysis. If adjustment needed, wait 24 hrs to analyze. | For each 250mls sample, add 1mls of 1:1 nitric acid. pH checked at the bench prior to analysis. If adjustment needed, wait 24 hrs to analyze. | Slowly pour 500mls of concentrated nitric acid into 500mls ultra pure di water to make 1:1mix | Use 1:1 Nitric Acid and preserve to <2, good for 180 days, (except Chrome VI, 28 days) | Use 1:1 Nitric Acid and preserve to <2, good for 180 days, (except Chrome VI, 28 days) |
| Sulfate, Chloride, Nitrate, Nitrite, TDS, TSS, Alkalinity 1 one liter plastic unpreserved | 1 one liter polyethylene plastic. Do not preserve. (If testing for Nitrate plus Nitrate (NOX) only, then can preserve with 1:1 sulfuric to extend hold time.) If testing for an individual parameter, smaller volumes may be used. Example: Nitrite 125ml, unpreserved. | Unpreserved. (If analyzing for NOX only, then can pH 250ml sample with 1:1 sulfuric and analyze by other than IC.) If testing for an individual parameter, smaller volumes may be used. Example: Nitrite 125ml, unpreserved. | Unpreserved. (If analyzing for NOX only, then 250ml sample with 1:1 sulfuric and analyze by other than IC.) | Run Nitrate, Nitrite, within 48 hours when unpreserved. (If testing for Nitrate plus Nitrate (NOX) only, then preserve with 1:1 sulfuric to extend hold time to 28 days.) | Thermally preserve at < 6 degrees C. Run Nitrate, Nitrite within 48 hours. Chloride, Sulfate, Fluoride hold time of 28 days, Alkalinity 14 days. TSS, TDS 7 days. |
| TSS | 1 one liter polyethylene plastic. Do not preserve. | 1 one liter polyethylene plastic. Do not preserve. | Unpreserved. | In no case hold sample more than 7 days. Bring samples to room temperature before analysis | Aqueous samples must be preserved at ≤6 °C, run within 7 days |
| (Anions) Sulfate, Chloride, Nitrate, Nitrite, Bromide, Fluoride, Orthophosphate | 125 mls polyethylene plastic. Do not preserve. | 125 mls polyethylene plastic. Do not preserve. | Unpreserved. | Run Nitrate, Nitrite, O-Phos within 48 hours when unpreserved. Sulfate, Bromide, Fluoride, Chloride 28 days. (If testing for Nitrate plus Nitrate (NOX) only, then preserve with 1:1 sulfuric to extend hold time to 28 days.) | Run Nitrate, Nitrite, O-Phos within 48 hours when unpreserved. Sulfate, Bromide, Fluoride, Chloride 28 days. (If testing for Nitrate plus Nitrate (NOX) only, then preserve with 1:1 sulfuric to extend hold time to 28 days.) |
| TDS/Alkalinity | 500 mls polyethylene plastic. Do not preserve. | 500 mls polyethylene plastic. Do not preserve. | Unpreserved. | As soon as practical. | Run within 7 days for TDS, 14 days for alkalinity |
| Ammonia, TKN, Total Nitrogen, Total Phosphorus 250 ml (H2SO4) | 1 250ml polyethylene plastic. Do not over preserve, affects analysis, especially for Ammonia and NOX. Over preservation shall be corrected at bench. | For each 250ml poly, add precisely 500 uls of 1:1 sulfuric acid. | To make 1:1 Sulfuric acid, carefully and slowly pour 250mls of concentrated sulfuric acid into 250mls of ultra-pure di water . Use water bath or let cool for 5 hours. | Ammonia, TKN, TN, TP 6 degrees sulfuric acid preservation, 28 days. | Ammonia, TKN, TN, TP < 6 degrees C, sulfuric acid preservation, 28 days. |

| | | | | | |
|--|--|--|---|--|---|
| Total Phenolics 1 L Amber Glass (H2SO4) | 1 liter glass amber with 1:1 sulfuric acid | For each 1 liter amber , add 2mls of 1:1 sulfuric acid. | To make 1:1 Sulfuric acid, carefully and slowly pour 250mls of concentrated sulfuric acid into 250mls of ultra-pure di water . Use water bath or let cool for 5 hours. | Cool to < 4 degrees C, sulfuric acid preservation, 28 days. | Cool to < 6 degrees C, sulfuric acid preservation, pH <2, 28 days. |
| Orthophosphate 125 ml, field filtered (with syringe and filter) | 1 125ml polyethylene plastic. Unpreserved | One 125ml poly unpreserved | N/A | 48 hour hold time preserved at < 6 degrees C. | 48 hour hold time preserved at < 6 degrees C. |
| Total Organic Carbon 2-40 ml vial (HCL) | 2 preserved vials. Preserved vial purchased from vendor pre-preserved and in foil pack to prevent contamination. If inventory low, prep acid vials in volatiles department using 1:1 HCL | 2 HCL preserved vials. Preserved vial purchased from vendor pre-preserved and in foil pack to prevent contamination. 0.5mls of 1:1 HCL for each 40ml vial. | 0.5mls of 1:1 HCL for each 40ml vial. | If analysis not performed within 2 hours of collection, pH sample to <2 with HCL or Sulfuric acid | Cool to < 6 degrees C, HCL, sulfuric, or phosphoric acid, preservation pH <2, 28 days. |
| Waters / Aqueous | Comments | Preservation makeup | Acid and Reagent Mix | EPA (or SM) Method | FDEP FS1000 |
| Cyanide 8 oz Amber Glass (NaOH) | Never acidify, can release cyanide vapors. Always preserve with NaOH. | one 8 oz amber (250mls) preserved with 2mls 5 Normal NaOH. | 5 Normal made by adding 200g of NaOH pellets (solid) to 1000mls di water. Mix slowly-becomes very hot. Also can be made from 15N concentrate at 1:2 ratio. | Cool to 6 degrees, Standard Methods pH 12 to 12.5, hold time not listed. | Cool to < 6 degrees C, raise pH to >10 with NaOH, add reducing agent of oxidize present, analyze within 14 days. |
| Sulfide SM4500-S-D 500 ml plastic (NaOH+ZnAc) | One 500ml poly prepreserved at lab with zinc acetate and NaOH. | one 500ml poly with 1ml of 2M zinc acetate and 4 mls 5N NaOH. | Zinc Acetate purchased as 2Molar solution or 55grams Zinc Acetate brought up to 250mls with di water, 5 Normal NaOH made by adding 200g of NaOH pellets (solid) to 1000mls di water. Mix slowly-becomes very hot. Also can be made from 15N concentrate at 1:2 ratio. | Add zinc acetate and sodium hydroxide solutions into sample bottle before filling it with sample. Use 0.2 mL 2M zinc acetate solution per 100 mL sample. Increase volume of zinc acetate solution if the sulfide concentration is expected to be greater than 64 mg/L. The final pH should be at least 9. Add more NaOH if necessary. Fill bottle completely | Cool to < 6 degrees C, add zinc acetate plus sodium hydroxide, pH to >9, analyze within 7 days. |
| BOD, CBOD by SM5210B | 1 Liter polyethylene unpreserved. | 1 Liter polyethylene unpreserved. | N/A | Standard Methods states 24hrs from collection. Using FDEPs allowance to extend to 48hrs. | 48 hour hold time preserved at < 6 degrees C. |
| COD EPA 410.4 | 125ml polyethylene with 1:1 sulfuric acid | For each 125ml plastic , add 250 uls of 1:1 sulfuric acid. | To make 1:1 Sulfuric acid, carefully and slowly pour 250mls of concentrated sulfuric acid into 250mls of ultra-pure di water. Use water bath or let cool for 5 hours. | Cool to < 4 degrees C, sulfuric acid preservation, 28 days. | Cool to < 6 degrees C, sulfuric acid preservation <2, 28 days. |
| Oil & Grease 1664 | 1 liter wide-mouth glass amber with 1:1 sulfuric acid | 1 one liter wide-mouth glass amber with 2mls of 1:1 H2SO4 1:1 (HCL if from client is acceptable also) Grab one duplicate per batch, batch consisting of 10 samples or less. | To make 1:1 Sulfuric acid, carefully and slowly pour 250mls of concentrated sulfuric acid into 250mls of ultra-pure di water. Use water bath or let cool for 5 hours. | Cool to < 4 degrees C, H2SO4 or HCL preservation, 28 days. If a sample is known or suspected to contain greater than 500 mg/L of extractable material, collect a proportionately smaller volume of sample. | Cool to < 6 degrees C, sulfuric acid or HCL preservation, pH <2, 28 days. |

| | | | | | |
|---|--|---|--|---|---|
| Color SM2120 | 250ml glass amber unpreserved, pH checked at lab during testing | <u>250ml glass amber unpreserved</u> | N/A | Amber glass or covered plastic to protect from light, Cool to < 4 degrees C, 24 hour hold time | Cool to < 6 degrees C, 48 hours. |
| Odor SM2150B | 1 liter glass amber unpreserved | <u>1 liter glass amber unpreserved</u> | N/A | Glass with Teflon lined caps, never plastic, Cool to < 4 degrees C, analyze as soon as possible | Cool to < 4 degrees C, only 6 hours hold time for Non-potable water. (24 hours on DW) |
| Chromium VI SM3500Cr-D 19th Ed. | 1 250 ml plastic poly. Unpreserved and unfiltered | <u>1 250 ml plastic poly.</u> Unpreserved and unfiltered. | N/A | SM3500Cr-D 19th edition. Analyze within 24 hours for Chromium VI. | FDEP table states 28 days when by Method EPA 218.6 and pH preserved to 9.5 (IC and conversion to Chromate). AEL using SM3500Cr-D method's 24 hour unpreserved procedures. |
| Surfactants SM5540C | One 500ml poly unpreserved. | <u>one 500ml poly unpreserved.</u> | N/A | Not listed | Plastic or glass, Cool to < 6 degrees C, 48 hour hold. |
| Salinity SM2530B | One 250ml glass unpreserved with parafilm seal. Cut small squares of parafilm to go out with containers. When sample collected, stretch parafilm over top of bottle and cap threads, then screw on lid. | <u>One 250ml glass unpreserved with parafilm seal.</u> Cut parafilm to go out with containers. When sample collected, stretch parafilm over top of bottle and cap threads, then screw on lid. | Parafilm to go out with bottle kits | Not listed | Glass, wax seal, 30 days(or more per footnote). Thermal preservation not required. |
| Soils / Waste | Comments | | Preservation makeup | EPA (or SM) Method | FDEP FS100 |
| Volatile Organics by GC/MS T-Handle Syringe w/ 4 vials (2 DI / 1 unpr / 1 MEOH), 8260 | Core 5 grams of sample using the T handle device. (approximately 1inch of soil in tube) Push the core of soil into a preweighted vial containing 5mls di water and stir bar, vials purchased from vendor. Repeat with second vial. For 3rd vial, again collect 5 grams an place into vial containing 5mls Methanol. For 4th vial, fill unpreserved vial fully with soil and cap. If TCLP testing may be needed, collect also a 25gram Encore using an Encore device. (25g Encore must be purchased from lab or provided by client). Deliver to lab with 48 hours and freeze all samples and Encores. | | Thermal preservation only. Freeze volatiles. | Soils must be frozen within 48 hours to -10 degrees C or less. Samples must be analyzed as soon as thawed. Total hold time of 14 days to analyze. | Soils must be frozen within 48 hours to -10 degrees C or less. Samples must be analyzed as soon as thawed. Total hold time of 14 days to analyze. |
| Semi-volatile Extractable Organics by GC/MS 8oz Soil jar, 8270 | Fill 8oz glass jar with soil sample and cap | | Thermal preservation only. | Glass, 8 oz widemouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days extract, 40 days analyze. (E8141 non-FL,7 /40) |
| Polynuclear Aromatic Hydrocarbons 8oz Soil jar, 8270-SIM | Fill 8oz glass jar with soil sample and cap | | Thermal preservation only. | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze |
| FL-PRO / TRPH / DRO 8oz Soil jar | Fill 8oz glass jar with soil sample and cap | | Thermal preservation only. | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze |
| Polynuclear Aromatic Hydrocarbons + FL-PRO 8oz Soil jar | Fill 8oz glass jar with soil sample and cap | | Thermal preservation only. | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze |
| Chlorinated Pesticides 8oz Soil jar, 8081 | Fill 8oz glass jar with soil sample and cap | | Thermal preservation only. | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze |
| PCB's 8oz Soil jar, 8082 | Fill 8oz glass jar with soil sample and cap | | Thermal preservation only. | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze |
| Chlorinated Pesticides + PCB's 8oz Soil jar | Fill 8oz glass jar with soil sample and cap | | Thermal preservation only. | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze |

| | | | | |
|---|--|---|---|--|
| Organophosphorous Pesticides 8oz Soil jar, 8141 | Fill 8oz glass jar with soil sample and cap | Thermal preservation only. | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze (7/40 for out of state) | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze |
| Chlorinated Herbicides 8oz Soil jar, 8151 | Fill 8oz glass jar with soil sample and cap | Thermal preservation only. | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze | Glass, 8 oz wide mouth jar with Teflon lined lid. 14 days to extract, 40 days to analyze |
| EPH/VPK Kit (Sub-Lab Provided) T-Handle Syringe w/ 2 vials (10mL-MEOH) + 4oz Amber | Core 5 grams of sample using the T handle device. (approximately 1inch of soil in tube) Push the core of soil into a preweighted vial containing 10mls MEOH, vials purchased from vendor. Repeat with second vial. For 4oz jar fill with soil and cap. | Thermal preservation only. | 14 days to extract, 40 days to analyze | 14 days to extract, 40 days to analyze |
| TCLP VOC/SVOC/Metals/Pest- Herb 8oz Soil jar | For TCLP SVOC/Metals/Pest/Herb/Volatiles- fill 8oz glass jar with soil sample and cap. | Thermal preservation only. Freeze volatiles. | Total hold time to perform TCLP extraction: 14 days for Organics, 28 days Mercury, 180 days other metals. | For TCLP SVOC/Pest/Herb & Volatiles, 14 days TCLP extraction. 28 days for HG TCLP extraction, 28 days to analyze, 180 days TCLP extraction for other metals |
| Soils / Waste | Comments | Preservation makeup | EPA Method | FDEP FS100 |
| SPLP SVOC/Metals/Pest/Herb 8oz Soil jar | For SPLP SVOC/Metals/Pest/Herb- fill 8oz glass jar with soil sample and cap. For volatiles, collect also a 25gram Encore using an Encore device. (25g Encore must be purchased from lab or provided by client). Deliver to lab within 48 hours and freeze all samples and Encores. | Thermal preservation only. Freeze volatiles. | Volatiles must be frozen within 48 hours to -10 degrees C or less. Samples must be analyzed as soon as thawed. Total hold time of 14 days to analyze. | For SPLP SVOC/Pest/ Herb 14 days SPLP extraction/ 7days prep extraction /40 days analysis. 28 days for HG SPLP extraction, 28 days to analyze, 180 days SPLP extraction for other metals |
| SPLP VOC ENCORE w/ Sampler | For volatiles, collect 8oz jar and also a 25gram Encore using an Encore device. (25g Encore must be purchased from lab or provided by client). Deliver to lab within 48 hours and freeze all samples and Encores. | Thermal preservation only. Freeze Encore. | Volatiles must be frozen within 48 hours to -10 degrees C or less. Samples must be analyzed as soon as thawed. Total hold time of 14 days to analyze. | Volatiles must be frozen within 48 hours to -10 degrees C or less. Samples must be analyzed as soon as thawed. Total hold time of 14 days to analyze. |
| All Metals Except Mercury and Chrome VI | Fill 8oz glass jar with soil sample and cap | None | 6 months to analyze | Collect in glass or plastic, 6 months to analyze |
| Mercury | Fill 8oz glass jar with soil sample and cap | Thermal preservation only. | Collect in glass or plastic, cool to 4 degrees C +/- 2 degrees, 28 days to analyze | Collect in glass or plastic, cool to 4 degrees C +/- 2 degrees, 28 days to analyze |
| Chrome VI | Fill 8oz glass jar with soil sample and cap | Thermal preservation only. | Not stated in method. | Collect in glass or plastic, cool to 4 degrees C +/- 2 degrees, 1 month to extract, 4 days to analyze |
| BTEX/Napthalene/TRPH in Air - TO-18 Tedlar Bag | Fill Teldar bags with sample, deliver to lab as soon as possible. | None | Not stated in method. | 72 hour hold time. |
| Drinking Water | Comments | Preservation makeup | Acid and Reagent Mix | EPA Method |
| For Full SOC kits | For full SOC kits, collect an extra 1 liter glass amber unpreserved for QC and confirmations. | | | |
| 504.1 EDB/DBCP | 3 clear 40ml vials pre-preserved with sodium thiosulfate. | 3 clear 40ml Clear vials, pre-preserved from vendor with sodium thiosulfate. If inventory low, prep vials in volatiles department by adding 50ul of 10% sodium thiosulfate solution. | Weight 40 grams reagent grade sodium thiosulfate and add to 500mls of DI water. Then add 100ul of this solution to 40ml vial. | 40ml vials get 3mg of sodium thiosulfate crystals just prior to shipping |
| | | | | sodium thiosulfate, 4 degrees C, 14 days hold to extract, then 24hrs for analysis. |

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| 508 Pesticides | 1 liter amber (plus QC volume). DO NOT acid preserve. Pre-preserve with sodium thiosulfate. | 1 liter amber (plus QC volume). DO NOT acid preserve. Normally unpreserved in bottle kits. Pre-preserve with sodium thiosulfate. | Weight 40 grams reagent grade sodium thiosulfate and add to 500mls of DI water. Then add 1ml of this solution to 1 liter of sample. | if residual chlorine is present, 80mg of sodium thiosulfate per liter, extracts good for 14 days | sodium thiosulfate, 4 degrees C, 7 days hold to extract, 14 days for analysis |
| 515.3 Herbicides | Bottle kits 3 unpreserved 40 ml amber vials. Test for residual chlorine to ensure not present. | 3 unpreserved 40 ml amber vials. Only if residual chlorine present or expected, add 100ul of 5% sodium sulfite solution to 40ml vial. | Only when chlorine present, Weight 25 grams reagent grade sodium sulfite and add to 500mls of DI water. Then add 50ul of this solution to 40ml vial. | if residual chlorine is present, 4mg of sodium thiosulfate per 50mls to sample bottle (vial) prior to collecting sample. (AEL Note: this preservation will destroy the analysis-if necessary to dechlorinate, use sodium sulfite. From 515.4 sodium sulfite, 2mg/40ml.) | sodium thiosulfate, HCL<2, 4 degrees in dark , 14 days to extract (AEL Note: this preservation will destroy the analysis-if necessary to dechlorinate, use sodium sulfite) |
| 524.2 Volatile Organics | Bottle kits 3 pre-preserved from vendor HCL 40 ml vials. Testing Cl at lab to ensure not present. Add ascorbic acid for regulated volatiles only if residual chlorine anticipated. Vials with sodium thiosulfate if for THMs only. | 3 Vials with HCL - 524.2, 3 vials with sodium thiosulfate if for THMs only. Preserved vial purchased from vendor pre-preserved and in foil pack to prevent contamination. If inventory low, prep acid vials in volatiles department by adding 500ul of 1:1 HCL to 40ml vial. | Pesticide grad or better HCL mixed with ultrapure DI water from volatiles department at a ratio of 1:1. | 25mg ascorbic acid per 40ml vial. (Sodium thiosulfate when for THMs only), then ph <2 1:1 HCL. Ship vials with ascorbic acid, add HCL at site. | Ascorbic Acid, HCL<2, cool to 4 degrees C, 14 days |
| 525.2 Semivolatile GC/MS | 1 liter amber (plus QC volume). Preserved with sodium thiosulfate. Ph to <2 at sampling site. | 1 liter amber, sodium thiosulfate solution at 1ml per liter, ph < 2 with HCL at sampling site. | Weight 40 grams reagent grade sodium thiosulfate and add to 500mls of DI water. Then add 1ml of this solution to 1 liter of sample. | Residual chlorine reduced at sampling site with 40-50mg sodium sulfite per liter. (Note: recoveries affected by sodium sulfite, AEL using sodium thiosulfate as dechlorination agent).Then ph<2 with 6 N HCL | Sodium Sulfite (Note: recoveries affected by sodium sulfite, AEL using sodium thiosulfate as dechlorination agent), Dark, Cool, 4 degrees C, HCL <2, 14 days |
| 531.1 Carbamates | 1 clear 40ml vials with Monochloroacetic acid buffer. Testing Cl to ensure not present. | 1 clear 40ml vials with Monochloroacetic acid buffer (MCAA) purchased from vendor with 1.2 mls added to each 40ml vial, checking residual chlorine at lab. | 1.2 mls to each 40ml vial of MCAA buffer purchased premade from vendor, 1.2 mls to each 40ml vial. MCAA buffer makeup 73% wtr, 14.4% MCAA, 5.8% potassium hydroxide, 6.0% acetic acid. | Ship with 1.8 mls monochloroacetic acid buffer to 60ml vial, if residual chlorine present, add 80mg per liter of sodium thiosulfate | Sodium thiosulfate, Monochloroacetic acid, 28 days |
| 547 Glyphosate | 1 clear 40ml vials pre-preserved with sodium thiosulfate. | 1 clear 40ml Clear vial, pre-preserved from vendor with sodium thiosulfate. If inventory low, prep vials in volatiles department by adding 50ul of 10% sodium thiosulfate solution. | Weight 40 grams reagent grade sodium thiosulfate and add to 500mls of DI water. Then add 125ul of this solution to 40ml vial. | Remove residual chlorine with 100mg/L of sodium thiosulfate,cool to 4 degrees | sodium thiosulfate, 4 degrees C, 14 days , (18 months if frozen) |
| 548.1 Endothall | 1 amber 250ml bottle preserved with sodium thiosulfate | 1 250ml amber with 250ul of sodium thiosulfate solution. If 1 liter bottle used, add 1ml. | Weight 40 grams reagent grade sodium thiosulfate and add to 500mls of DI water. Then add 250ul of this solution to 250 ml bottle. | If residual chlorine present, add 80mg sodium sulfate per liter (If high bio-activity ph 1.5 - 2 with 1:1 HCL) | Sodium thiosulfate (HCL pH 1.5-2 if high bio activity) Cool, 4 degrees C, Dark, 7days |
| Drinking Water | Comments | Preservation makeup | Acid and Reagent Mix | EPA Method | FDEP FS100 |
| 549.2 Diquat | 1 high density amber PVC bottle preserved with sodium thiosulfate | 1 high density amber PVC bottle preserved with 1ml of sodium thiosulfate solution. | Weight 40 grams reagent grade sodium thiosulfate and add to 500mls of DI water. Then add 1 ml of this solution to 1 liter bottle. | amber PVC high density bottles, if known or suspected chlorine residual add 80mg/L sodium thiosulfate. If bi-active, pH <2 with H2SO4 | High density amber plastic, Sodium thiosulfate (H2SO4 to pH <2 if biologically active) Cool, 4 degrees C, Dark, 7 days |

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|------------------------------------|---|---|--|---|---|
| 552.2 Haloacetic Acids | 3 amber 40ml vials pre-preserved with Ammonium Chloride. | <u>3 amber 40ml vials pre-preserved with Ammonium Chloride, purchased from vendor.</u> | If vials not purchased premade, weight 4 grams reagent grade ammonium chloride and add to 50 mls of DI water. Then add 50ul of this solution to 40ml vial. | Add prior to shipping vials, ammonium chloride-5mg per 50mls | Ammonium chloride, Cool, 4 degrees C, 14 days, dark |
| Cyanide 8 oz Amber Glass (NaOH) | Never acidify, can release cyanide vapors. Always preserve with NaOH. | <u>one 8 oz amber (250mls) preserved with 0.2mls 5 Normal NaOH to start.</u> (Note method cites NaOH as interferent. pH to >10, use only the minimum amount necessary of NaOH) | 5 Normal made by adding 200g of NaOH pellets (solid) to 1000mls di water. Mix slowly-becomes very hot. Also can be made from 15N concentrate at 1:2 ratio. | Cool to 6 degrees, Standard Methods pH 10-12, hold time not listed. (Note method cites NaOH as interferent. pH to >10 , use minimal NaOH) | Cool to < 4 degrees C, raise pH to >12 with NaOH, add ascorbic acid, analyze within 14 days. (Note method cites NaOH as interferent. pH to >10 , use minimal NaOH) |
| Odor SM2150B | 1 liter glass amber unpreserved | <u>1 liter glass amber unpreserved</u> | N/A | Glass with Teflon lined caps, never plastic, Cool to < 4 degrees C, analyze as soon as possible | Cool to < 4 degrees C, 24 hours on DW (only 6 hours hold time for Non-potable water) |
| Lead and Copper | <u>1 liter plastic poly with 1:1 nitric.</u> Can be sent without acid. pH is checked at the bench prior to analysis. If adjustment needed, wait 24 hrs to analyze. | For each <u>1 liter</u> sample, add <u>4mls of 1:1 nitric acid.</u> Send bottle kits unpreserved if shipped by commercial carrier. pH checked at the bench prior to analysis. If adjustment needed, wait 24 hrs to analyze. | Slowly pour 500mls of concentrated nitric acid into 500mls ultra pure di water to make 1:1mix | Use 1:1 Nitric Acid and preserve to <2, good for 180 days | Use 1:1 Nitric Acid and preserve to <2, good for 180 days |
| Low Level Mercury | 2 x 40 mL VOA vials HCL for sample, 2 x 40 mL VOA vials HCL for duplicate, 2 x 40 mL VOA vials HCL and Blank Water for field blank | 2 x 40 mL VOA vials HCL for sample, 2 x 40 mL VOA vials HCL for duplicate, 2 x 40 mL VOA vials HCL and Blank Water for field blank | Use pre-preserved vendor vials. | 5 mL/L 12N HCl or 5 mL/L BrCl17 | Samples collected for the determination of trace level mercury (<100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. Analyzed within 90 days of sample collection. |
| Other General Chemistry | See Bottles and Preservations as listed in Waters/ Aqueous | | | | |
| Other Metals | See Bottles and Preservations as listed in Waters/ Aqueous | | | | |
| Gross Alpha | <u>1 1 liter plastic poly with 1:1 nitric.</u> | For each <u>1 liter</u> sample, add <u>4mls of 1:1 nitric acid.</u> | Slowly pour 500mls of concentrated nitric acid into 500mls ultra pure di water to make 1:1mix | for sub-lab | Use 1:1 Nitric Acid and preserve to <2, good for 6 months, ship on ice, refrigerate |
| Radium 226 Radium 228 | <u>1 1 liter plastic poly with 1:1 nitric.</u> | For each <u>1 liter</u> sample, add <u>4mls of 1:1 nitric acid.</u> | Slowly pour 500mls of concentrated nitric acid into 500mls ultra pure di water to make 1:1mix | for sub-lab | Use 1:1 Nitric Acid and preserve to <2, good for 6 months, ship on ice, refrigerate |

7.0 Facilities and Equipment

- 7.1 AEL consists of seven laboratories that are located in Jacksonville, Tampa, Miami (Miramar), Gainesville, Orlando (Altamonte Springs), Tallahassee, and Fort Myers. The addresses are listed on the cover page of this manual.
- 7.2 AEL Jacksonville is a full-service laboratory and is also home of the corporate headquarters. AEL Tampa and AEL Miami are also full-service laboratories. AEL Gainesville, Orlando, Tallahassee, and Fort Myers perform inorganic chemistry and microbiology testing.
- 7.3 The goal of AEL is to provide its employees with the most current technologically advanced equipment sufficient to meet or exceed all maximum contaminant limits or method detection limits, as required by the regulatory agencies, FDEP or EPA. AEL continually updates its equipment to keep up with the changes in technology and regulations.
- 7.4 The facilities are of sufficient size to meet all analytical and regulatory requirements.
- 7.5 The lab certification and scope of accreditation for each facility are maintained in the custody of the QA Officer with copies on the designated Quality Assurance (Q) drive of the AEL networked servers.
- 7.6 The attached spreadsheets making up the majority of this section, provide an inventory listing of the equipment stored in each facility, separated by the room number of each individual laboratory. All instruments are assigned identification in the Laboratory Information Management System as follows:
- 7.6.1 Site Location; J for Jacksonville, T for Tampa, A for Orlando, G for Gainesville, M for Miami, S for Tallahassee, and F for Fort Myers.
 - 7.6.2 Room Location: The room numbers are listed at the top in the following pages.
 - 7.6.3 Letter designation: Each instrument is assigned a one or two letter identifier.
 - 7.6.4 As example, the first GC/MS in Jacksonville would be assigned J7A, which corresponds to Jacksonville, room 7, instrument A.
- 7.7 Electronic records, bench sheets, and data sheets will also reference the instrument ID using the assignment conventions as listed above in section 7.6. Model and serial number can also be referenced on bench sheets but are not required. However, the physical identification on the instrument itself shall only need to consist of the letter designation only. Each room is to be identified by number on or near the entryway to the room. Lab location is self-evident.
- 7.8 Copies of the floor plans of the individual facilities are maintained current on the designated Quality Assurance (Q) drive of the AEL networked servers.

| Location | ID | Instrument Type | Instrument Make and Model |
|----------|----|-----------------|---|
| J1 | A | GC-MS | Gas Chromatograph, Shimadzu, Model GC-2010Plus, Serial # O215355 01145 |
| J1 | A | GC-MS | Mass Spectrometer, Shimadzu, Model GCMS-2010SE Serial # O205355 50370 |
| J1 | A | GC-MS | Purge and Trap Concentrator, EST Analytical Model: ENCON Evolution Serial # EV879092117 |
| J1 | A | GC-MS | Autosampler, EST Analytical Model: Centurian W/S Serial # CENTS487022117 |
| J1 | Z | GC-MS | Gas Chromatograph, Agilent, Model 6890N, SN US10533041 |
| J1 | Z | GC-MS | Mass Spectrometer, Agilent, Model 5973, SN US52440684 |
| J1 | Z | GC-MS | Purge and Trap Concentrator, EST Analytical Model: ENCON Evolution Serial # EVX1039020419 |
| J1 | Z | GC-MS | Autosampler, EST Analytical Model: Centurian W/S Serial # CENTS843011022 |
| J1 | Z | GC-MS | ULVAC Pump, SN 1747621A |
| J1 | N | GC-MS | Mass Spectrometer, Shimadzu, Model: GCMS-QP2010SE, S/N 0210953 00777 |
| J1 | N | GC-MS | Gas Chromatograph, Shimadzu, Model: GC-2010Plus, S/N 0205353 50269 |
| J1 | N | GC-MS | Purge and Trap Concentrator, EST Encon Evolution, S/N EV371080211 |
| J1 | N | GC-MS | Autosampler, EST Analytical, Model: Centurian W, S/N CentW549041016 |
| J1 | J | Purifier | Water Purifier, Barnstead Ultrapure Water System Model D7031, Serial # 703930790239 |
| J1 | P | GC-FID/PID | Agilent 5890 Series II, SN: 2750A19127 |
| J1 | P | GC-FID/PID | OI Analytical Eclipse Model 4660 SN: 0607466345P |
| J1 | P | GC-FID/PID | Autosampler, EST Analytical Model: Centurian W/S Serial #CENTS305051713 |
| J1 | H | Balance | Balance toploader max 720g, Citizen NV212 S/N: 8337466159 |
| J1 | K | Refrigerator #1 | Refrigerator S/N 6327171519060504 |
| J1 | L | Refrigerator #2 | Refrigerator S/N 6188171519050305 |
| J1 | M | Refrigerator #3 | Mini Refrigerator S/N A1710217860001802 |
| J1 | O | Freezer #1 | Freezer S/N WB54163918 |

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|-----------------|-----------|-------------------------------------|--|
| J1 | Q | Freezer #2 | Freezer S/N 6056171419030804 |
| J1 | T | Refrigerator #4 | Mini Refrigerator Model: DCR032CLBDB S/N: 5019083033130 |
| J1 | X | Headspace Anlyazer | Perkin Elmer Headspace Anlyazer HS-40 SN: 2595 |
| J1 | X | GC-FID/PID | Perkin Elmer 600 GC/FID Autosystems SN: 56004A |
| J1 | L8 | Label Maker | Brother, S/N U61041-D7G875364 |
| J1 | L9 | Label Maker | Brother, S/N U62829-K4Z836771 |
| J2 | BC | Barcode Reader | Wonenice Barcode Scanner PN WN6300 S/N 202011260786 Plug-and-Play |
| J2 | BC1 | Barcode Reader | Wonenice Barcode Scanner PN WN3300 S/N 202161279071 Plug-and-Play |
| J2 | BC2 | Barcode Reader | Wonenice Barcode Scanner PN WN6300 S/N 202111270651 Plug-and-Play |
| J2 | BC3 | Barcode Reader | Wonenice Barcode Scanner PN WN6300 S/N 202011260566 Plug-and-Play |
| J2 | I | Incubator | BOD Incubator, Thermo Precision Low Temp. Incubator S/N 101N0040 |
| J2 | J | Incubator | BOD Incubator, VWR Model TFFU20F2QWB S/N WB94081089; Thermo Model 3733A S/N 300389672 |
| J2 | O | Refrigerator | Refrigerator, Frigidare Model: MRT18DNGW2, S/N: BA03518535 |
| J2 | B | Refrigerator | Refrigerator, Danby Designer Model DAR044A4WDD-6, SN 4321013065342 |
| J2 | B3 | Balance | Balance, Mettler Toledo XS205 SN:1123181555 |
| J2 | B4 | Balance | Balance, VWR-220B2T, SN:691952 |
| J2 | H | Titration | Mettler Toledo DL50 SN 5121350103 |
| J2 | OB | Oven | Solids Oven, Curtis Matheson Equatherm S/N 10AT-10 |
| J2 | OC | Oven | Solids Oven, Curtis Matheson Equatherm S/N 10AW-9 |
| J2 | OA | Oven | Solids Oven, Curtis Matheson Equatherm S/N 10AU-8 |
| J2 | OD | Oven | Solids Oven, Curtis Matheson Equatherm S/N 10AW-6 |
| J2 | OE | Oven | Solids Oven, Lab Line Instruments Model#299-744. Serial #1093-3293 |
| J2 | OF | Oven | Solids Oven, Quincy Lab Oven, Model 120GC, S/N: G2-08947 |
| J2 | U | Spec | DR 5000 HACH Spektralphotometer UV/VIS S/N 1235577 |
| J2 | C1 | Reactor block | Hach COD Reactor Model: DRB200 S/N 21080C0427 |
| J2 | D1 | DO Meter | DO Meter, YSI 5000 S/N 090100530 w/ Probes YSI Model 5010 |
| J2 | D1P1 | DO Meter Probe | Probe information in maintenance log books (Lot 19M100051) |
| J2 | D1P2 | DO Meter Probe | Probe information in maintenance log books (Lot 20K100117) |
| J2 | E2 | Dessicator | Dessicator, Bel-Art Products, Secador Cat# 4207411116 S/N 5011 |
| J2 | FR | Flowrater | Dwyer Flowrater Model RMA-14-TMV, SN# 6823 |
| J2 | V1 | Vacuum Pump | Vacuum Pump, Barnant Model: 400-3901, S/N: C94001794 |
| J2 | S4 | Stir Plate | Sitr Plate, Corning Scholer 171 S/N 023103093856 |
| J2 | S6 | Stir Plate | Sitr Plate, Thermo Model: SP88850100, S/N: C3010012061503732 |
| J2 | H | Auto titrator | Mettler Toledo DL50 Graphix |
| J2 | W | Waterbath | Water Bath, Precision Scientific S/N 697040366 |
| J2 | MA | Moisture Analyzer | Moisture Analyzer Mettler Toledo HB43-S, SN 4.554.988/5787.600 |
| J2 | MB | Moisture Analyzer | Moisture Analyzer OHAUS MB45 SN:J2MB001 |
| J2 | MC | Moisture Analyzer | Moisture Analyzer OHAUS MB120 SN:C111331237 |
| J2 | NC | Conductivity Meter | Conductivity Meter, Thermo Orion Model 115, S/N 003782 |
| J2 | NC-P | Conductivity Meter Probe | Probe information in maintenance log books |
| J2 | PH2 | pH meter | pH meter Mettler Toledo, Model SevenEasy, S/N 1227196089 |
| J2 | PH-P2 | pH meter Probe | Probe information in maintenance log books |
| J2 | XM | StableWeigh Station | StableWeigh Manifold, 6 Place Filling Station, Environmental Express model TDS600F, lot# 59-8043 |
| J2 | XS | StableWeigh Antistatic Bar | StableWeigh Antistatic Bar/Box Mettler Toledo model EN-C SN: 180009 |
| Location | ID | Instrument Type | Instrument Make and Model |
| J2 | YH | Hood | Hood, Captair, Toxicap 1200, S/N E54522 |
| J2 | Z | Hood | Hood, Labconco 6 foot S/N Wetchem |
| J2 | CLR | Color | Nessler tubes, matched, 50 mL, tall form |
| J2 | Q | Ion Chromatograph | Metrohm model 881 Compact IC Pro, SN:03137 |
| J2 | Q | Ion Chromatograph | Auto-sampler Model 858, S/N: 02565 |
| J2 | Y | BOD Analyzer | ManTech CBOD AutoAnalyzer, Interface Module S/N MS-0E9-125, Rinse Pump 75RPM MS-0E9-147, Reagent Pump1 12ml/m S/N MS-H9-423, Reagent Pump2 12ml/m S/N MS-H9-41, Titrant Rinse Pump1 172RPM S/N MS-0F9-203, Titrant Rinse Pump2 172RPM S/N MS-0F9-202 |
| J2 | Y | BOD Analyzer | Liquid Handler, Gilson S/N 260A9N013 |
| J2 | Y | BOD Analyzer | DO Meter, YSI 5100 S/N 08a101707 w/ Probes YSI Model 5905 |
| J2 | Y-P | BOD Analyzer | Probe YSI 5095; Probe information in maintenance log books |
| J2 | Y-A | BOD Aerator | Aquaculture aerator pump |
| J2 | L | Color/Chlorine meter | HACH DR300 SN22020B000669 |
| J2 | AA | Karl Fisher | Karl Fisher AQV-300 Aquacounter S/N 9421026-03 |
| J2 | TX | TOX | EST, Trace Elemental Instruments, Xplorer SN: 2017.017 w/ titration cell SN:2017.0831 |
| J2 | TX | TOX Autosampler | TE Instruments, Tuscan V2, SN 2020.010 |
| J2 | SX | TOX-Prep | EST, sample prep chamber, Xprep-3 SN: 2017.034 |
| J2 | XX | Inhibitor Dispeser | Hach Nitrification Inhibitor Dispeser |
| J2 | R6 | Regulator | Oxygen Regulator |
| J2 | F1 | Flashpoint | Flash Point Tester, Erdco S/N 539829 (Moved to Room J10 on 04/05/2021, and back to J2 on 09/10/2021) |
| J3 | G | Mercury Analyzer | Perkin Elmer FIMS 100, PN50509550, S/N: 101521030601 |
| J3 | G | Autosampler | Autosampler S23, PN N0830010, S/N: 032101523 |
| J3 | GH | Hood | Hemco Fume Hood S/N L08-1619 |
| J3 | M | ICP-MS | ICP-MS Thermo Fisher Model ICAP Q, S/N 0722 |
| J3 | M | ICP-MS | Autosampler CETAC ASX-520 S/N 111326A520 |
| J3 | A | ICP-OES | ICP Thermo Scientific icap 7400, SN#: IC74Duo285 |
| J3 | A | ICP-OES | Cetac ASX-560 S/N: 021501A560 |
| J3 | E | Balance | Sartorius Universal - Type U6100D=**V20C S/N 39030020 |
| J3 | P | Hot Block Digester | Questron Technologies, S/N QW14040B |
| J3 | Q | Hot Block Digester | Questron Technologies, S/N QW14040C |
| J3 | W | Hot Block Digester | SCP Science DigiPrep Keypad, S/N: KPX1019304165 |
| J3 | U | Hot Block Digester | Environmental Express Hot Block/SC154, S/N 944CEC0974 |
| J3 | C1 | Digestion Block Controller | SCP Science DigiPrep Keypad S/N: KPX1019304165 |
| J3 | C2 | Digestion Wireless Block Controller | Questron Technologies Corp S/N: QW14039A |
| J3 | C3 | Digestion Block Controller | Questron Technologies Corp S/N: QW14040.1 |
| J3 | C4 | Digestion Block Controller | Questron Technologies Corp S/N: QW14140.1 |
| J3 | AD | AutoBlock Digester | Environmental Express,120/230V, Fuse 12A/6A, SN AB4001-0318-096 |
| J3 | N | Sonicator | Model 2510 Branson S/N RLA110735474E |
| J3 | R | ICP-MS | ICP-MS Thermo Fisher Model ICAPRQ, S/N: ICAPRQ02518 |
| J3 | R | ICP-MS | Autosampler CETAC Model: ASX-560; S/N: 052002A560 |
| J3 | R | ICP-MS | Chiller Thermo Fisher Model: ThermoFlex2500; S/N: 1171123101200527 |
| J3 | S | Shaker | Shaker, VWR, S/N 201933595 |
| J3 | T | Turbidimeter | Hach 2100P Turbidimeter S/N: 030300030552 |
| J3 | L6 | Label Maker | Zebra Technologies Corporation, Model LP2824, S/N 22J142000024 |
| J3 | L7 | Label Maker | Dymo Label Manager 160 |
| J3 | R7 | Regulator | Helium Regulator |
| J3 | V4 | Vacuum Pump | Vacuum Pump, Thomas model 905CA23-814A, S/N: 31001657526 |

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|-----------------|-----------|--------------------------|---|
| J3 | DPHCL2 | Dispenser Pipette | Dispensette S 1-10mL |
| J3 | DPHNO3 | Dispenser Pipette | Dispensette S 1-10mL |
| J3 | DI | DiH2O System | ELGA Model: CLXXXUVM2-US, Serial No. CLA00003480 |
| J3 | T1 | Turbidimeter | Turbidimeter, Hach Model 2100N SN: 010700007053 |
| J4 | A | Chiller (J3A) | Chiller Polyscience S/N 1709-05880 |
| J4 | M | Chiller (J3M) | Chiller Thermo Flex 2500 S/N ME04026-25 |
| J4 | R18 | Regulator | Nitrogen Regulator 2 tanks (Right (R) & Left (L)) |
| J4 | B | Nitrogen Generator | Generon, GN2 S/N MM201003 |
| J4 | B | Nitrogen Generator | IR Ingersoll Rand, Model 47672061004, S/N (M) 1/29/2020-S11483-3372 |
| J4 | B | Nitrogen Generator | Oil Free Scroll Compressor, Model SLAE05E, S/N XG5550 |
| J5 | R1 | Regulator | Air regulator |
| J5 | R2 | Regulator | Hydrogen regulator |
| J5 | R4 | Regulator | Argon regulator |
| J5 | R20 | Regulator | Helium Regulator - 2 tanks (Left (L) & Right (R)); Airgas Manifold Model # 5264071-20-001, S/N 19714502 |
| J5 | R21 | Regulator | Nitrogen Regulator - 2 tanks (Left (L) & Right (R)); Airgas Manifold Model # 5264071-20-001, S/N 19C14TV7 |
| J6 | A | Incubator | Isotemp Fisher S/N 60800235 / 650D |
| J6 | B | Waterbath | ThermoScientific, Precision Model 2862 s/n 2014896-326 |
| J6 | N | Waterbath | ThermoScientific, Precision (small) S/N 605041205 |
| J6 | S | Waterbath | Thermo Scientific, Precision Waer Bath Model # 2866, SN 202324-181 |
| J6 | C | Autoclave | Autoclave Tuttnauer Model 2540M, SN:9902128 |
| J6 | J/G share | Incubator | B/T Sure Incubator Block Fisher S/N 1041011085380 |
| J6 | D | Hot Plate | Corning PC-4200 S/N 033507291113 |
| J6 | E | Microscope | VWR VistaVision Compound Binocular Planar SN:0831287 |
| J6 | F | UV Lamp | MMO-Mug Lamp Spectroline: E-series S/N876324 |
| J6 | G | Dessicator | Dessicator Dry Keeper S/N: 6246001 |
| J6 | H | Incubator | Fisher Econotemp, Model 55D, S/N 110 |
| J6 | X | Incubator | Isotemp Fisher S/N 209N0293 / 650D |
| J6 | J | Membrane Dispenser | EZ- Filter Membrane Dispenser Millipore S/N 006774 |
| J6 | K | Colony Counter | Quebec Colony Counter S/N 11158-1 |
| J6 | V5 | Vacuum Pump | Vacuum pump GE, Model 5KH33DN16HX, S/N G8GCX |
| J6 | M | Manifold | Manifold for 6 funnels S/N 0057 |
| J6 | M | Filter Funnels | 6 Filter funnels Gelman Scientific |
| J6 | PC | Conductivity Meter | Conductivity Meter, Mettler Toledo, Model SevenMulti, S/N 123135105 |
| J6 | PC-P | Conductivity Meter Probe | Probe information in maintenance log books |
| J6 | P | pH meter | pH meter, Mettler Toledo, Model SevenMulti, S/N 123135105 |
| J6 | P-P2 | pH meter probe | Probe information in maintenance log books |
| J6 | R | Refrigerator #1 | Refrigerator S/N BA81617862 |
| J6 | Q | Refrigerator #2 | Refrigerator S/N LR734900 |
| J6 | U | UV Sterilizer | U.V. Sterilizer Millipore S/N 655995 |
| J6 | W | Quanti-Tray Sealer | Quanti-Tray Sealer PLUS, IDEXX, SN#QTP13173302808 |
| J7 | E | FID | FID Gas Chromatograph, Perkin Elmer, Clarus 500 with autosampler, Serial # 650N4032301 |
| J7 | B | FID | FID Gas Chromatograph, Perkin Elmer, Clarus 500 with autosampler, Serial # 650N407602 |
| J7 | A | FID | FID Gas Chromatograph, Perkin Elmer, Autosystem GC model 9000, Serial # 610N3051706 |
| J7 | M | FID | Dual FID Gas Chromatograph, Perkin Elmer, Clarus 500 with autosampler, Serial # 650N6042707 |
| Location | ID | Instrument Type | Instrument Make and Model |
| J7 | P | GC/MS | Gas Chromatograph, Agilent, Model 6890N (G1530N) S/N US10623036 |
| J7 | P | GC/MS | Mass Spectrometer, Agilent, Model 5973 (G2577A) S/N US52440695 |
| J7 | P | GC/MS | Injector, Agilent, Model 7683 (G2613A) S/N CN13922353 |
| J7 | P | GC/MS | Autosampler Tray, Agilent, Model 7683 (G2614A) S/N US54715576 |
| J7 | SPARE | GC-MS | Autosampler Tray, SN US63115648 with Tower, S/N US93108491 |
| J7 | L | GC/MS | Gas Chromatograph Shimadzu GC GC-2030 serial# 460-28069-15 2021-03-03 interfaced to Mass Spectrometer, Shimadzu, GCMS-QP2020 NX Serial # O21745850499 with Autosampler, Shimadzu, Model # AOC-20i Plus, Tower Serial # C12345809864 and Tray Serial #C12135818443. Pump Edwards Model A65201906 Serial #210376590. Computer GCMSInsight SW Package QP SNOS0255950233 w/ GCMSolutions v. 4.50. |
| J7 | T | GC-MS | Gas Chromatograph Shimadzu model GC-2010 Plus, Serial #10681550 interfaced to Mass Spec QP2010SE, Shimadzu, Serial # 020534850003, with Autosampler, Shimadzu, Model # AOC-20i, Serial # C11314813186SA |
| J7 | H | GC-MS | Gas Chromatograph Shimadzu model GC-2010, Serial #626455 interfaced to Mass Spec QP2010, Shimadzu, Serial # C70264000216, with Autosampler, Shimadzu, Model # AOC-20i, Serial # C11314101671SA |
| J7 | G | NPD | Dual ECD Gas Chromatograph, Perkin Elmer, Clarus 590 with autosampler, Serial # 590S21070808. Computer Dell Optiplex XE3 SN WCAPELJ94KDF3 w/TotalChrom v. 6.3.4 |
| J7 | Z | ECD | ECD Gas Chromatograph, Perkin Elmer, Clarus 500 with autosampler, Serial # 650N6051605 |
| J7 | Y | ECD | ECD Gas Chromatograph, Perkin Elmer, Clarus 500 with autosampler, Serial # 665N7020907 |
| J7 | F | NPD | Dual NPD Gas Chromatograph, Perkin Elmer, Clarus 500 with autosampler, Serial # 650N8021502 |
| J7 | J | NPD | Dual NPD Gas Chromatograph, Perkin Elmer, Clarus 590 with autosampler, Serial # 590S21071501. Computer Dell Optiplex XE3 SN WCAPEL5X6ZC3 w/TotalChrom v. 6.3.4 |
| J7 | X | ECD | ECD Gas Chromatograph, Perkin Elmer, Clarus 590 with autosampler, Serial # 590S1801037 |
| J7 | N | Refrigerator | Frigidaire Refrigerator Top.BT |
| J7 | R8 | Regulator | Helium Regulator |
| J7 | R9 | Regulator | Helium Regulator |
| J7 | R10 | Regulator | Air Regulator - 2 tanks (Left (L) & Right (R)); Manifold Model # 20668350, S/N 188125PE |
| J7 | R11 | Regulator | Hydrogen Regulator |
| J7 | R12 | Regulator | Helium Regulator |
| J7 | R13 | Regulator | P5 Regulator |
| J7 | R14 | Regulator | Helium Regulator |
| J7 | R15 | Regulator | Helium Regulator |
| J8 | DI | DiH2O System | Evoqua Water Technologies SN1859297-5 |
| J8 | DE | Shaker | 8 position Sample Shaker, Custom |
| J8 | DD | Shaker | Mid Range 3D Sample Shaker, Glas-Col, Model VS20012, Serial# 380113 |
| J8 | EE | Shaker | Glas-Col Model 099A BT1000ST, Serial# 11334691 |
| J8 | FF | Balance | Balance-Open Top Loader, Ohaus Scout SPX222, SN C103966311 |
| J8 | GA | Standards Refrigerator | Magic Chef 4.4 cubic ft Model MCRB 440S2 S/N 2700102202 |
| J8 | G | Centrifuge | Centrifuge, Damon/IEC Division, Model IEC Spinette, Serial# 49002109 |
| J8 | J | Vacuum | Vacuum Pump, GAST Manufacturing,, Model 0523-V4F-G582DX, Date Code 0894, MFG# F947 |
| J8 | T | Vacuum | Vacuum Pump, GAST Manufacturing,, Model 0211-V45F-G8CX, Date Code 0895 |
| J8 | K | Vacuum | Vacuum Pump, Marathon 0523-V4A-G588DX SN F11J20040 |
| J8 | MA | SPE Manifold | Restek, Fishbowl Manifold no serial number |
| J8 | MB | SPE Manifold | Saipurui, Fishbowl Manifold no serial number |
| J8 | MC | SPE Manifold | Saipurui, Fishbowl Manifold 200-TO-HJWJL-3763 |
| J8 | MD | SPE Manifold | Saipurui, Fishbowl Manifold no serial number |
| J8 | ME | SPE Manifold | Restek Resprep QR-12, Fishbowl Manifold no serial number |
| J8 | MF | SPE Manifold | Supelco Visiprep 12port, Fishbowl Manifold no serial number |

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|-----------------|-----------|------------------------|---|
| J8 | H | Hood | One Pointe Solutions Model N/A, Serial N/A (Custom Built Hood) |
| J8 | CO | Hood | Safeaire, Fisher Hamilton. |
| J8 | M | Hood | Lab Hood, Custom made canopy hood, Serial NA |
| J8 | N | Hood | Lab Hood, Labconco, Cat # 72861003726, Serial # 990361003 |
| J8 | P | Hood | Lab Hood, Labconco, Cat# 48801003726, Serial# 990861956 |
| J8 | 8R"SN" | Sonicator | Sonicator, Branson, Model 3510R-MT, Serial # PMA090033034E |
| J8 | 8S"SN" | Sonicator | Sonicator, VWR, Ultrasonic Cleaner 20.8L, 97043-984, SN 2119A1325 |
| J8 | O | TurboVap | TurboVap Concentrator, Zymark, Model TurboVap II, Serial # VV0312N11590 |
| J8 | V | 3 door Refrigerator | ATOSA, Model MBF8006GR, SN MBF8006GRAUS1T0321020600C40015 |
| J8 | W | TurboVap | TurboVap Concentrator, Calliper Life Sciences, Model TurboVap II, Serial # TV0511N12209 |
| J8 | X | TurboVap | TurboVap Concentrator, Calliper Life Sciences, Model TurboVap II, Serial # TV0636N13247 |
| J8 | Y | TurboVap | TurboVap Concentrator, Zymark, Model TurboVap II, Serial # TV9824N8174 |
| J8 | ME | TurboVap | TurboVap Concentrator, Zymark, Model TurboVap II, Serial # TV0846N14910 |
| J8 | Z | Waterbath | VWR Scientific Water Bath Model #1235PC S/N 1202391 |
| J8 | CC | Oven | Drying Oven, Cole Palmer Instrument Company, Model 52412-88 S/N 1A045479 |
| J8 | Q | Dishwasher | Frigidaire Model FFBD2406NW |
| J8 | VX | Vortexer | Fisher model 231, SN# 808N0371 |
| J8 | L3 | Label Printer | Zebra Tchnologies Corporation, Model LP 2824, S/N 22J142000087 |
| J8 | L4 | Label Printer | Zebra Tchnologies Corporation, Model LP 2844, S/N 64A050500901 |
| J8 | L5 | Label Printer | Brother, S/N U61041-A5J733961 |
| J8 | R17 | Regulator | Nitrogen Regulator |
| J9 | A | IR Gun | ETEK CITY Infrared Thermometer Model Lasergrip 1080; S/N US04417G0-32 |
| J9 | B | Pipet | Dispensette Pipet SN 07M 28936 |
| J9 | C | Pipet | Dispensette Pipet SN 07M 28942 |
| J9 | L1 | Label Printer | Zebra Tchnologies Corporation, Model LP 2844, S/N 42A063001966 |
| J9 | L2 | Label Printer - Fed EX | Zebra Tchnologies Corporation, Model ZP 505 S/N: 27J201400322 |
| J9 | S | Scale | Scale, Model: 4010-8B, S/N 000395 |
| J10 | AB | Balance | VWR-220B2T SN 691956 |
| J10 | SO | Oven | Quincy Lab Model 20GC, SN G2-09563 |
| J10 | B | Hood | Air Science, Model PTEFH-48, SN# PTEFH70703 |
| J10 | DP HCL | Dispenser Pipette | Dispensette S 1-10mL |
| J10 | C | TOC analyzer | OI Analytical Aurora, Model 1030, SN P044730653P |
| J10 | C | TOC analyzer | OI Analytical Solids, Option-1030S Solids 115V, Item 326917, SN21C102200 |
| J10 | C | TOC analyzer | PICG-1030W/1088/ATOC, 115V, Item 325254, SN21B104128 |
| J10 | C | TOC analyzer | Model TOC 1030W 110V, Item 322110, SN21B104056 |
| J10 | HG | Low Level Hg | Teledyne Quick Trace M-8000 S/N: US15268009 (Relocated to J2 on 04/02/2021 and back to J10 on 09/20/2021) |
| Location | ID | Instrument Type | Instrument Make and Model |
| J11 | A | LC/MS | Multisampler, Agilent, Model 1260 (G7167A), S/N DEAGX00166 |
| J11 | A | LC/MS | Binary Pump, Agilent, Model 1260 (G7112B), S/N DEAE900549 |
| J11 | A | LC/MS | Column Compartment, Agilent, Model 1260 MCT (G7116A), S/N DEAE18242 |
| J11 | A | LC/MS | QQQ (Triple Quad), Agilent, Model 6470 LC/TQ (G6470A), S/N SG1729D102 |
| J11 | A | LC/MS | Source, Agilent, Model G1958-65138, S/N SG17229039 |
| J11 | A | LC/MS | Rough Pump, Agilent, Model G1960-80040, S/N 1TZ0055079 |
| J11 | B | Balance | Electronic Balance, Ohaus, Model SPX2202, S/N B941389328 |
| J11 | C | Evaporator | N-Evap 111, Organomation, Model 5585, S/N 63234 |
| J11 | M | Evaporator | N-Evap 111, Organomation, Model 5585, S/N 63832 |
| J11 | D | Refrigerator | Mini Refrigerator, Danby, Model DAR033A6BSLDB, S/N 4319023068347 |
| J11 | E | Vortex | Miniature Vortex Mixer, Ward's Science, Model BV101-R, S/N 19091938 |
| J11 | F | Manifold | Restek Resprep QR-12, Fishbowl Manifold no serial number |
| J11 | G | Refrigerator | Refrigerator, Atosa Model MCF8705GR, S/N MCF8705GRAUS100320011000C40017 |
| J11 | H | Manifold | Restek Resprep QR-12, Fishbowl Manifold no serial number |
| J11 | DW | Manifold | Restek Resprep QR-12, Fishbowl Manifold no serial number |
| J11 | I | Hood | Air Science, Model Pur Air - P30-XT, Serial # P92943 |
| J11 | K | HPLC | HPLC with Post Column reactor consisting of nine components |
| J11 | K | HPLC | Agilent 1100 Series Quaternary Pump, Model G1311A, Serial # DE62959726 |
| J11 | K | HPLC | Agilent 1100 Series Degasser, Model G1379A, Serial # JP13212634 |
| J11 | K | HPLC | Agilent 1100 Series Autosampler, Model G1313A, Serial # DE33224082 |
| J11 | K | HPLC | Agilent 1100 Series Thermostatted Column Compartment, Model G1316A, Serial # DE33237128 |
| J11 | K | HPLC | Agilent 1100 Series Fluorescence Detector(FLD), Model G1321A, Serial # DE33205207 |
| J11 | K | HPLC | Agilent 1100 Series Diode Array Detector(DAD), Model G1315B, Serial # DE22616014 |
| J11 | K | HPLC | Mulan Laboratory Post Column Reactor, ASI Model 310-0501B, S/N: 1801 |
| J11 | K | HPLC | Post Column Reagent Pump#1, Model Series 1, Serial # Z0051898 |
| J11 | K | HPLC | Post Column Reagent Pump#2, Model Series 1, Serial # Z0425421 |
| J11 | S | HPLC | Thermo Scientific Vanquish System consisting of six components |
| J11 | S | HPLC | Column compartment (inside) - model VC-C10-A; S/N 6508913 |
| J11 | S | HPLC | Column compartment (outside) - model VC-C10-A; S/N 6508950 |
| J11 | S | HPLC | Variable wavelength detector (top) - model VC-D40-A; S/N 8328758 |
| J11 | S | HPLC | Variable wavelength detector (bottom) - model VC-D40-A; S/N 8329087 |
| J11 | S | HPLC | Dual split autosampler - model VF-A40-A; S/N 8329874 |
| J11 | S | HPLC | Dual pump - model VC-P32-A; S/N 8329722 |
| J11 | S | HPLC | Computer |
| J11 | R5 | Regulator | Nitrogen Regulator |
| J11 | R23 | Regulator | Nitrogen Regulator |
| J11 | R24 | Regulator | Nitrogen Regulator |
| J11 | V3 | Vacuum | Vacuum Pump, Welch, Model 2546B-01, S/N 071000002287 |
| J11 | L | Centrifuge | VWR Centrifuge, PN 76018-988, S/N: LC19AAG0000005 |
| J11 | O | Sonicator | VWR Ultrasonic Cleaner; Model # 97043-980; serial # 2118A2171 |
| J11 | P | Cooling Unit | Cole Parmer low temperature cooler; Model # P60N2C101B; serial # 2111-02663 |
| J12 | A | Rotator | Rotary Extractor, Lars Lande Mfg, Serial #1270 |
| J12 | B | Rotator | Environmental Express, Model LE1002, SN 2022128622025 |
| J12 | C | Hood | Air Science, Model Pur Air P5-48-XT S/N P90212 |
| J12 | E | Balance | Balance-Open Top Loader, Ohaus Scout SPX222, SN C050695155 |
| J12 | FZ | Freezer | Wood's Freezer Model C05BBA Serial # 01705046CJ |
| J12 | HP | Hot Plate | Hot Plate with stir, Thermo Scientific, Cimarec+, S/N C3010018041627041 |
| J12 | M | Refrigerator #1 | Refrigerator, GE, Model TBX18LLB, S/N TD570495 |
| J12 | K | Refrigerator #2 | Refrigerator, Frididair, Model MET18DNGW1, S/N BA03206471 |
| J12 | PH | pH meter | Fisher Accumet pH Meter 25 S/N C0000676 |
| J12 | PH-P | pH meter probe | Probe information in maintenance log books |
| J12 | T | Torque Wrench | Seekonk Precision Tools BT-2R at 48 In.lbs |
| J12 | S | Hotplate/Stirrer | Cole Parmer Multi Hotplate/Stirrer Stuart SB162-3 S/N R360002135 (Position 1, 2, 3) |
| J12 | ST | Stir Plate | 5 position stir plate, model 505C, SN: 170907050537 |
| J12 | V6 | Vacuum | Vacuum Pump, Emerson Model 5BA-4-G482X; SN 0788 |

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|-----------------|-----------|---------------------------------------|--|
| J13 | E3 | Dessicator | Dessicator, orange box. |
| J13 | G | Manifold | O&G vacuum Manifold 6 position-custom |
| J13 | H2 | Hot Plate | Hot Plate, Corning Model PC-600D S/N 013606286531 |
| J13 | N | Kiln | Cress Electric Kiln (240AC 23A), Model B-18-H, SN# 6606 |
| J13 | V2 | Vacuum Pump | Vacuum Pump, Millipore S/N 030800000525 |
| J14 | A | Upright Refrigerator | Model D55RG, 115VAC, 60Hz, 730W, 8.6A, R290/4 58oz, 3120484 DUKERS |
| J14 | B | Hood | Erlab, Model Captair 481 Smart S/N: 4481-1902 (Moved from J10 09/10/2021) |
| J15 | A | AutoShaker | Four E's Scientific Model: MI0103002 Double S Orbital and Linear Shaker Serial#: MYN000107 |
| JF | pH | Portable pH Meter - Field Sampling | Ecosense pH100A, S/N JC04132 |
| JF | pHP | Portable pH Meter - Field Sampling | YSI Environmental 211103SIA605377 |
| JF | pH2 | Portable pH Meter - Field Sampling | Ecosense pH100A, S/N JC04145 |
| JF | pHP2 | Portable pH Meter - Field Sampling | YSI Environmental |
| JF | MM | Portable Multi-Meter - Field Sampling | YSI 556 MPS Multi Probe S/N 13E101561 |
| JF | MMP | Portable Multi-Meter - Field Sampling | YSI Environmental 115562-4M-13J54, pH 005565, L/N:210712SEN005565 |
| JF | MM2 | Portable Multi-Meter - Field Sampling | YSI Pro Quatro Multi Probe S/N 21K103610 |
| JF | MM2P | Portable Multi-Meter - Field Sampling | YSI Pro Quatro S/N 21K100079 |
| JF | MM2FC | Portable Multi-Meter - Field Sampling | YSI 6850 Flow Cell S/N 12103494 |
| JF | T1 | Turbidimeter | HAC 2100Q LPG439.01.00002, SN11060C010250 |
| Location | ID | Instrument Type | Instrument Make and Model |
| G1 | GLT-1 | IR Gun | Digital Infared Thermometer Raytek Minitemp FS |
| G2 | B | Coldroom | American Cold Storage/6909 |
| G2 | C | Refrigerator | Refrigerator with freezer-Holiday LR-1 |
| G3 | A | Weight set | SN 302 - 100, 50, 10, 1, 0.1 g set |
| G3 | B | Ion Chromatograph | Metrohm 930 Compact IC Flex S/N: 1930200014153; 1858002005369 |
| G3 | B | Autosampler | Metrohm IC Autosampler Plus SN06108 |
| G3 | C | Autosampler | Autosampler QuAAtro SEAL/XY-2/4744A12633 |
| G3 | D | QUATTRO | SEAL/QuAAtro/7542206 |
| G3 | E | Weight set | SN FN3419 - 100, 50, 10, 1, 0.1, 0.001 g set |
| G3 | H | pH Meter | Mettler Toledo Five Easy F20 SNC029710281 |
| G3 | I | Probe | Mettler Toledo/LE438 |
| G3 | K | pH Meter | pH Meter Fisher/Accumet Basic/08773 |
| G3 | L | Probe | Orion 9106BNWP |
| G3 | KT | Temp Probe | Accumet |
| G3 | P | Spectrophotometer | Thermo Fisher/Aquamate 8000/2W2T327209 |
| G3 | Q | Spectrophotometer | Spectronic/Genesys 20/4001/4 (SN:3SGG285007) |
| G3 | S | Vortexer | Thomas Scientific/Fixed Speed Vortex Mixer |
| G3 | T | Stir Plate | Corning/PC131/322383 |
| G3 | U | Hot/Stir Plate | Thermo Scientific/Cimarec+ (SN:C3710011041625703) |
| G3 | V | Turbidimeter | Hach/2100N/010300006795 |
| G3 | W | Burette #1 | Alkalinity |
| G3 | X | Color Tester | Orbeco-Hellige Aquatester |
| G3 | Y | COD Digestion Block | Seal Analytical/50-place block (SN:5146U01263) |
| G3 | YY | Hot Plate | Corning PC-35 |
| G3 | ZZ | Burette #2 | Alkalinity |
| G3 | AA | Digestion Block | Hach COD Reactor SN# 971200016951 |
| G3 | AB | Incubator | CBOD/BOD Incubator Precision/815/600041515 |
| G3 | AO | Stir Plate | VWR Scientific/360/2281 |
| G3 | AP | API | Astoria Pacific A2 |
| G3 | AQ | DO Meter | YSI/5000/050022/13H 101776 |
| G3 | AR | DO Probe | YSI/5905 |
| G3 | AS | Vortexer | VWR Scientific/G-560/2-225462 |
| G3 | AT | Stir Plate | VWR 360 Stirrer (SN:2277) |
| G3 | AV | Conductivity Meter | YSI/3100/00H1395 |
| G3 | AW | Chlorine Meter | Hach/Pocket/030200030726 |
| G3 | AY | TOC analyzer | Shimadzu/TOC-VCSH/H51104435219 |
| G3 | AZ | Autosampler | TOC autosampler Shimadzu/ASI-V/H52104401932 |
| Location | ID | Instrument Type | Instrument Make and Model |
| G3 | BB | BALANCE | VWR-Model 124B2 SN#659029 |
| G3 | BC | Centrifuge | Damon IEC/ 42900893 |
| G3 | BE | Digestion Block | Cyanide Block Digester Westco/AD-40/20 Heater Base/1159 |
| G3 | BF | Controller | Cyanide Block Controller Westco/114-B400-01/1323 |
| G3 | BG | Vacuum Pump | Gast DOA-P704-AA (Solids) |
| G3 | BI | Stir Plate | VWR Scientific/205/5859 |
| G3 | BZ | Vacuum Pump | Gast DOA-P704-AA (Cyanide) |
| G3 | BO | Probe | Conductivity YSI 3252 |
| G3 | BR | digestion block | Seal Analytical/50-place block/5148U00498 |
| G3 | BS | digestion block | Seal Analytical/50-place block/5146U01264 |
| G3 | BV | waterbath | Precision |
| G3 | VM | manifold | Residue manifold |
| G3 | BV | Waterbath | Precision |
| G3 | VM | Manifold | Residue manifold |
| G4 | A | Waterbath | BlueM/MW-1130-A1/M5-17669 |
| G4 | B | Autoclave | Tuttnauer Brinkmann/2340M/9712788 |
| G4 | C | Dessicator | Sanplatec Corp/Dry Keeper |
| G4 | D | Dessicator | Sanplatec Corp/Dry Keeper |
| G4 | F | Waterbath | Precision 66566 (SN:51220035) |
| G4 | FS | Hot/Stir Plate | VWR/VMS-C4 (SN:C4/07.184059) |
| G4 | G | Refrigerator | Frigidaire/FFTR1814TW0/BA74026903 |
| G4 | H | Incubator | Gallenkamp IPR225.XX1.1 (SN:SG92/08/113) |
| G4 | J | UV Lamp | UVP, Inc./Black-Ray UVL56 |
| G4 | K | UV Sterilizer | Millipore/XX6370000 |
| G4 | L | Filter Funnel | Gelman Sciences Filter Funnel |
| G4 | M | Filter Funnel | Gelman Sciences Filter Funnel |
| G4 | N | Filter Funnel | Gelman Sciences Filter Funnel |
| G4 | P | Filter Dispenser | Millipore/EZDISP001/00899 |
| G4 | Q | Funnel Manifold | Manifold for 3 Filter Assemblies/Gelman Scientific |
| G4 | U | Vacuum Pump | Gast/0523-V191Q-G582DX/0006118657 - 4F740 (No Oil) |
| G4 | V | Colony Counter | Gallenkamp CNW 325-030Y S/N: 13 |
| G4 | W | Qtray Sealer | Quantitray Sealer Plus IDEXX, SN#QT13164401504 |
| G5 | AO | Spec Standards | Thermo Fisher Standards Kit 333150-000, SN# SA0137 |
| G5 | A | Dessicator | NL |
| G5 | B | Balance | VWR MOD: 124B2, SN: 695285 |

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| G5 | C | Dessicator | Fisher |
| G5 | K | Muffle Furnace | TableTop Furnace Company, SN: G5K20200416 |
| G5 | F | Oven | VWR/1340 |
| G5 | G | Oven | Lindberg Blue/LO-3 |
| G5 | I | Balance | Mettler Toledo/A2104/1228420311 |
| Location | ID | Instrument Type | Instrument Make and Model |
| T1 | A | Pump | Homasy Model: GD034B, SN: 20F11 (aerator for T1W) |
| T1 | B | Incubator | VWR Forced Air Incubator 2.3CF SN: 42721730 |
| T1 | C | Refrigerator | Hotpoint Model HTS16ABMFRWM S/N VF752347 |
| T1 | D | Autoclave | Market Forge Autoclave Model STM-86, S/N: 8186 |
| T1 | G | Incubator | VWR F Air 13.4 CF SN: 41892478 |
| T1 | H | Incubator | VWR CO2 Incubator, Lab World Asset# 12368, Cat# 10810-888, SN Scratched off |
| T1 | J | Manifold | Nalgene 3 - Fecal Coliform Filtering Manifold |
| T1 | K | Manifold | Nalgene 3 - Total Coliform Filtering Manifold |
| T1 | M | Incubator | Binder Incubator, BD 56-UL, SN16-14035, Asset# 8223 |
| T1 | P | Dessicator | Pyrex Brasil Round Dessicator |
| T1 | T | UV Sterilizer | Millipore UV Sterilizer, S/N: XX6370000 |
| T1 | AA | UV Lamp | Spectroline EA-160 Longwave UV Lamp SN: 1103053 |
| T1 | CC | Dessicator | Dessicator |
| T1 | AD | Refrigerator | Haier Compact Fridge Model HC46SF10SB S/N: 1403016308 |
| T1 | AJ | Incubator (35°C) | Gallenkamp Incubator 1PR225.XX1.1 S/N SG92/08/116 |
| T1 | AL | Heating Block | Thermo Multiblok Model 2050 S/N C1648110831870 |
| T1 | AM | Stir/Hot Plate | Thermo Model 88857100 S/N C3710002061634197 |
| T1 | S | Stir/Hot Plate | Corning Model PC-320 |
| T1 | AV | Waterbath | LW Scientific Model DSB-1000D S/N 1212103 |
| T1 | AN | Waterbath | LW Scientific Model WBP-20L7-HD71 S/N SBD2-21030025 |
| T1 | AO | Membrane Dispenser | EZ-Pak Millipore Model EZDISP001 S/N 001196 |
| T1 | AP | Membrane Dispenser | EZ-Pak Millipore Model EZDISP001 S/N 003099 |
| T1 | AQ | Pump (FC) | Gast Model DOA-P704-AA SN: 0717004367 |
| T1 | AR | Pump (TC) | Gast Model DOA-P704-AA S/N 1212052200 |
| T1 | AT | Dessicator | Sanplatec Dry-Keeper (Dessicator) |
| T1 | SE | Q-Tray Sealer | Quanti-Tray Sealer PLUS, IDEXX, model 89-0003936, SN: QTP13182503880. |
| T1 | W | Waterbath | Thermo Scientific Waterbath, Mod: TSGP20, SN: 300264452 |
| T2 | BU | Pipette | Wheaton Socorex 100-1000 uL S/N: 17041133 |
| T2 | A | Pipette | Eppendorf 2-20uL Pipette SN 391418A |
| T2 | B | Conductivity meter | YSI Pro 30 S/N: 21F104109 (M) 20B140039 (P) |
| T2 | C | Pipette | Thermo Scientific S/N: SU19558, Model: Finnpiptette F2 1-10mL |
| T2 | D | Settling Cone | Bel-Art Imhoff Settling Cone; 1000ml, Mod 389900000, No SN |
| T2 | E | Settling Cone | Bel-Art Imhoff Settling Cone; 1000ml, Mod 389900000, No SN |
| T2 | F | Oven | VWR Oven Gr Con 3.7CF, Mod 89511-406, SN: 42553851 |
| T2 | G | BOD/CBOD Analyzer | Seal ML V3 200M 2BOD-Prep YSI, SN: 8593 |
| T2 | G | BOD/CBOD Meters | YSI Pro Solo, SN: 200203735 & 200201873 |
| T2 | H | Spectrophotometer | Hach DR 5000 Spectrophotometer S/N: 1191482 |
| T2 | I | Pipette | Wheaton Socorex 1-10 mL S/N: 16091243 |
| T2 | J | BOD/CBOD Probe | YSI ProOBOD, SN: 20B121937 |
| T2 | K | Oven (104C) | Thomas Scientific TSOV2G S/N: 10009307 |
| T2 | L | BOD/CBOD Probe | YSI ProOBOD, SN: 20B121933 |
| T2 | M | Dessicator | Sanplatec Dry-Keeper (Dessicator) |
| T2 | N | Dessicator | Dry Keeper Sandplate Corp Dessicator for Balance Weights |
| T2 | O | Shaker | Stuart Orbital Shaker SSLI, SN RSR2021J013 |
| T2 | P | BOD/CBOD meter | HACH HQ40d Multimeter, SN: 080100016991 |
| T2 | Q | BOD/CBOD probe | LDO LBOD101, SN: 080213031424 |
| T2 | R | Pipette | Thermo Scientific Finnpiptette F1 0.5-5mL, SN: RU22403 |
| T2 | S | BOD Incubator | Thermo Scientific MOD: 3733A, SN: 300377023 |
| T2 | T | pH Probe | InLab413/IP67, SN 1460845 (backup) |
| T2 | U | Vacuum Pump | Gast Vacuum Pump Model DOA-P704-AA, S/N: 0421008765 |
| T2 | V | Vortexer | Scientific Industries, Vortex Genie 2, Model G-560 |
| T2 | W | Turbidimeter | Hach 2100N Turbidimeter, S/N: 10030C026187 |
| T2 | X | Waterbath | Precision Scientific, 265 Circulating Water Bath, SN: 696120773 |
| T2 | Y | ph/Ion Meter | Mettler Toledo Five Easy F20 8641137989 |
| T2 | Z | Hot plate | Fisher Stir-Plate/Hot-Plate, S/N: 1000019 |
| T2 | AA | Pipette | Thermo Scientific Finnpiptette F1 0.5-5mL, SN: SU19559 |
| T2 | AB | Weights | Christian Becker Calibration Weights SN: 59110 |
| T2 | AC | pH Probe | Mettler Toledo InLab 413/IP67 |
| T2 | AD | Spectrophotometer | HACH DR6000 SN 1592211 |
| T2 | AE | Stirplate | Magnetic Stirrer SH-2, SN02 |
| T2 | AF | Balance | Mettler Toledo ME104 T1100, SN C20441435 |
| T2 | AG | Centrifuge | Thermofisher Multifuge x4 Pro-MD, SN 42886157 |
| T2 | AO | SEAL | SEAL AQ2e Discrete Autoanalyzer SN: 090617 |
| T2 | AT | SEAL | SEAL Model: AQ300 Discrete Autoanalyzer SN: 031031 |
| T2 | AX | SEAL | Seal Quattro 39, SN: 8035329 |
| T2 | AZ | Waterbath | ThermoScientific Model 2845 SN: 204769 |
| T2 | CC1 | Ion Chromatograph | Metrohm 930 Compact IC Flex S/N: 1930200014153; 1858002005369 |
| T2 | EE | Titration Stand | N/A |
| T2 | II | Digestion Block | COD Reactor (Bioscience Inc.), S/N: COD-B0165 |
| T2 | LL | TOC Autoanalyzer | Shimadzu TOC-VCSH S/N: H51104335138 |
| T2 | LL | TOC Autoanalyzer | Shimadzu ASI-V S/N: 40952843 |
| T2 | MK | Hot Block | Environmental Express SC100- SN#424CEC0573 |
| T2 | EEE | Vortex | Immunotec Inc Vortex, S/N: 148-000446 |
| T2 | KK | Pipette | Wheaton Socorex 1-10 mL S/N: 13091133 |
| T2 | DU | Balance | Cole Palmer S-PA 224E, S/N: PL9Y4N86 |
| T2 | BA | Balance | Mettler Toledo AL104 Balance S/N 1228420314 |
| T2 | BB | Fluoride Probe | Cole-Parmer Electrode, Fluoride 27502-19 |
| T2 | BD | SEAL | SEAL AQ2e Discrete Autoanalyzer SN: 090615 |
| T2 | BE | Isotemp | Isotemp 220 S/N 91ONO477 |
| T2 | BL | Pipette | Wheaton Socorex 100-1000 uL S/N: 06041167 |
| T2 | BM | BOD Autoanalyzer | ManTech PC-BOD analyzer |
| T2 | BM | BOD Autoanalyzer | PC-1000-102/4 S/N: MS-0C9-553 |
| T2 | BM | BOD Autoanalyzer | PC-1000-408 S/N: MS-0L8-390 |
| T2 | BM | BOD Autoanalyzer | PC-1000-408 S/N: MS-0L8-391 |
| T2 | BM | BOD Autoanalyzer | PC-1000-416 S/N: MS-0C8-178 |
| T2 | BM | BOD Autoanalyzer | PC-1000-416 S/N: MS-0D8-181 |

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| T2 | BM | BOD Autoanalyzer | PB-10021 S/N: MS-0D8-126 |
| T2 | BM | BOD Autoanalyzer | PC-1104-00 S/N: MS-0B9-914 |
| T2 | BM | BOD Autoanalyzer | GX-271 S/N: 260J8N273 |
| T2 | BM | BOD Autoanalyzer | YSI DO Meter S5100 with probe Mantech PCE80-PH1013, Lot: 4169 |
| T2 | XX | Nitr Inhibitor Dispenser | Nitrification Inhibitor Dispenser |
| T2 | BP | Solid Sample Module | Shimadzu Solid Sample Module SSM-5000A S/N: H52504600424NK |
| T2 | BR | Refrigerator | Frigidare All Refrigerator S/N: FRU17G4JW9 |
| Location | ID | Instrument Type | Instrument Make and Model |
| T2 | BS | Shaker | Burrell Wrist Action Shaker Model 75 SN: J000259 |
| T2 | BX | Micro Distillation | Lachat Micro Distillation System S/N: 100700002080 |
| T2 | BZ | QUATTRO | Quattro S/N: 8004332 |
| T2 | BZ | QUATTRO | Quattro XY-2 Sampler S/N: 5019A15442 |
| T2 | CD | Pipette | Fisherbrand Finnpipette II 100-1000uL SN: HH87983 |
| T2 | CJ | Digestion Block | SEAL BD50 Digestion Block S/N: 5146U00666 |
| T2 | CJ | Digestion Block | SEAL BD "s" Controller S/N: 5146U00667 |
| T2 | CK | Refrigerator | Haier Refridgerator, Model-HBCN05FVS S/N: 1108000036 |
| T2 | CL | Pipette | Socorex 20-200 uL S/N: 22011118 |
| T2 | DX | Dispenser | Barnstead Labindustries Repipet III 0.5-10mL |
| T2 | CM | Pipette | Socorex 20-200 uL S/N: 22011115 |
| T2 | CN | pH/Ion meter | Fisher Scientific Accumet XL250 Dual Channel pH/Ion/Cond Meter S/N:XL94102693 |
| T2 | CO | BOD Incubator | VWR BOD Incubator Model 3733A S/N 300479670 |
| T2 | CP | Refrigerator | Frigidaire FRU17G4JW22 S/N: WA34202295 |
| T2 | CQ | Cyanide Manifold | 12 position manifold |
| T2 | CR | Cyanide Manifold | 12 position manifold |
| T2 | CS | Vacuum Pump | Gast Vacuum Pump S/N: 15006438 |
| T2 | CU | Pipette | Socorex 20-200 uL S/N: 21041098 |
| T2 | CZ | Vacuum Pump | GE 5KH33DN16HX S/N: 220290 |
| T2 | DA | Centrifuge | International Equipment Clinical Centrifuge S/N: 428-24101 |
| T2 | DB | Stir/Hot Plate | Thermo Scientific SP88857100 S/N: C3710015041500829 |
| T2 | DC | Stir/Hot Plate | Corning PC-420 S/N: 230597148652 |
| T2 | DD | Stir Plate | Corning PC-353 S/N: N/A |
| T2 | DE | Stir Plate | Hanna HI190M S/N: 1066416 |
| T2 | DF | Dessicator | Sanplatec Dry-Keeper (Dessicator) |
| T2 | DG | Balance | AE Adam CQT202 S/N: AE75314173 |
| T2 | DJ | Vacuum Pump | Gast Vacuum Pump S/N: 0616006832 |
| T2 | DK | BOD Probe | YSI 5905 BOD Probe Lot: 17A100338 |
| T2 | DM | TOC Autoanalyzer | Shimadzu TOC-V CPH S/N: H51304635160 CS, Auto sampler: Shimadzu ASI-V |
| T2 | DN | TOC Autoanalyzer | Tekmar Phoenix 8000 S/N: US01267001, Auto sampler: Tekmar S/N: 190J1359 |
| T2 | DO | BOD Incubator | Precision MFU20F3GW6, S/N: WB91702561 |
| T2 | DQ | Oven | Quincy Lab 20GC, S/N: G2-3736 |
| T2 | DT | Oven | Quincy Lab Oven, Model 40E, SN-G4E:00592 |
| T2 | DR | pH Probe | Thermo Scientific Orion 9107BNMD, Lot: VY1 exp: 8/18 |
| T2 | DS | pH meter | Thermo Scientific Orion Star A121, S/N: H 05815 |
| T2 | DU | Analytical Balance | Cole Palmer S-PA 224E, S/N: PL9Y4N111 |
| T2 | DV | Vacuum Pump | Gast Vacuum Pump S/N: 0517000679 |
| T2 | DW | Hood | Air Science PURAIR-P5-48, S/N: P80376 |
| T2 | DZ | TKN-TP Digestor | Gerhardt Model EBLs SN: 5713180088 |
| T2 | PA | Pipette | Socorex Acura 825 20uL pipette-SN 28061072 |
| T2 | PB | Pipette | Socorex Acura 825 1000uL pipette-SN 27091783 |
| T2 | PC | Pipette | Socorex Acura 825 1000uL pipette-SN 28012162 |
| T2 | PP | Pipette | |
| T2 | EA | Dessicator | Nalgene Cat #: 5317-0120 |
| T2 | SA | Vacuum Pump (Residues) | Gast Model: DOA-P704-PA, S/N: 0220313348 |
| T2 | SR | Manifold | Filter Funnel Manifold 3-place PVC |
| T2 | SW | Manifold | Stable Weigh filling station SN: 56-8025 |
| T2 | RR1 | Aerator | Aqua Culture SN: 031510 |
| T2 | RR2 | Aerator | Aqua Culture SN: 031510b |
| T2 | RR3 | Aerator | Unicliffe Aerator |
| T2 | RR4 | Aerator | Second Nature – Model: Whisper 400, SN: Jan 08 1997 |
| T3 | A | Pipette | Thermo Scientific Finnpipette F2 SN: QU39846 1-10mL |
| T3 | B | Oven | Drying Oven, Equatherm |
| T3 | C | Hood | Fisher American Model 6-31-SWNXX-XX, SN: 001670061020 |
| T3 | D | Shaker | GLAS-COL VS5502, SN: 253003 |
| T3 | F | Centrifuge | IEC Clinical |
| T3 | G | Re-pipettor | Kontes 60mL re-pipettor |
| T3 | H | Hood | Labconco Mdo 206514 SN: c2247300 |
| T3 | I | Pipette | Thermo Scientific Finpipette F1 1-10mL SN: SU16919 |
| T3 | J | Dispenser | Dispensette S by Brand Scientific Inc DE-M 15 and SN: 09N59557 |
| T3 | K | Oven | Gallenkamp Incubator 1PR225.XX1.1 S/N SG92/08/113 |
| T3 | O | Hood | Hemco Mod 31411, SN# H11-4797 |
| T3 | AA | Hood | Labconco Purifier Class II Safety Cabinet 36208-00 SN: 223243 |
| T3 | AB | Hood | Nualve Model: NU-425-600, SN: 23636 WW |
| T3 | AD | Hood | Fisher American Model 6-31-SWNXX-XX, SN: N/A |
| T3 | R | Shaker | Shaker, Thames Technologies, Inc., 4 position |
| T3 | S | Sonicator | Sonicator, Branson, Model 8510, S/N: RPA100734054F |
| T3 | T | Turbovap | Concentrator, Zymark model: TurboVap II, S/N: TV0351N12079 |
| T3 | U | Turbovap | Caliper Life Sciences, Turbo Vap II S/N: TV10846N14915 |
| T3 | V | Turbovap | Caliper Life Sciences, Turbo Vap II S/N: TV1048N16240 |
| T3 | W | Turbovap | Caliper Life Sciences, Turbo Vap II S/N: TV9835N8307 |
| T3 | W2 | Waterbath | ThermoFisher Model 180 series 2835 S/N: 295627-1153 |
| T3 | AE | Refrigerator | Atosa B Series, Model: MCF8707, S:MCF870707716091800C40013 |
| T3 | BA | Balance | Cole Palmer 12 vac 800ma SN: PL98001181 |
| T3 | AA | Dispenser | Barnstead Labindustries Repipet III 0.5-10mL Dispenser |
| T3 | AH | Refrigerator | Hotpoint Fridge HTS18GBSARWW S/N RM738789 |
| T4 | B | FID | Perkin Elmer Clarus GC, FID detector S/N: 650N3111209 |
| T4 | C | ECD | Perkin Elmer Clarus 500 GC, dual column, dual ECD, single injector, S/N: 650N8022904 |
| T4 | D | GC-MS | Shimadzu Mod: GCMS-QP2020 NX, SN: O21745850334 |
| T4 | E | FID | Perkin Elmer Clarus GC, FID detector S/N: 650509082705 |
| T4 | M | ECD | Perkin Elmer Clarus 500 GC, dual column, dual ECD, S/N: 650N5022501 |
| Location | ID | Instrument Type | Instrument Make and Model |
| T4 | G | Refrigerator | Whirlpool, cat#WH31S1E, ser # T88170909159 |
| T4 | I | FID | Perkin Elmer Clarus GC, FID detector SN 650N4032903 |

| T4 | W | GCMS | Shimadzu GCMS-QP2020NX, SN: O21745950521 |
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| T4 | X | ECD | Perkin Elmer Clarus 590 – dual ECD detector, SN: 590S21070501 |
| T5 | A | GC-MS | Shimadzu GC-2010, GCMS-QP2010 SE S/N 020535350268 US |
| T5 | A | GC-MS | Concentrator: EST SN: EVX1214072420 |
| T5 | A | GC-MS | Autosampler: EST Cent WS SN: CENTS555041018 |
| T5 | E | Refrigerator | Hotpoint Fridge Model HTS18GBSARWW S/N RM738833 |
| T5 | H | Balance | Ohaus Scout Pro Balance S/N 7130441177 |
| T5 | C | GC-MS | Shimadzu GCMS-QP2010SE S/N: 020535350270 |
| T5 | C | GC-MS | Concentrator - EST S/N: EV672051415 |
| T5 | C | GC-MS | Autosampler: EST Centurion WS S/N: CENTS208121510 |
| T5 | D | GC-MS | Shimadzu GCMS-QP2010SE S/N: O20535550377 |
| T5 | D | GC-MS | Shimadzu GC-2010m Plus -S/N 17 08 |
| T5 | D | GC-MS | Concentrator: EST SN: EV877092117 |
| T5 | L | Refrigerator | Whirlpool EST WRR56X 18FW00 S/N: U62106567 |
| T5 | K | Refrigerator | Fridgidaire LFFH20F3QWC S/N: WB61145404 |
| T5 | M | Pipette | Thermo Scientific Finnpipe F2 0.5-5 mL S/N: MH47289 |
| T5 | N | Water purifier | Thermo Scientific, Barnstead Micropur ST, SN41759414 |
| T6 | A | Rotator | Bodine Electric Company Model: DC-20 8 - place rotator No:07410072, S/N: 5685XCBA0023 |
| T6 | B | Rotator | Bodine Electric Company Rotator, S/N: 0685EPGA10106 |
| T6 | D | Rotator | 8 position Bodine Rotator, S/N: 5685SMAP0042 |
| T6 | C | Rotator | Thames Tech Rotator |
| T6 | D | Timer | Fisher Scientific Traceable Timer S/N 130133083 |
| T7 | A | Water purifier | Veolia MOD: CLXXUVM2-US, SN: CLA00003436 |
| T7 | B | Mercury analyzer | FIMS 100 Mercury analysis system from Perkin Elmer, SN:101S20090901 |
| T7 | B | Mercury autosampler | Cetac S23 Autosampler from Perkin Elmer, SN:092020S23 |
| T7 | C | Mercury Analyzer | AquaCounter HG400 & Autosampler S/N: P638022-05 - out of service |
| T7 | D | Digestion Block | Environmental Express MOD: SC154, SN: 2019CECW5264 |
| T7 | E | Pipette | Socorex 20-200 uL S/N: 17121020 |
| T7 | F | Pipette | Wheaton Socorex 0.2-2.0 mL S/N: 17121020 |
| T7 | G | ICP | Thermo Scientific, ICAP PRO SERIES, SN: iCAPPRO60094 |
| T7 | G | ICP autosampler | Teledyne ASX-560, SN: 0320142A560 |
| T7 | G | ICP Chiller | Thermo Fisher Scientific Mod: Flex 900, SN: 1122603401190515 |
| T7 | I | Digestion Block | CPI MOD Block S/N: 05-C0530 |
| T7 | H | Hood | 6' Fisher American Chemical Fume Hood, Model: 6-31 |
| T7 | J | Digestion Block | CPI MOD Block S/N: 4030311 |
| T7 | K | GFAA | Graphite Furnace AAnalyst 600, SN: 600S6010101 |
| T7 | M | Balance | Highland HCB602aM Adam Equipment Toploading Balance SN: AEA3F00045 |
| T7 | N | Pipette | Thermo Scientific Finpipette F1 1-10mL SN: SU16920 |
| T7 | PB | Pipette | Socorex 0.2-2mL Pipette MOD: Acura 835 SN: 29091058 |
| T7 | Q | Pipette | Wheaton Socorex 0.2-2.0 mL S/N: 08062293 |
| T7 | R | Pipette | Socorex 10m pipette Model 832 SN: 29071011 |
| T7 | S | GFAA | PinAAcle 900Z S/N:PZAS16030901 |
| T7 | S | GFAA Autosampler | AS900 S/N:AS9S1632302 |
| T8 | A | pH Meter | Extech Inst Palm pH PH220, SN A035745 |
| T8 | B | Probe | YSI 1001 pH/temp, SN 10126S3N60510 |
| T8 | B | Probe | YSI 1001 Cond/Temp, SN 17100682 |
| T8 | B | Probe | YSI 1001 DO Pro2003, SN 19L100575 |
| T8 | Q | Probe | YSI 556 MPS Multi Probe S/N 08F101190 (Loaner) |
| T8 | Q | Probe | YSI 5560 COND/TEMP probe S/N: 08F100094 (Loaner) |
| T8 | Q | Probe | YSI 5565 pH/ORP probe S/N: YSI556508F (Loaner) |
| T9 | B | Refrigerator | Kool It, Mod: KGM-75, S/N: KGM75170701002 |
| T9 | C | Walk-in | Walk-in Refrigerator-Iso Panel S/N: 36234 |
| T10 | A | IR Gun | Oakton TempTestr IR Infrared Thermometer Gun SN: BUKR000035935 |
| T10 | F | Dispenser | United LTD-10 (1-10mL) SN: 21202160 |
| T10 | D | Dispenser | Dispensette S 0.1-1.0 mL DE-M 19 S/N: 19H15737 |
| Location | ID | Instrument Type | Instrument Make and Model |
| A1 | A | Incubator | Total Coliform Incubator, Gallenkamp, S/N SG.92.12.036 |
| A1 | B | Waterbath | Fecal Waterbath, Blue M MW-1110A-1, S/N M5-12364 |
| A1 | C | Colony Counter | Colony Counter, Quebec 3325, S/N None |
| A1 | D | Autoclave | Autoclave, Market Forge STM-E, S/N 173491 |
| A1 | E | BT sure incubator | Model # DB104115, Serial # 1041021175479 |
| A1 | F | Millipore EZ-Pak | Millipore EZ-Pak Model: EZDISP001, Serial #: 8209 |
| A1 | G | pH Meter | HACH HQ440d multi,SN 150500000400 |
| A1 | GP | pH Meter Probe | HACH pHc201, SN 211262611344 |
| A1 | H | Refrigerator | Refrigerator, Hotpoint HT516ABMFRWW, S/N VF752407 |
| A1 | I | Dessicator | Dessicator #1 |
| A1 | J | Dessicator | Dessicator #2 |
| A1 | K | Pump | Marathon Electric Pump/Motor SN: J08J070092 |
| A1 | L | Pump | Imagitarium Powerhead Pump SN: E487688 |
| A1 | M | Millipore EZ-Pak | Millipore EZ-Pak Model: EZDISP001, Serial #: 006714 |
| A1 | N | Hot Plate | Thermo Hot/Stir plate SP88857100 S/N:C371001810652422 |
| A1 | O | Manifold | 3-place manifold, Gelman 15402 |
| A1 | Q | Quantitray Sealer | IDEXX Sealer Plus Model: 89-0003936-00, Serial #: QTP13193400207 |
| A1 | R | Manifold | 3-place manifold, Gelman 15402 |
| A1 | FF | Balance | AE106 Balance s/n 38600 067 10 |
| A1 | MM | Dessicator | Dessicator |
| A1 | P | Conductivity Meter | Mettler-Toledo SN: 1227077080 |
| A1 | PP | Conductivity Meter Probe | Mettler-Toledo Inlab 731 |
| A1 | PP | UV Lamp | Spectroline UV Lamp SM#1291640 |
| A1 | QQ | Hot Plate | Corning Stirrer/Hot Plate, Model #: PC-420, Serial #: 430506101001 |
| A1 | S | Pump | Micro Waterbath Head Power Pump no Serial #: Power Head IMAG-PH1 |
| A1 | T | Waterbath | Thermo Scientific, Model 2845, S/N - 219839-620 |
| A1 | V | Quanti-Tray Sealer Plus | IDEXX, Quanti-Tray Sealer Plus, Serial # QTP13193400207 |
| A2 | A | Stirplate | Corning Mod PC-410, SN 350401235734 |
| A2 | B | Pump | Marathon Electric Pump/Motor SN: A14J80041 |
| A2 | D | Oven | Drying Oven, Fisher Model#516G, SN 502N0070 |
| A2 | E | DO Meter | Dissolved O2 Meter, YSI 5100 SN #06G1684 AI |
| A2 | EP | DO Probe | YSI S/N 18G100113 |
| A2 | F | Incubator | CBOD/BOD Incubator |
| A2 | M | Water Bath | Precision, Series 280, S/N: 604021321 |
| A2 | H | Dessicator | Dessicator, Fisherbrand model/cat# 08-642-23B |
| A2 | G | Dessicator | Dessicator, Dry Keeper/Sanplatec, model: H42056-0001, SAN 12922546 |

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| A2 | L | Spectrophotometer | Thermo Spectronic, Mdoel Genesys 20, SN: 3SGG280010 |
| A2 | N | StableWeigh | Stable Weigh filling station SN: |
| A2 | Q | Pipette | Eppendorf Reference 100uL to 1000uL SN: 1192695 |
| A2 | S | Refrigerator | Hussmann Refrigerator- 3 door |
| A2 | T | Incubator | RCA RFR741, SN A2107405560000110 |
| A2 | X | Colorimeter | Hach Pocket II SN:1306E225105 |
| A2 | Y | Balance | AL-104 Balance Metler Toledo 1228430420 |
| A2 | C | Turbidimeter | Hach 2100N Turbidimeter, S/N: 05090C020058 |
| A2 | BB | Refrigerator | Kool-it Model #KGM-75, SN 75170701003 |
| A2 | FF | Oven | Fisher Scientific Isotemp Oven Model 625G S/N 1578071077248 |
| A2 | HH | Weights | Weight Set |
| A2 | NN | Dessicator | Dry Keeper Dessicator Sanplatec Corp |
| A2 | CLR | Nessler Tubes | Color Nessler Tubes |
| A2 | XX | Nitr Inhibitor Dispenser | Hach Nitrification Inhibitor Dispenser |
| A2 | AB | Bubbler | Bubbler/Air Pump no Serial #: AirTech-2K5 Penn-Plax |
| A2 | AC | Portable BOD Incubator | N-Con BOD-Cubator Model 00-170T Serial # T050406 |
| A3 | A | Ion Chromatograph | 930 Compact IC Flex, 19301200, SN: 1930120016146 |
| A3 | A | Ion Chromatograph autosampler | IC Autosampler Plus, 19190020, SN: 1919002006102 |
| A3 | U | Refrigerator | Frigidare HME030210N, SN: 340-91290201 |
| A3 | V | Refrigerator | GE Minifridge S/N: RF311750 |
| A3 | Z | Ion Chromatograph | Dionex ICS 1000 07120326 |
| A3 | Z-AS | Ion Chromatograph | Dionex Autosampler AS40 S/N:07120348 |
| A4 | A | Refrigerator | GE S/N: LV776914 |
| A4 | B | IR gun | Fisher Scientific Serial #: 160486988 |
| Location | ID | Instrument Type | Instrument Make and Model |
| M1 | A | Pipette | Dragon Lab Pipette (100 - 1000 uL) S/N: YE4A327532 |
| M1 | B | Digestion Block | Env. Express 54 capacity Model SC154 SN: 944CEC0976 |
| M1 | C | AA Analyzer | AA Perkin Elmer 2380, CN: 123896, MFG-Feb 1982, SN: 60056010101 |
| M1 | D | Pipette | New Cayon .1 - 1.0 mL Pipette S/N: CU0279411 |
| M1 | E | Pipette | Socorex 0.2-2.0 ml pipetter S/N: 19061047 |
| M1 | F | Pipette | Socorex 20-200 ul pipetter S/N: 18091260 |
| M1 | G | Pipette | Socorex 1-10 ml pipetter S/N: 19041055 |
| M1 | I | Autopipette dispenser | Brandtech Scientific 1-10 ml |
| M1 | J | ICP | ICP-Thermo Scientific 6500 DUO (USA) s/n IC5D20120522 |
| M1 | JA | ICP | ICP Autosampler CETAC Model: ASX-520 SN: 021609A520 |
| M1 | J2 | ICP | ICP Chiller ThermoScientific SN: N0772026/G41742 |
| M1 | J3 | ICP | Compressor Husky 30 gallon SN:H1506FWH / 21529 (Confirming what it is) |
| M1 | K | Autopipette dispenser | Dispensette Organic 1-10 ml |
| M1 | M | Digestion Block | Environmental Express, Model SC154, SN: 3703CEC1778 |
| M1 | N | Balance | Citizen Balance CT602 SN: 2B0289 |
| M1 | P | Hg Analyzer | Perkin Elmer FIMS 100, S/N 1383, Autosampler AS90 S/N: 3443 |
| M1 | Q | Pipette | Calibra 1-10mL Pipette S/N: 29051037 |
| M1 | R | Autopipette dispenser | Dispensette 1-10mL, S/N: 10561931 |
| M2 | A | Multimeter | YSI ProQuatro S/N: 21J102891 |
| M2 | A1 | Multimeter Probe | YSI Pro 10102030 Probe Lot#: 21K100079 |
| M2 | B | Field Laptop | Dell Inspiron 15, R-41000710 |
| M2 | C | Turbidimeter | Hach Turbidimeter model 2100P S/n 10040C039956 -Out of Service |
| M2 | D | Autosamplers | Isco autosamplers Model 3710 |
| M2 | E | Autosamplers | Isco autosamplers Model 3710 |
| M2 | F | pH meter | Field pH meter, mV. Temp Model pH220 Phpalm SN#018360 |
| M2 | F1 | pH probe | Extech pH probe |
| M2 | G | Colorimeter | Hach Model Pocket Colorimeter II S/N: 13090E230000 |
| M2 | H | pH Meter | OakTON pH 5T SN: 2265358 |
| M2 | I | pH Meter | YSI Ecosence pH 100A SN: JCO1805 |
| M2 | J | Multimeter | YSI ProSeries Plus SN: JCO29587 |
| M2 | J1 | Multimeter | YSI Multimeter Probe, Model #: 10102030 S/N: 17J100190 |
| M2 | K | Turbidimeter | La Motte SN: 5423-4304 |
| M2 | L | Turbidimeter | HF Scientific MicroTPW S/N: 202003510, 07/2020 |
| M2 | M | Colorimeter | Hach Pocket Colorimeter II, S/N: 05080C035428 |
| M2 | N | Rotator | Dayton model 5K939E SN C0106 |
| M2 | R | Rotator | Environmental Express Rotator |
| M2 | O | Freezer | Thermo Electron Freezer, Model: ULT1050-9-A32, S/N: P20R-229469-PR |
| M2 | P | Probe | YSI pH/Temp Probe S/N: 2007295IA605377 |
| M2 | Q | Probe | YSI pH/Temp Probe S/N: YSI605377181 |
| M2 | S | Pump | GeoPump S/N: 5466 / K18004851 |
| M2 | T | Pump | GeoPump S/N: 2346 : GeoPump Model: 900-1280 S/N: J12002659 |
| M2 | U | Water Leveler | Solinst Water Level Model 101 S/N: 57409 |
| M2 | V | Water Leveler | Geotech Water Level Meter S/N: 6148 |
| M2 | W | Field Kit: Alkalinty | Alkalinity DRT Field Kit P/N: 3467-01, Lot #: 348821 |
| M2 | X | Turbidimeter | Geotech Portable Turbidimeter WL S/N: 22013767 |
| M3 | A | IR Gun | Klein Tools IR5 S/N: None IR Gun 1 |
| M3 | B | Refrigerator | Intertek T-Series Model: MBF8006 S/N: MBF8006AUS100319012500C40004 |
| M3 | C | Shipping Scale | UPS Shipping Scale S/N: |
| M3 | D | Moisture Analyzer | OHAUS Moisture Analyzer S/N: B811543044 |
| M3 | E | Label Printer | Zebra Labeler Model: LP2844 S/N: 42A063001992 |
| M3 | F | Autopipette dispenser | BrandTech Scientific Dispensette pipetter 0.5-5.0 ml |
| M3 | FA | Autopipette dispenser | VWR Bottle Top Dispenser, 1-10mL, S/N: 21200347 |
| M3 | FB | Autopipette dispenser | VWR Bottle Top Dispenser, 0.5 - 5mL, S/N: 20402207 |
| M3 | FC | Autopipette dispenser | VWR Bottle Top Dispenser, 0.25 - 2.5mL, S/N: 20402187 |
| M3 | G | Pipette | Socorex Calibra 832 1 - 10mL, S/N: 29071095 |
| M3 | H | Refrigerator | Atosa Model: MBF8006GR, SN: MBF8006GRAUS1T0320110500C40020 |
| M3 | K | Refrigerator | Tor Rey Sliding Glass Doors Model VRD28-ULH S/N A14-000877 |
| M3 | P | Refrigerator | Silver superior Model T-49 SN: 1388130 |
| M3 | Q | Refrigerator | SABA Model S-72RG S/N: 6187171519050302 |
| M3 | R | Refrigerator | Intertek Bttm Mount Reach-ins Vertical Cooler, Model: MBF8508, S/N MBF8508AUS100319102300C40005 |
| M3 | T | Refrigerator | RCA small S/N: A1704197490000745 - Out of Service |
| M4 | A | GC-MS | Shimadzu GCMS QP2020NX S/N: 021745850327 |
| M4 | A | GC-MS | AOC-20s Autosampler, S/N:C1213581760 |
| M4 | A | GC-MS | AOC-20i Injector, S/N: C12345706476 |

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| M4 | A | GC-MS | Pump: Edwards RV3 S/N: 200286317 |
| M4 | B | ECD | GC-Dual ECD, Perkin Elmer model Clarus 500 SN:650S-10020401 |
| M4 | B | ECD | ECD Autosampler |
| M4 | C | FID | GC/FID, Perkin Elmer model Clarus 500, SN: 650S-10020303 |
| M4 | C | FID | FID Autosampler |
| M4 | I | FID | GC/FID, Perkin Elmer model Clarus 580, SN: 580S-12051001 |
| M4 | I | FID | FID Autosampler |
| M4 | E | GC-MS | Gas Chromatograph, Shimadzu, Model GC17A with Mass Spectrometer, Shimadzu, |
| M4 | E | GC-MS | Model GCMS-QP52010SE, Serial # C020534950006 |
| M4 | E | GC-MS | Autosampler AOC-20i, SN: C11314814728SA |
| M4 | E | GC-MS | Edwards rough pump, SN: 119470268 |
| M4 | F | Refrigerator | Refrigerator & Freezer Kenmore Model 253.6882015 S/N BA3702790 |
| M4 | G | ECD | Perkin Elmer Clarus 590 S/N: 590S19090206, with ECD Autosampler |
| M4 | L | Refrigerator | Refrigerator and Freezer, Daewoo Electronics Model FRG2120BRW SN: 57201767GL |
| Location | ID | Instrument Type | Instrument Make and Model |
| M5 | A | Digestion Block | Block Digestor for Lachat BD-46 |
| M5 | B | Pipette | Socorex Calibra 832 1 - 10mL S/N: 20121079 |
| M5 | C | Sonicator | Digital Pro Ultrasonic Cleaner, JPS-100A 11/2020 |
| M5 | D | Hotplate | Presto P/N: 4014-007-2-2, S/N: 46171US2; #: 0705305 |
| M5 | E | Imhoff Cone | Cone B |
| M5 | G | Turbovap | Zymark Turbovap SN# 1048N16239 |
| M5 | H | Turbovap | Zymark Turbovap SN# 1048N16238 |
| M5 | I | Turbovap | Zymark Turbovap SN# TV9933N9050 |
| M5 | J | Turbovap | Zymark Turbovap SN# TV9835N8307 |
| M5 | K | Drying Bath | Thermo Sci Dying Bath Stdrd 4blck 100-120V P/N: 88870003 S/N: K5BT70003022 |
| M5 | L | Shaker | 3D Shaker Glas-Col S/N: 380497 |
| M5 | M | Shaker | 3D Shaker Glas-Col S/N: VH2000S |
| M5 | N | Pump | Gast Model: DOA-P704-PA, S/N: 0820301879 |
| M5 | O | Balance | Balance VeriTas Model S622 SN: CH1601999 |
| M5 | P | Varispenser | Volumetric dispenser, Varispenser Plus, eppendorf, 03M73651 |
| M5 | Q | Hotplate | Bella Model: 14648 S/N: 1948 |
| M5 | R | Digestion Block | Gerhardt Digestion Block |
| M5 | S | Centrifuge | LW Scientific, Model: Ultra-8V, SN: V127178 |
| M5 | T | Pipette | Despenette pipette 1-10mL, S/N: 19C 39617 |
| M5 | U | Digestion Block | Mercury Digestion Block, S/N: 424VAR200A |
| M5 | V | Pipette | Dipensette S Pipette 1-10mL, S/N: 19D 50714 |
| M5 | W | Sieve | USA Standard Test Sieve, 0.375inches (9.5mm), S/N: 194720563 |
| M5 | X | Autopipette dispenser | Dispensette S DE-M, 110ml S/N: 17G82269 |
| M5 | Y | Autopipette dispenser | Dispensette S DE-M, 110ml S/N: 18d11854 |
| M5 | Z | Pump | Gast |
| M6 | A | Stirrer | Thermo SP88857100 S/N: C3710015041500846 |
| M6 | AB | Color Tester | Orbeco-Hellige Aqua Tester (color) |
| M6 | AB-A | Color Wheel | Orbeco-Hellige Aqua Tester color wheel 611-11 |
| M6 | AG | Turbidimeter | Hach 2100N Turbidimeter, SN: 960600002574 |
| M6 | B | SpecAnalyzer | Seal QuAAtro39, SN: 8022310 w/ Autosampler Model: XY2 Sampler SN: 5506A36885, Lachat BD40 |
| M6 | CA | DO Meter | DO meter, YSI 5000, S/N 16D105050 |
| M6 | CA1 | Probe | Probe model 5010 lot# 09E100939 |
| M6 | CE | Pipette | 20-200 uL Calibra digital 822.0200, S/N: 27061059 |
| M6 | D | Balance | Analytical Balance, Cole Palmer Model S-PA 224I, S/N: PL9YCN235 |
| M6 | E | Weights | Calibration weight set, Troemner, SN: 82987 |
| M6 | F | Incubator | Incubator (BOD) Kenmore fridge w/ Goldline Setpoint Temp Control Model SP-332 |
| M6 | G | Stirrer | VWR Stirrer Student C3 120V, Model: 986968, SN: 100816016 |
| M6 | H | Conductivity Merter | VWR Traceable Multimeter (Conductivity) SN: 200489949, In-service: 01/11/2021 |
| M6 | H1 | Multimeter Probe | VWR Multimeter Probe S/N: 200489949 P/N: 23226-505, In-Service: 01/11/2021 |
| M6 | I | OB Valorimeter | PARR Oxygen Bomb Calorimeter, Model: 1341EB, SN: 5760 |
| M6 | J | Hot Plate | Nuova Hot plate/stirrer, Model: SP18425, SN: 757001203535 |
| M6 | K2 | Digestion Block | COD Block digestor Hach Model: DRB200 S/N: 17020C0346 |
| M6 | L | Spectrophotometer | Hach DR 2010 Spectrophotometer S/N 000600018246 |
| M6 | M | IR Gun | Klein Tools IR1 S/N: None, IR Gun 3 |
| M6 | MA | Refrigerator | Hotpoint Refrigerator and Freezer model HTS18GBSFRWW, S/N ZS7624 55 |
| M6 | MH | Moisture Analyzer | |
| M6 | N | Chlorine Meter | Hach Model Pocket Colorimeter II SN:0E167529 Cat#59530-00, P/N 5953000 |
| M6 | O | Pump | Gast model 0523-101Q Grainger Vacuum Pump |
| M6 | P | ph meter | Thomas Scientific, Orion Star A211 Model#: STARA2110 S/N:X60902 |
| M6 | P2 | ph meter probe | Thomas Scientific, SN: YY1-19973 |
| M6 | P1 | ph meter probe | Thomas Scientific, Triode Refillable pH Ag/AgCl Orion 9157 BNMD, S/N: SPI-13055 |
| M6 | PA | Pipette | Socorex micro Pipette 20-200 uL S/N: 18091153 |
| M6 | PB | Pipette | Socorex micro Pipette 0.2-2.0 ml S/N: 19061050 |
| M6 | PD | Pipette | Socorex Calibra Digital 832 1 -10mL S/N: 29071062 |
| M6 | PG | Pipette | Dragon Lab Pipette (100-1000 uL) S/N: YE4A285240 |
| M6 | PH | Pipette | Socorex 10mL Macro Calibra, S/N: 29011026 |
| M6 | PK | Pipette | Socorex Calibra Digital 832 0.2 - 2.0 mL S/N: 29071103 |
| M6 | PL | Pipette | Calibra 1-10mL Pipette, S/N: 29051007 |
| M6 | PM | Pipette | New Cayon .1 - 1.0 mL Pipette S/N: CU0279498 |
| M6 | PN | Pipette | Socorex Macro Calibra Digital 832 Pipette 0.2-2.0 ml S/N: 31021015 |
| M6 | PO | Pipette | Socorex Macro Calibra Digital 832 Pipette 0.2-2.0 ml S/N: 31021014 |
| M6 | Q | Aerator Pump | Tetra Aeration Pump, S/N: 01L31, In-service: 01/2013 |
| M6 | R | Autopipette dispenser | Technologies Jade bottle top Dispenser, 5-60ml S/N: 18205731 |
| M6 | S | Autopipette dispenser | VWR Labmax bottle top dispenser 2.5 - 25 mL S/N: 040119096 |
| M6 | T | Hotplate | Bellas Model: 14648 S/N: 1922 |
| M6 | U2 | Ion Chromatograph | Metrohm Compact IC pro, Model: 1881, SN: 188100008134 |
| M6 | U2A | Ion Chromatograph | Autosampler Profesional sample processor model 858 |
| M6 | V | Manifold | Vaccum manifold with funnels |
| M6 | W | Colorimeter | Hach Model Pocket Colorimeter II S/N: 17050E329512 |
| M6 | X | Multimeter | WTW Multi 340i Multimeter, Serial # 02300046 |
| M6 | XX | Nitr Inhibitor Dispenser | Nitrification Inhibitor Dispenser |
| M6 | Y | Stirrer | Benchmark Model: H4000-S S/N: 20140926020 |

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| M6 | Z | StableWeigh | Environmental Express, StableWeigh TDS600F, Lot # 66-8080 |
| M7 | A | Oven | Model: LAC1-67-2, S/N: 139724 |
| M7 | B | Balance | Analytical Balance, Cole Palmer Model S-PA 224I, S/N: PL9YCN213 |
| M7 | G | Muffle Furnace | Paragon model E-14A SN# 346549 |
| M7 | J | Dessicator | Desicator with Hygrometer Sanplitec Corp. Dry keeper |
| M7 | N | Oven | Oven, Shel Lab model SGO4E, SN: 10005614 |
| M7 | P | Oven | Barnstead Thermolyne Mechanical Convection Oven Model OV4730-26 SN: 1297040691175 |
| M7 | T | Dessicator | Sanplitec Corporation Desiccator, Dry keeper hygrometer, Serial # N/A |
| M8 | A | Incubator | Gallenkamp Cat# 1PR225.XX1.1 S/N: 9092/08/123 |
| M8 | B | Waterbath | VWR model 1285PC, for fecal coliforms S/N:0401007 |
| M8 | C | Refrigerator | Kenmore 253-68802013 SN WA0380 1313 |
| M8 | D | UV Lamp | Long Wave-Spectroline Model EA160 |
| M8 | E | Pump | Vacuum pump, GAST Model: G588DX S/N: 108J190224 |
| M8 | F2 | Autoclave | |
| M8 | G | Manifold | Vacuum manifold with funnels GM1, GM2, GM3 |
| M8 | H | Microscope | Leica CME, model# 149521X, SN: 0712229 |
| M8 | I | BT sure incubator | Thermo Scientific, Model 2050, S/N: 1648100474393 |
| M8 | J | Desiccator | Dessicator Boeckel for agars and medias |
| M8 | K | Desiccator | Dessicator Glass round with glass lid |
| M8 | L | Incubator | Quincy Labs, Inc. Model#: 10-140, Serial #: L-10578 |
| M8 | M | Incubator | Isotemp model 525D, SN: 1569070975781 |
| M8 | O | Colony Counter | Quebec M8O Cambridge Instruments Model 9925, S/N 10006-8 |
| M8 | P | Waterbath | Fisher Scientific, ISOTEMP 228,SERIAL: 808N0202 |
| M8 | Q | Filter Dispenser | Filter Dispenser ECDSPO01, S/N: N006707 |
| M8 | R | Sealer | IDEXX Quanti-Tray Sealer Plus, P/N: 89--0003936-00, S/N: QTP13191405145 |
| M8 | S | Stirplate | Thermo Model 8P88857100 S/N: C3710023032004481 |
| M8 | T | Dessicator | Dessicator Boeckel for agars and medias |
| M9 | A | GC-MS | Gas Chromatograph, Shimadzu, Model GC17A SN: 02054950037 |
| M9 | A | GC-MS | Mass Spectrometer, Shimadzu, Model GCMS SN: 02054950037 |
| M9 | AC | GC-MS | Purge & Trap Concentrator, EST Evolution Model#: EV848051217 SN: 416081970617 |
| M9 | AP | GC-MS | Edwards rough pump Model code RV3, SN 996339389 |
| M9 | B | GC-MS | Shimadzu, Model 2010SE SN: 0205535-50275 (2nd S/N: 021095300725) |
| M9 | BC | GC-MS | EVO Concentrator: Model#: EVX 1215072420 S/N: 416083715895 |
| M9 | C | GC-MS | Shimadzu GCMS: QP2010SE Serial#: 0205355-50369 |
| M9 | CC | GC-MS | EST Evolution Concentrator Model: EV880092117 S/N: 416081970735 |
| M9 | D | Autosampler | EST Ceturion Autosamper: Serial#: CENTS720072420 |
| M9 | E | Water Purifier | ELGA Model: CLXXVUN2-US S/N: CLAO0003443 |
| M9 | F | Balance | Citizen Balance CZ-1002 SN: 5217073345 |
| M9 | G | Rotator | Bodine Electric Company Rotator, S/N: 5685WGAG0010 / 42R6BFCI-FX3 |
| M9 | H | Refrigerator | Kenmore Refrigerator and Freezer, Model 253.68802015 SN BA13703626 |
| M9 | I | Autosampler | EST Centurion Autosampler SN: CENTW814081121 |
| M9 | J | Refrigerator | Frigidaire Freezer Model FF714F5HWF SN WB94057171 |
| M9 | S1 | Syringe | Trajan SGE Syringe - 5mL - P/N: 008770 - Batch#: 49v-402366A(Cat#: 24888) |
| M9 | S2 | Syringe | Hamilton 1710RNR Syringe - 100uL - Cat#: 21263 - Lot#: 1023578(P/N:1023578) |
| M9 | S3 | Syringe | Hamilton 1702RNR Syringe - 25uL - Cat#: 21261 - Lot#: 1018846(P/N:80265) |
| M9 | S4 | Syringe | Hamilton 75N Syringe - 5uL - Cat#: 24938 - Lot#:1018146 (P/N:87900) |
| M9 | S5 | Syringe | Hamilton 7101KH Syringe - 1uL - Cat#: 24549 - Lot#: 1000973 (P/N:86211) |
| M9 | AA | Refrigerator | Kenmore Refrigerator Model 253.60722009 SN WA03801313 |
| Location | ID | Instrument Type | Instrument Make and Model |
| S1 | A | Oven | VWR Model 1305U SN:0303897 |
| S1 | B | Oven | VWR Gravity Oven S/N:42557034 |
| S1 | C | Balance | Mettler Toledo Balance, Model AL 104, SN: 1228280442 |
| S1 | E | Oven | Drying Oven-Desiccator, SN: TLH1006 |
| S1 | F | Manifold | Filtration Manifold for 3 (gray), SN: TLH1001 |
| S1 | G | Conductivity | Oakton conductivity meter SN:137742 |
| S1 | GP | Conductivity Meter Probe | Oakton conductivity meter probe |
| S1 | J | Pump | WW Grainger series oil-less vacuum pump, Model 4F740A, L10J060255 |
| S1 | L | Hach Kit | Hach Chlorine Test Kit, Model CN-66F, Lot# A3058B |
| S1 | M | Colorimeter | Orbeco Hellige Nesslerizer, Model 711-A-1, P/N L322240 |
| S1 | M1 | Color Wheel | Orbeco Hellige color wheel 0-30 SN: L284150 |
| S1 | M2 | Color Wheel | Orbeco Hellige color wheel 30-70 SN: L284160 |
| S1 | N | DO Meter | YSI Dissolved Oxygen Meter, Model YSI52CE, SN: 06L1606 AB |
| S1 | NA | DO Probe | BOD Probe-Self Stirring, Model 5905, Lot# 11A100739 |
| S1 | NP | Aerator Pump | Aqua Culture SN# 111510 |
| S1 | XX | Inhibitor Dispeser | Hach Nitrification Inhibitor Dispeser |
| S1 | R | Ion Chromatograph | Ion Chromatograph Thermo Scientific, Dionex Aquion, P/N 22176-6002, SN190220071 |
| S1 | S | Turbidimeter | Hach 2100N SN: 11080C027766 |
| S1 | T | pH meter | Mettler Toledo Model: SevenEasy, SN: 1228225041 |
| S1 | TP | pH meter probe | Mettler Toledo Model: SevenEasy |
| S1 | V | Thermometer (IR Gun) | Ryobi Tech IR Thermometer |
| S1 | X | Hot Plate | Corning, PC-320 SN 440898 |
| S1 | U | Freezer | Haier Freezer SN:BFOEWOEO300BTABF1331 |
| S2 | Y | Hot Plate | Corning. PC-620 S/N: 230898123995 |
| S1 | ZA | Colorimeter | Hach Pocket Colorimeter II SN 12040E196573 |
| S1 | B1 | Refrigerator | Kenmore Refrigerator SN: 653.60722012 |
| S1 | B2 | Incubator | Precision 815 low temperature incubator |
| S1 | XX | Nitrification Bottle | Nitrification Bottle |
| S1 | B3 | Hot Plate/Stir Bar | Fisher Isotemp SN C3720022061814272 |
| S2 | B | Incubator | Fisher Scientific Model 637D, SN: 70800141 |
| S2 | C | Manifold | Filtration Manifold for 3 (white), SN: TLH1001 |
| S2 | F | Pump | WW Grainger series oil-less vacuum pump, Model 4F740A, L10J060253 |
| S2 | GR | Refrigerator | Kenmore Refrigerator (2.5 CuFt), Model 93382, BK12106208931291 |
| S2 | J | UV Lamp | UV Lamp, Blak-Ray Model UVL-56, cert # 1010.1-Oc |
| S2 | K | Autoclave | Market Forge 10-6226-P |
| S2 | L | Waterbath | Thermo-Fisher waterbath precision Model 283 SM: 265160-2288 |
| S2 | M | Incubator | Thermo Fisher Multiblok incubator |
| S3 | A | Refrigerator | Fridgidare SN BA00808702 |
| Location | ID | Instrument Type | Instrument Make and Model |
| F1 | A | IR gun | Cen-Tech Class II SN: 364381506 |
| F1 | B | 3 door Fridge | Saba Air ST-72RG SN: 66391715-17010601 |

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| F1 | C | 3 door Fridge | Saba Air ST-72RG SN: 66391715-17010604 |
| F1 | D | Ice Chest | Fridgidare FFFC09M1RW 1D54157671 |
| F1 | E | Fridge | Superior MT1B SN: 2503845 |
| F1 | F | Bottle-Top Dispenser | MICROLIT Bottle Top Dispenser Model #SCI-5 S/N: 20407557 |
| F2 | C | Autoclave | Omni-Clave Model OCR-50 SN: A4-36174 |
| F2 | D | Incubator | Curtin Matheson Scientific |
| F2 | E | Filter Manifold | Gelman Science 15402 |
| F2 | H | UV Lamp | Spectroline EA-160 SN: 1108151 |
| F2 | I | UV Lamp | Black-Ray Lamp Model UVL-56 SN: 696 |
| F2 | J | Water Bath | Fisher 20liter Water Bath SN: 912991 |
| F2 | K | Stirrer Hotplate | Thomas Scientific HOT/STIR 120V SN: 170-315003 |
| F2 | L | Water Bath | Thermo Scientific Precision Water Bath, Model 2864, S/N: 176654-2036 |
| F2 | M | BT Sure Incubator | BT Sure Incubator 140 S/N ET17021083 |
| F2 | N | Refrigerator (small) | IGLOO Model FR326M-C-Black, S/N A1709220820000664 |
| F2 | P | Autoclave | Palton & Crane Model OCR SN: A4-36174 |
| F2 | R | Incubator | Fisher Scientific 550D Isotemp Incubator |
| F2 | Q | Quanti-Tray Sealer | IDEXX Quanti-Tray Sealer Mod 2X, Model: 89-10894-00, SN: 01311 |
| F2 | S | Dispensor | Millipore Filter Dispensor |
| F2 | T | Microscope | Digital Microscope DM4 |
| F2 | U | Balance | Fristaden Lab Model: JNB10002 S/N:JNB10002.060820 |
| F3 | C | Filter Manifold | 3 place manifold |
| F3 | D | Dessicator | Fisher Scientific |
| F3 | E | Multimeter | Hach Model HQ40d SN: 0801000-16991 |
| F3 | F | 105C Oven | Curtin Matheson Scientific, Inc Model 4001/4 SN: 10AT-6 |
| F3 | G | 180C Oven | Curtin Matheson Scientific, Inc Model D1578 SN: 10AW-3 |
| F3 | I | pH Meter | YSI Model Tru Lab pH 1110 SN: 15310110 |
| F3 | IP | pH Probe | YSI Model Tru Lab pH 1110 SN: 1705 |
| F3 | J | Conductivity Meter | Hach Model sensION 7 S/N: 09070C170005 |
| F3 | JP2 | Conductivity Probe | Hach Model 51975-00 CND SN: 9259004 |
| F3 | JP3 | Conductivity Probe | Hach Model 51975-00 CND SN: 0167176 |
| F3 | K | Turbidity Meter | Orbeco-Heilige model 966 SN: 2417 |
| F3 | XX | Nitr Inhibitor Dispenser | Hach Nitrification Inhibitor Dispenser |
| F3 | L | Pocket Colorimeter II | HACH Model HAH-14060501 SN: 17030E322394 |
| F3 | M | CBOD/BOD Incubator | VWR Cat #: 10753-894, S/N: 300383039 |
| F3 | N | Large Dessicator | Scenceware Model H42056-001 SN: 12922512 |
| F3 | P | Stir Plate | BigWave IKA ColorSquid SN 03.001898 |
| F3 | Q | CBOD aerator pump | IMAG-AP1 Aquarium Air Pump |
| F3 | R | TDS Station | TDS Station-Environmental Express Stableweigh Filling Station-Model TDS600F |
| F3 | S | Digital Thermometer | Spere-Scientific Model 800011 S/N: 0510-22103 |
| F3 | T | Desicator | DryKeeper Sanplate |
| F3 | U | Turbidity Meter | Hach 2100N Turbidimeter s/n 020400007507 |
| F3 | V | Turbidity Meter | Field Turbidity Meter, HF Scientific, Model Micro TPW, S/N 202003438 |
| F3 | X | Water Bottle Sampler | Wildco Water Bottle Sampler, Model: 1920-G65, S/N 3020 286 |
| F4 | A | DO Meter | Meter-YSI Model 5010-115 SN: 17E10157 |
| F4 | B | Spectrophotometer | Thermo Spectronic Model Genesys 20 SN: 3SGK093011 |
| F4 | C & C1 | pH Meter/probe | Oakton pH/mV/C meter SN: EP100/12524 |
| F4 | F | Pipetter | Eppendorf Reference 2, 100-1,000 uL, S/N I11837G |
| F4 | G | Ion Chromatograph | Metrohm 861 Advanced Compact IC, S/N : 1761002013160 |
| F4 | H | Pipetter | Scorex 1-10mL pipetter s/n 27031023 |
| F4 | HB | Pipetter | Socorex (Acura 835) 1-20mL Pipettor S/N: 29061078 Recvd: 07/16/2020 |
| F4 | K | Water Bath | VWR Model 211 SN: 102204 |
| F4 | M | Stir Plate | Corning Model PC-310 SN:03038068 |
| F4 | N | Burette | Nalgene 50ml |
| F4 | P | Waterbath | Waterbath-Scientific Products-Model 13200, SN0973-Room F4 |
| F4 | Q | Hot-block | Hot Block-Therolyne Cimarecl-Model HP46515, SN1067990220243-Room 4 |
| F4 | R | BOD Probe | BOD Probe-YSI 5010 BOD-lot 19E100194-Room 4 |
| F4 | S | Water Filter | Barnstead Nanopure, Water Filter Model D11931, S/N 1193020210419 |

8.0 Calibration, Verification, and Maintenance

See also ADMIN SOP-035 Ethics and Data Integrity.

See also ADMIN SOP-038 Calibration, Manual Integration, and Rules for Chromatography.

See also TECH SOP-009 Multi-Peak Compound Identification for Organics.

See also TECH SOP-010 Establishing and Maintaining Retention Time Windows.

See also Section 5.0 of this Quality Manual for discussion of traceability and calibration of support equipment, such as thermometers, weights, balances, and glassware.

8.1 Calibration

- 8.1.1 Sections 8.1 through 8.4 specify the essential elements that shall define the procedures and documentation for initial instrument calibration. See sections 8.3 and 8.4 for rules governing proper calibration. See the individual analytical SOPs for procedural details for calibrating each test method. If there are more stringent standards listed in the test method or regulatory requirements than those listed here, than those more stringent standards shall be used.
- 8.1.2 All calibrations are performed according to the analytical method specifications.
- 8.1.3 The individual analytical SOPs, referenced in Section 3.0 of this Manual, explain in detail how the calibrations are to be performed.
- 8.1.4 The methods that require calibration are listed in Section 14.0 as part of the list of analytical methods performed by AEL.
 - 8.1.4.1 This section details the number of calibration points used to perform the calculation.
 - 8.1.4.2 The frequency of the calibration is also listed.
- 8.1.5 Calibrations are performed to verify the instrument is reading known standards accurately.
- 8.1.6 All calibrations are performed using standards that are traceable to a known standard via the Certificate of Analysis, as described in Section 5.0.
- 8.1.7 The acceptance criteria for calibrations are defined in the analytical SOP (referenced in Section 3.0), but generally, a correlation coefficient of 0.990 or greater is required for all tests requiring this to be calculated.

8.2 Verifications

- 8.2.1 All calibrations are verified before analyzing any samples.
- 8.2.2 The verification procedures and standards are detailed in the analytical SOPs referenced in Section 3.0.
- 8.2.3 All initial calibration verifications will be performed by a second source standard, meeting one of the requirements listed:
 - 8.2.3.1 The standard is from a different manufacturer than that used to make the calibration.
 - 8.2.3.2 If no second source vendor is available, then the standard is a different lot # from the same manufacturer, but not of the same standard mix as used in the calibration.
- 8.2.4 The acceptance criterion for calibration verification is documented in the analytical SOPs (referenced in Section 3.0).
- 8.2.5 Some methods require calibration verification at a set interval, such as every 12 hours or after every 10 samples. This frequency is defined in the analytical SOPs as well.
- 8.2.6 There are two types of calibration verifications performed:
 - 8.2.6.1 Relative error calculation as specified in Section 8.3.6 and Initial calibration verification (ICV), which is the second source standard that must be run after every new calibration and must meet its acceptance criteria before any samples can begin analysis.
 - 8.2.6.2 Continuing Calibration Verification (CCV), which is the standard that is analyzed at the method defined interval (time or samples). If this standard does not meet its acceptance criteria, the samples between the last passing ICV or CCV and the failed CCV are not useable, under most circumstances.
 - 8.2.6.2.1 Instances where out of control data may be accepted are listed in Section 10.0 of this Manual. See also below in section 8.2.8.
 - 8.2.6.2.2 The Continuing Calibration Verification is from the same source as the calibration standards. (Unless otherwise specified in the client specific

Quality Assurance Program, or in the individual method SOP) For DoD work, the CCV must be made from the same source standard as that of the original curve.

- 8.2.7 Once the criteria are met for the ICV and CCV(s) encompassing all analytical data, the sample results can then be reported according to Section 12.0.
- 8.2.8 The acceptance criteria for the CCV and ICV are listed in each analytical SOP. Data associated with unacceptable calibration verification may be fully useable under the following special condition.
 - 8.2.8.1 When the acceptance criteria for the continuing calibration verification are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported without qualification for a continuing calibration verification failure. This special condition does not apply to any work done for a DoD project.

8.3 Proper Calibrations.

- 8.3.1 Initial instrument calibration procedures shall be defined in each analytical SOP. Some common requirements are listed here. (See also Admin SOP-038 and TECH SOP-009.)
- 8.3.2 A visual inspection of the chromatograms used for making the curves, where available, shall be done. If excessive peak tailing, irregular peak shape, or some other anomalous condition is observed, then that curve should not be used, and maintenance should be performed to correct the anomalous condition.
- 8.3.3 Peak identification will follow the guidelines set forth in the analytical SOP. Where not defined, the proper peak identification will be determined by its correct retention time, elution order, and relative peak strength. If it is a multiple peak analyte, the peaks to calibrate must be identified in the analytical SOP and shall remain the same throughout all quality control and sample analysis. 4 peaks shall be used when available. In the instances where a matrix interferent is on top of one or two of the calibration peaks of the multiple peak analyte, then an alternate peak or peaks can be used to calibrate and accurately quantitate the concentrations of that sample whose interferent is involved. For methods using

mass-spectral data, the qualitative identification of each compound determined is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

- 8.3.4 Sufficient raw data records must be maintained to permit reconstruction of the initial instrument calibration, e.g., calibration dates, test method(s), instrument, analysis date, each analyte name, analyst's initial or signature, concentration and response, and calibration curve or response factor.
- 8.3.5 All curves must be verified with a standard from a source separate than that used to make the initial curve. This shall be a standard from a different vendor or at a minimum, a different lot number of from the same vendor.
- 8.3.6 Unless otherwise dictated in the test method and analytical SOP, the calibration curve shall be made using the best combination of curve fit and/or the relative error. The curve used should be the one which gives you the best calculation of the calibrations points back against the calibration curve. (16.18 Appendix 3)

The calibration curve criteria are:

The %RSD of the calibration must meet method specific levels or at a minimum be \leq to 20% in order for the curve to be considered linear and to be used for analysis for samples.

If the method allows and a first or higher order curve fit is used a correlation coefficient meeting the method criteria shall be used. If no criterion is stated in the method for this fit, no less than a 0.990 correlation coefficient can be used for determining acceptability of the curve. Review by the department supervisor shall be required before an analyst is to use a second or higher order fit unless historically utilized for the analysis.

The relative error shall be documented and calculated as specified below:

For calibrations evaluated using an average response factor, the determination of the relative standard deviation (RSD) is the measure of the relative error.

For calibrations evaluated using correlation coefficient or coefficient of determination, the laboratory shall evaluate relative error by either:

Measurement of the Relative Error (%RE) Relative error is calculated using the following equation:

$$\% \text{ Relative Error} = \frac{x'_i - x_i}{x_i} \times 100$$

x_i = True value for the calibration standard

x'_i = Measured concentration of the calibration standard

This calculation shall be performed for two (2) calibration levels: the standard at or near the mid-point of the initial calibration and the standard at the lowest level.

The Relative Error at both of these levels shall meet the criteria specified in the method.

If no criterion for the lowest calibration level is specified in the method, then +/- 50% criterion will be the default lowest calibration level criterion and specified in the laboratory SOP.

If no criterion for the mid-point of the initial calibration level is specified in the method, then the methods CCV acceptance criterion will be the default mid-point of the initial calibration criterion and specified in the laboratory SOP.

or,

Measurement of the Relative Standard Error (%RSE)

Relative Standard Error is calculated using the following equation:

$$\% RSE = 100 \times \sqrt{\frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}}$$

x_i = True value of the calibration level i

x'_i = Measured concentration of calibration level i

p = Number of terms in the fitting equation

(average = 1, linear = 2, quadratic = 3)

n = Number of calibration points

The RSE shall meet the criterion specified in the method. If no criterion is specified in the method, the maximum allowable RSE shall be numerically identical to the requirement for RSD in the method. If there is no specification for RSE or RSD in the method, then the RSE shall be set to a default of 20% and specified in the laboratory SOP.

- 8.3.7 A visual inspection of the curve (where software makes this available) shall be taken. If the high point does not reasonable fall on the curve, then that point should be calculated back onto the curve to see its recovery passes CCV criteria. If it falls outside normal CCV acceptance criteria, then the curve has reached a point where it has exceeded its linear dynamic range. If the minimum amount of calibration points will still be met, then this high point can be dropped from the curve. Consideration should be made to lower that concentration for the next calibration event. If the high point is needed to meet the minimum number of calibration points, then the concentration of this high point shall be lowered to a level where it is linear.
- 8.3.8 The lowest point on the curve shall be at the establish PQL level, which is to be greater than that of the MDL level. This point must be included in the curve. If this point does not work in the curve, then maintenance shall be performed on the instrument to correct for the failure and a new curve should be analyzed.

- 8.3.9 No result quantitated higher than the high point of the curve shall be reported, unless circumstances dictate such, at which point those results must be qualified as having less certainty. Samples with results above the high point of the curve shall be diluted until results fall within the range of the curve.
- 8.3.10 For metals only: the following shall occur for instrument technology (such as ICP or ICP/MS) with validated techniques from manufacturers or methods employing standardization with a zero point and a single point calibration standard.
- 8.3.10.1 Prior to the analysis of samples, the zero point and single point calibration shall be analyzed and the linear range of the instrument shall be established by analyzing a series of standards, one of which shall be at or below the LOQ. Sample results within the established linear range will not require data qualifiers.
- 8.3.10.2 A zero point and single point calibration standard shall be analyzed with each analytical batch.
- 8.3.10.3 A standard corresponding to the limit of quantitation shall be analyzed with each analytical batch and shall meet established acceptance criteria. The linearity is verified at a frequency established by the method and/or the manufacturer.
- 8.3.11 If the initial instrument calibration results are outside established acceptance criteria, corrective actions shall be performed, and all associated samples re-analyzed. If re-analysis of the samples is not possible, data associated with an unacceptable initial instrument calibration shall be reported with appropriate data qualifiers.

8.3.12 TNI specifies the minimum number of points for establishing the initial instrument calibration as:

For regression or average response/calibration factor calibrations, the minimum number of non-zero calibration standards shall be as specified in the table below:

| Type of Calibration Curve | Minimum Number of Calibration Standards ^b |
|--------------------------------|--|
| Threshold Testing ^a | 1 |
| Average Response | 4 |
| Linear Fit | 5 |
| Quadratic Fit | 6 |

^aThe initial one-point calibration shall be at the project-specified threshold level.

^bFewer calibration standards may be used only if equipment firmware or software cannot accommodate the specified number of standards. Documentation detailing that limitation shall be maintained by the laboratory.

8.4 Improper Calibrations-Ethic rules that cannot be violated.
(See also Admin SOP-035 Ethics and Data Integrity, Fraud Prevention and Detection)

8.4.1 Any internal point of the calibration curve cannot be dropped to meet the curve acceptance criteria. (In a case of a mis-spike, poor injection, or error in making the standard that is clearly evident, and can be documented as such, then dropping that point, upon the supervisor's approval, will be allowed.)

8.4.2 The low point of the curve cannot be dropped.

8.4.3 Only the most current curve can be used to calculate QC and sample results. Going back and using an older curve to give the appearance of passing QC is not acceptable.

8.4.4 Performing improper manual integrations, including peak shaving, peak enhancing, or baseline manipulation to meet QC criteria or to avoid corrective action is not acceptable.

8.5 Maintenance

8.5.1 All maintenance performed on the analytical instruments used by AEL will be documented.

- 8.5.1.1 This includes maintenance performed by AEL employees or service technicians for the instrument manufacturer.
- 8.5.2 Each instrument will have a dedicated maintenance logbook. This logbook will detail the following.
 - 8.5.2.1 Instrument Name or Number
 - 8.5.2.2 Instrument Serial Number
 - 8.5.2.3 Instrument location
 - 8.5.2.4 Date the maintenance is being performed.
 - 8.5.2.5 The person performing the maintenance.
 - 8.5.2.6 A detailed description of what maintenance was performed.
- 8.5.3 It is the supervisor's responsibility to ensure these logbooks are kept up to date and accurate.
- 8.5.4 If the maintenance performed drastically alters the sensitivity or functionality of the instrument, a new MDL study will be performed before the analysis of samples can begin. The basis for determining whether the instrument has been drastically altered will be left up to the analysts and supervisors of the individual departments, but some basic guidelines are listed below.
 - 8.5.4.1 Generally, if the instrument can analyze an ICV successfully, then the maintenance performed was not severe enough to cause a new MDL study to be generated.
 - 8.5.4.2 The ICV is typically run after a new calibration following the maintenance procedure, but is not a requirement, unless specified by the analytical methods.
- 8.5.5 Maintenance can include any of the following, but is not limited to this list:
 - 8.5.5.1 Changing pump tubing for peristaltic pumps
 - 8.5.5.2 Changing the gas supply cylinders
 - 8.5.5.3 Cleaning the source of the GC/MS
 - 8.5.5.4 Changing a liner on the GC

8.5.5.5 Changing a membrane on the DO probe

8.5.5.6 Installing new light bulbs in a spectrometer

8.5.5.7 Changing air filters

8.5.5.8 Installing a new column in the GC

8.5.5.9 Cleaning and lubricating autosampler arms

8.5.5.10 Any work on the electronics of the instrument or autosampler.

8.5.6 The maintenance frequency of preventative maintenance will be listed in the analytical SOPs.

8.6 TNI Standards requirements for calibration and verification

8.6.1 The TNI Standards detail certain criteria for the calibration and verification, which are required for some of the analytical methods. Checklists for reviewing the analytical methods are provided by the Florida Department of Health (FDOH) on their website and prior to any scheduled FDOH audit. These documents are referenced when writing the analytical SOPs and the requirements in these documents are incorporated into the SOPs in addition to or in combination with the analytical method.

8.6.2 The TNI Standards and the FDOH checklists are stored on the designated Quality Assurance (Q) drive of the AEL networked servers and maintained current by the QA Officer.

8.6.3 Other documents may be used only if they are from an approved source. Check with the QA department to ensure that the publication referenced is approved and the most current information or revision.

9.0 Proficiency Testing (PT)

9.1 Fields of Testing

- 9.1.1 AEL shall perform PTs under the guidelines set forth in Module 1 of the TNI 2016 Standards. (For Jacksonville, also under the DoD QSM 5.4 guidelines)
- 9.1.2 AEL performs PT studies for all certified parameters that are listed in the TNI PT Fields of Proficiency Testing (FoPT) as found on the TNI website at: <http://www.nelac-institute.org/fopt.php>
- 9.1.3 Field of Proficiency Testing (FoPT) is defined as matrix, technology/method, and analyte combination. A PT sample is performed for all FoPT that AEL holds accreditation when listed on the most current listing of TNI Fields of Proficiency Testing.
- 9.1.4 When performing analysis under Department of Defense (DoD) ELAP accreditation, PTs are performed on every FoPT where a PT is reasonably available. This implies that American and European Vendors shall be reviewed for PT availability.
 - 9.1.4.1 When PT samples for an analyte-matrix-method combination cannot be obtained from any PT provider and the analyte-matrix-method combination is required for a scope of accreditation, the laboratory shall submit this fact in writing to the DoD ELAP Accreditation Body. Other measures (e.g., precision, bias, and selectivity) as outlined in the appropriate 2016 TNI Standard Test Modules must be performed to satisfy the PT requirement until those PT samples are available.

9.2 Frequency of testing

- 9.2.1 As required by the standards, AEL performs two PT studies per matrix each fiscal calendar year.
 - 9.2.1.1 The PTs matrices are:
 - 9.2.1.1.1 Water Source (WS), equivalent to drinking water on each lab's FDOH Scope of Accreditation and as drinking water on DoD ELAP Accreditation. All methods on the scope must be reported, not just each technology.

9.2.1.1.2 Water Pollution (WP), equivalent to non-potable water as listed on each lab's FDOH Scope of Accreditation and as aqueous on DoD ELAP Accreditation.

9.2.1.1.3 Solids equivalent to solid and chemical matrices as listed on each lab's FDOH Scope of Accreditation and as solids on DoD ELAP Accreditation.

9.2.2 For Continued Accreditation with current scope, the requirement for Florida is to participate in two studies within the fiscal year of the State, which goes from July 1 – June 30.

9.2.2.1 The closing dates of subsequent PT study samples for a particular accreditation FoPT (shall be no more than seven (7) months apart.

9.2.2.2 The opening date of PT study samples for a particular field of accreditation must be at least seven (7) calendar days after the closing date of a PT study for the same field of accreditation.

9.2.3 For Initial Accreditation to obtain new scope, the lab shall achieve a history of two (2) successful PT studies out of the most recent three (3) attempts for each field of accreditation specified for which the lab is seeking accreditation.

9.2.3.1 The two PT studies must be performed no more than 18 months prior to obtaining initial accreditation with the most recent PT no more than 6 months prior to submittal of an application with no longer than 7 months between attempts at successful performance.

9.2.3.2 The opening date of PT study samples for a particular field of accreditation must be at least seven (7) calendar days after the closing date of a PT study for the same field of accreditation. PTs for a second study cannot be shipped until after the close of the previous study.

9.3 Analysis of PTs.

9.3.1 The PTs must be performed at the lab at which they are received.

9.3.2 Communication between lab personnel about open study results prior to the close of the study is prohibited.

- 9.3.3 No lab personnel shall attempt to obtain assigned values for any portion of an open PT study from the PT provider prior to the close of the study.
- 9.3.4 By NELAC requirement, PT samples must be handled in the same manner as client samples using the same staff, the same routines, the same equipment, the same facilities, and the same frequency of analysis. PT samples cannot be run multiple times unless that is the same procedure as used when analyzing client samples.
- 9.3.5 It should be noted that most reporting by AEL is reported at the MDL level. The TNI 2016 TNI Standards allow for the reporting at the LOQ level or PRTL level. However, PT providers will now be grading PTs performance down to the PRTL (Proficiency Testing Reporting Limit defined as the lowest level at which the PT can be spiked by the PT provider).
- 9.3.5.1 If the lab reports $< (\text{LOD}) \text{ MDL}$ or $< \text{LOQ (PQL)}$ when those values are above the PRTL where the analyte is present, the PT shall be scored as “Not Acceptable” by the PT provider.
- 9.3.6 Dilutions are allowed under this requirement, as it is normal procedure to run and report a result within the range of the instrument calibration. If a PT sample has multiple analytes at different ranges, the dilutions should span the acceptable ranges for those analytes.
- 9.3.7 All daily operating procedures must be followed when analyzing a PT sample. This is to ensure that a PT sample is handled in the same way as a client sample. If the method requires an instrument blank, calibration checks, degradation breakdown checks, LCS, MS, and MSD, then they must be performed when analyzing a PT sample.
- 9.3.8 If the QC fails, the data is to be qualified or rerun as would a client sample.
- 9.3.9 If the PT is the only sample for that day's analysis or preparation, then either the PT sample can be used as the matrix spike sample, or a previously analyzed sample with a similar matrix can be used as spike sample, or a LCS and LCS duplicate pair can be analyzed. A case narrative should accompany an LCS and LCS duplicate pair, as that would be the normal procedure when reporting client samples.

9.4 Results

- 9.4.1 The requirement for proficiency to be proven is to have 2 acceptable results for every parameter out of the previous 3 results.
- 9.4.2 Any parameter that does not meet this requirement will require makeup PT samples.
- 9.4.3 The Project folders for each study are kept in the QA Officer's custody. The project folders contain all information about the samples, from receipt to distribution to raw results to benchsheets, etc.
- 9.4.4 The approved results from the PT Providers are stored in binders in the QA Officer's custody. These contain only the approved results and no other information. They are referenced to the project folder by the study name and date.

9.5 Ordering

- 9.5.1 The ordering of the standards is maintained by the QA Officer to ensure compliance with the requirements.
- 9.5.2 As required in the rule, these samples are logged into the system as normal samples into the AEL LIMS system.
 - 9.5.2.1 This is to ensure these samples are treated as routine samples and no special consideration is placed on them.
- 9.5.3 Once logged in, the QA Officers act as PMs for these projects and do the final review on the data before reporting the results to the provider.
- 9.5.4 The QA Officer is responsible for submitting the results to the PT Provider.

9.6 PT Sample Login

- 9.6.1 All PT orders are received by the QA Officer and logged into the LIMS under the QA Officer's supervision. This allows for the order to be checked for accuracy by the person doing the ordering and ensure the certified parameters mentioned in the attached tables and covered effectively.
- 9.6.2 Sample numbers from the individual labs will be provided to the QA Officer at log-in of the study from the provider.

- 9.6.3 The individual project numbers are then used to log each study in for each lab as a project with the correct Project Number Prefix for each lab. Example: Studies for Jax will have project numbers that start with a J, Gainesville with a G, Tampa with a T, and Orlando with an A, as defined in ADMIN-005, section 6.13.1.
- 9.6.4 Once the studies are logged in correctly and reviewed by the QA Officer, the samples and project folders will be distributed to the individual labs for analysis.

9.7 Tracking or Results

- 9.7.1 The results are tracked according to study and matrix with those tracking spreadsheets saved electronically on the designated Quality Assurance (Q) drive of the AEL networked servers.
 - 9.7.1.1 An example of the tracking spreadsheet is displayed as Table 9.1.
- 9.7.2 The spreadsheets are used by the QA Officer to verify the frequency is correct and falls within the fiscal year as well as monitoring 2 out of 3 acceptable results for every certified analyte requiring a PT study.

9.8 New Certifications and Corrective Actions

- 9.8.1 For all parameters AEL tries to add to its certified analyte list, there is the option of doing quick response PT studies. These can be performed to speed up the process to allow for a faster certification.
- 9.8.2 The Initial Accreditation to obtain new scope, the lab shall achieve a history of two (2) successful PT studies out of the most recent three (3) attempts for each field of accreditation specified for which the lab is seeking accreditation.
 - 9.8.2.1 The two PT studies must be performed no more than 18 months prior to obtaining initial accreditation with the most recent PT no more than 6 months prior to submittal of an application with no longer than 7 months between attempts at successful performance.
 - 9.8.2.2 The opening date of PT study samples for a particular field of accreditation must be at least seven (7) calendar days after the closing date of a PT study for the same field of

accreditation. PTs for a second study cannot be shipped until after the close of the previous study.

- 9.8.3 Any parameter that is unacceptable in its result must have a corrective action generated to define the error and how it is being corrected as described in Section 10.0 of this Manual.
- 9.8.4 If this unacceptable result signifies a failure of 2 of the past 3 results, then a quick response PT can be analyzed to prove compliance is re-established (assuming the new result is acceptable).
- 9.8.5 The contracted Accreditation Bodies such as ANAB or Perry Johnson must be made aware of any multiple PT failures (when a 2 out of the last 3 are failed for an analyte) within 30 days of the lab being notified of that failure. The 30 days are in addition to the 21 days in which the PT providers are to provide results once a study is closed. Notification to the Accreditation Bodies shall be in the form of a Non-Conformity Form (NCF) or Notice of Corrective Action Report (NCR). All pertinent information shall be included on the notification such as the reported and true values, the cause and identification of the latest failure, the corrective action, and time frame for the correction.

9.9 Laboratory Conduct

- 9.9.1 The laboratory is not to knowingly accept PT samples from another laboratory for analysis or is the laboratory to send out a PT to another laboratory for analysis.
- 9.9.2 The laboratory management & staff are not to communicate with any individual at another laboratory (including inter-laboratory) concerning PT sample results.
- 9.9.3 The laboratory is not to attempt to obtain the assigned value of the PT from the provider until after the study has closed.
- 9.9.4 PT records are to be kept for 5 years.

Table 9.1: next page

Table 9.1:

Non-Potable Water Matrix

| Parameter PESTICIDES | T | Cert | Required PT | Technology | Methods | WP19-4 | WP20-2 | WP20-4 | WP21-2 |
|---------------------------|-----|-------|----------------|------------|----------------|------------|-----------|------------|-----------|
| | | | | | | 11/13/2019 | 5/15/2020 | 11/14/2020 | 5/19/2021 |
| Aldrin | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| alpha-BHC | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| beta-BHC | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| delta-BHC | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| gamma-BHC (Lindane) | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| gamma-chlordane | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| Alpha-Chlordane | A | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| Chlordane (total) | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| DDD | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| DDE | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| DDT | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| Dieldrin | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| Endosulfan I | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| Endosulfan II | P | NELAP | YES | GC/ECD | E608.3/SW8081A | F | A | A | S |
| Endosulfan Sulfate | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| Endrin | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| Endrin Aldehyde | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| Heptachlor | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| Heptachlor Epoxide (beta) | P | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| Methoxychlor | A | NELAP | YES | GC/ECD | E608.3/SW8081A | A | A | A | S |
| Toxaphene | P | NELAP | YES | GC/ECD | SW8081A | A | A | A | S |
| Endrin Ketone | P | NELAP | EXP | GC/ECD | SW8081A | A | A | A | S |
| PCBs in Water* | | | | | | | | | S |
| Aroclor 1016 | PCW | NELAP | YES | GC/ECD | E608.3/SW8082 | A | A | A | S |
| Aroclor 1221 | PCW | NELAP | YES | GC/ECD | E608.3/SW8082 | A | A | A | S |
| Aroclor 1232 | PCW | NELAP | YES | GC/ECD | E608.3/SW8082 | A | A | A | S |
| Aroclor 1242 | PCW | NELAP | YES | GC/ECD | E608.3/SW8082 | A | A | A | S |
| Aroclor 1248 | PCW | NELAP | YES | GC/ECD | E608.3/SW8082 | A | A | A | S |
| Aroclor 1254 | PCW | NELAP | YES | GC/ECD | E608.3/SW8082 | A | A | A | S |
| Aroclor 1260 | PCW | NELAP | YES | GC/ECD | E608.3/SW8082 | A | A | A | S |

A= acceptable S= scheduled F=failure

10.0 Non-Conformities and Out-of Control Data

10.1 Definitions

NOTE: Both Non-Conformities and Out-of Control Data are handled according to ADMIN SOP-016, titled "Non-Conformity Notification." To briefly summarize:

10.1.1 A non-conformance is anything that happens during the receipt or analytical process that is different than what should be expected or required by the method, the SOP, or routine policies and procedures.

10.1.1.1 At sample receiving, any sample with improper preservation, in an improper container, with a hold time that is exceeded, or received in any other abnormal condition will require an NCF and notification to the project manager. The project manager is then to contact the client for instruction on whether to continue with the testing and qualify the data, or to stop analysis and wait for a resample.

10.1.1.2 Each analytical SOP details which types of circumstances require filling out a non-conformity form (NCF), sometimes also referred to as corrective action report (NCR) in the industry, and which types of routine failures are satisfactorily explained within the scope of the SOP itself and require only data qualification. In cases where data quality objections are not met on method blanks, continuing calibrations, laboratory control spikes, and other QC of this nature, the issue will require a supervisor and lab manager to be notified. (All NCFs are distributed to the lab manager and appropriate department supervisor for review and action)

10.1.1.3 Permission to deviate from the SOP or other established procedures must be accompanied by an NCF, which is then reviewed by QA and the laboratory management. Where appropriate, and when properly documented, the laboratory management can permit departures from these documented policies and procedures and/or from the standard specifications. These departures (see ADMIN SOP-016 for a complete listing) can consist of cases such as reduction in sample volume used where low levels are not required, high dilutions on samples of an overly contaminated nature, and deviations from normal procedures when the sample's behavior exhibits unusual characteristics during analysis. Documentation through the NCF program and the inclusion of an explicit case narrative are part of the laboratory management's arrangements for handling exceptionally permitted departures. Client notification is also integral as part of dealing

with these "out of the ordinary" situations. This notification being by case narrative and when deemed necessary, by phone or e-mail.

10.1.1.4 If a condition exists that is not already defined in the SOP or if deemed that work should be suspended in a particular procedure due to a quality control issue, resumption of work is to be only after QA approval.

10.1.1.5 All audit findings shall be written onto an NCF and tracked through the NCF program.

10.1.1.6 All PT failures shall be written onto an NCF and tracked through the NCF program.

10.1.2 An out-of-control data situation is any data that is required to be reported where all acceptance criteria were not met for the calibration and/or the quality control data.

10.1.2.1 The analytical SOPs detail which types of out-of-control circumstances require a (NCF) to be generated, and which types of routine failures are satisfactorily explained in the SOP itself and/or proper qualification.

10.2 Non-Conformity Form (NCF) and Notice of Corrective Action Report (NCR)

10.2.1 NCFs and NCRs for the purposes of this Quality Manual will be used synonymously.

10.2.2 NCFs and NCRs are used to document occurrences in the lab that are seen as anomalies to normal operation. When used for the purpose of documenting a lab error or QC failure, the following elements must be included in the NCF or NCR:

10.2.2.1 **Identification:** Identification of the issue or condition and whether it requires corrective action.

10.2.2.2 **Root Cause:** If the issue is an error or a condition that requires corrective action, the root cause must be identified.

10.2.2.3 **Plan of corrective action:** Once identified, what specifically will be done to correct the issue at hand and prevent its future occurrence.

10.2.2.4 **Timetable:** When will the corrective action take place, and if not immediately, how long will it take to implement corrective action.

10.2.2.5 **Follow-up:** A time for when a follow-up is to take place to ensure that the corrective action will prevent future occurrences and/or to check that corrective measure remain in place shall be defined in the case narrative.

10.3 Documentation and Storage

10.3.1 Any event that requires an NCF is to be brought to the attention of a QA Officer. The QA Officer will take the information provided and seek out any other information as the situation requires and generate an NCF using the "Lab NCF" application found on the AEL Intranet website. NCFs at each stage of development will be automatically saved in each lab's designated folder on the Quality Assurance (Q) drive of the AEL networked servers.

10.3.2 The QA Officer will distribute the NCF to all pertinent personnel and will redistribute as updates are made and information added to the NCF. Upon completion, the QA Officer shall close the NCF.

10.3.3 All NCFs are tracked and trended using the application generated Excel spreadsheet on the AEL Intranet website. The form has menu filtering to select for a particular issue, type, or department. All information from the NCF is transferred to this spreadsheet for easy access and dissemination.

10.4 Corrective Actions

NOTE: The Admin SOP-016 details the exact procedure for handling a non-conforming situation but listed below is the brief overview.

10.4.1 When a non-conformity issue is found or any condition that requires corrective action is seen, the analyst responsible will initiate the NCF process by getting all relevant information to the QA Officer in an e-mail.

10.4.2 The QA officer shall also generate an NCF for any failed PTs, Audit findings, or any conditions needing action found while doing data reviews.

10.4.3 Next, a corrective action is developed. If the root cause and a corrective action is not readily apparent, then a plan for the discovery of the root cause shall be developed.

- 10.4.4 Once the analyst has completed the process of root cause discovery and corrective action, the NCF form is updated.
- 10.4.5 The QA Officer will review the non-conformity and corrective action and decide whether to approve or reject the NCF. A time will be set if follow-up is to occur.
- 10.4.6 Once approved, the final NCF shall be distributed to those involved or in need of notice of the NCF.
 - 10.4.6.1 If the corrective action involves client notification, the PM for that client will document by e-mail or other electronic means when the client was contacted and what decision was reached. The client is to be notified within 24 hours of any event, audit finding, identification of defective measurement, or error that would cast doubt on the validity of results.

10.5 Client Complaints

- 10.5.1 AEL utilizes the NCF form to track and resolve customer complaints as well.
- 10.5.2 The description of the non-conformity in this case will be the client's complaint or question.
- 10.5.3 The corrective action will detail how the question or complaint is resolved.

10.6 Approval

- 10.6.1 All decisions for proceeding with an out-of control situation will be determined by the QA Officer. The Lab Manager may be consulted, but final authority is given to the QA Representative involved with the situation.
- 10.6.2 The QA Representative will take into account the following when deciding whether to reject or accept the situation:
 - 10.6.2.1 How is the data affected?
 - 10.6.2.2 What is the end use of the data – compliance or non-regulatory?
 - 10.6.2.3 Is there enough sample to reanalyze?

10.6.2.4 Consult other data guidelines such as TNI quality control acceptance rules, FDEP Guidelines, CLP data guidance, or EPA's guidance on how severely the data is affected.

10.6.2.5 Call the client for consultation with non-conformities and let the client make the decision to proceed.

10.6.2.5.1 This is done with extreme caution and typically only for instances involving hold time issues or sample receipt issues.

10.6.2.5.2 The client may be consulted for other issues, but the laboratory will make the final decision regarding the data and not the client. All applicable qualifiers will be applied according to the rules and SOPs and not according to client request.

10.7 DoD Client Notifications.

10.7.1 The lab upon discovery, notifies all affected customers of potential data quality issues resulting from nonconforming work within fifteen (15) business days. Notification shall be performed according to a written procedure.

10.7.2 The notification shall be with electronic documentation. This can consist by sending an e-mail and receipt notice or client response. If notification is for a project in progress, this record shall also be attached, unless otherwise resolved (such as situation corrected and samples rerun, etc.).

10.7.3 Records of corrections taken or proposed corrective actions to resolve the nonconformance shall be submitted to the customer(s) within thirty (30) business days of discovery.

10.7.4 All DoD ELAP laboratories, must report any instances of inappropriate and prohibited laboratory practices, as detailed in Section 5.2.7 of the DoD QSM, to their AB within fifteen (15) business days of discovery.

10.7.4.1 If the AB is not notified within fifteen (15) business days the AB will immediately suspend the laboratory's DoD ELAP accreditation.

10.7.5 All DoD ELAP laboratories must submit records of associated corrections taken or proposed corrective actions to their AB within thirty (30) business days of discovery.

- 10.7.6 In the process of a review, such as in internal investigation or annual internal audit, the lab shall notify DoD/DOE clients within fifteen (15) business days of discovery of any investigation that casts doubt upon the validity of test results.

10.8 Case Narratives

- 10.8.1 Case narratives are addendums to the analytical report that explain anything out of the ordinary that happened with the samples during the sample receipt through the analytical processes.
- 10.8.2 The application and completion of case narratives are explained in detail in ADMIN SOP-028.
- 10.8.3 The LIMs database manages and maintains the case narratives through the AEL intranet.
- 10.8.4 The analyst initially compiles the case narrative. The case narrative explains anything that deviated from normal procedure with the samples.
- 10.8.4.1 The analytical case narratives are compiled for each analytical batch. They are broken down into sections – one for organics and one for inorganics. An example of the organic case narrative format is attached as Figure 10.2 and the inorganic example is attached as Figure 10.3 to the end of this section.
- 10.8.4.2 The analyst generated case narrative details everything that occurred in the analytical batch and all samples affected.
- 10.8.5 Once the analyst generates the case narrative, the case narrative or case number assigned number is available for the project manager (PM). The PM will then modify the case narrative to make a project specific case narrative. This modification process does not overwrite the one generated by the analyst, but simply creates a new narrative based upon the information the analyst entered.
- 10.8.5.1 The project specific narrative will be edited to take out any references to samples that are not included in the project being reviewed.
- 10.8.6 Once the project specific narrative is generated, it is attached to the end of the analytical report. If there were no irregularities that occurred during the process for a particular analytical batch, it is not necessary to attach a narrative for the analytical batch.

10.8.6.1 It may be possible to have multiple case narratives attached to the end of the report if multiple analytical batches had irregularities occur during the process.

10.8.6.2 If hard copy printouts of the case narratives are made, they are stored in the individual project files.

10.9 Preventive action

10.9.1 Preventive action is a pro-active process to identify opportunities for improvement rather than a reaction to the identification of problems or complaints.

10.9.2 Needed improvements and potential sources of nonconformance, either technical or concerning the quality system, shall be identified. If preventive action is required, action plans shall be developed, implemented, and monitored to reduce the likelihood of the occurrence of such nonconformance and to take advantage of the opportunities for improvement.

10.9.3 Procedures for preventive actions shall include the initiation of such actions and application of controls to ensure that they are effective.

10.9.4 Such preventative actions shall include the review of procedures at a corporate level by combining the efforts of each lab's QA officers. A review of the best procedures from the best interpretations of the methods, available literature, and industry lab practices, and by obtaining a consensus from all laboratory supervisors and lab managers, updates to operating procedures shall be improved. Where a method allows for some variability, the laboratory network will strive to find the best practice by developing uniformity in equipment purchased, reagents used, standards used, calibration points, documentation, and any other areas where consistency in quality and reporting will benefit.

10.9.5 Suggestion for areas for improvement are to be sent to the supervisors, lab managers, and/or QA officers. An atmosphere shall exist within the laboratory network that encourages input and an active involvement from all employees. The implementation of Tiger Teams is an example of such involvement.

10.9.5.1 Tiger Teams.

10.9.5.1.1 The Corporate Operations Manager and Corporate Technical Director will identify areas where

improvements in procedure shall improve either the quality or efficiency of a test procedure or lab process.

- 10.9.5.1.2 The person with the best base of knowledge for the procedure or process to be improved shall be assigned as team leader. Other team members shall be selected to the team so that a representative from each of the sister labs using the procedure or process can have an input in the development of the improvements.
- 10.9.5.1.3 Timelines are set for when the findings and improvements from the Tiger Team are to become effective. This is coordinated with the QA department, as an SOP is to be revised or created.
- 10.9.5.1.4 The final improvements are reflected in the revised or newly created SOP. The effective date of the SOP is the effective implementation date of the improvement to the procedure or process. Other documentation of the Tiger Team's efforts shall not be required.

Figure 10.1

CASE NARRATIVE
Organic Analysis

Laboratory Reference No./SDG#:

Client/Project:

I. RECEIPT

No exceptions were encountered unless a Sample Receipt Exception Report is attached to the Chain-of-Custody or a communication form is included in the addendum with this data package.

II. HOLDING TIMES

A. Sample Preparation: All holding times were met.

B. Sample Analysis: All holding times were met.

III. METHOD

Preparation: SW-846 3550B

Analysis: SW-846 8270C

IV. PREPARATION

Sample preparation proceeded normally.

V. ANALYSIS

A. Calibration: All acceptance criteria were met.

B. Blanks: All acceptance criteria were met.

C. Surrogates: All acceptance criteria were met.

D. Spikes: A project specific matrix spike was not requested for this sample set.

E. Internal Standards (if applicable): All acceptance criteria were met.

F. Samples: Sample analyses proceeded normally.

G. Other:

I certify that this data package is in compliance with the terms and conditions agreed to by **Laboratory Name** and by the client, both technically and for completeness, except for the conditions detailed above. The Laboratory Manager or his designee, as verified by the following signature, has authorized release of the data contained in this hard copy data package and in the computer-readable data submitted on diskette:

Signed: _____ Date: _____
Name Position

Figure 10.2

CASE NARRATIVE
Inorganic Analysis

Laboratory Reference No./SDG#:

Client/Project:

VI. RECEIPT

No exceptions were encountered unless a Sample Receipt Exception Report is attached to the Chain-of-Custody or a communication form is included in the addendum with this data package.

VII. HOLDING TIMES

C. Sample Preparation: All holding times were met.

D. Sample Analysis: All holding times were met.

VIII. METHOD

Preparation: 3050/7471

Analysis: 7020/7060/7740/7761/7471/6010

IX. PREPARATION

Sample preparation proceeded normally.

X. ANALYSIS

H. Calibration: All acceptance criteria were met.

I. Blanks: All acceptance criteria were met.

J. Spikes: All acceptance criteria were met.

K. Duplicates: All acceptance criteria were met.

L. Serial Dilution: All acceptance criteria were met.

M. Samples: Sample analyses proceeded normally.

N. Other:

I certify that this data package is in compliance with the terms and conditions agreed to by **Laboratory Name** and by the client, both technically and for completeness, except for the conditions detailed above. The Laboratory Manager or his designee, as verified by the following signature, has authorized release of the data contained in this hard copy data package and in the computer-readable data submitted on diskette:

Signed: _____ Date: _____

Name
Position

11.0 Audits and QA Review

11.1 Annual Internal Audits

- 11.1.1 The Corporate Technical Director, the lab QA Officer, or a QA designated person who is trained and qualified in the area under review, will conduct an audit of each laboratory's departments on an annual basis. (See section 11.1.8 for qualifications to perform internal auditing.)
- 11.1.2 A biennial assessment performed by FDOH (or for FDOH by a lab contracted auditor) cannot be substituted as an internal audit for the year it was performed.
- 11.1.3 The Internal Audit will be a review of the overall quality system and a detailed review of the analytical methods for the year (or audit period) that the internal audit covers. The Internal shall include review and reference to observations and issues seen over the course of the year summarized with the final report. The audit shall be organized by department with a final report issued listing any changes or corrective actions needed. Each finding shall be issued an NCF with set timelines for correction established. All records for these audits shall be kept with the QA records. All the materials collected and each department summary shall be collected into one report for re-evaluation and a final review.
 - 11.1.3.1 The FDOH checklists will be utilized as guidelines. These can be found on the FDOH website. If unavailable on the website, contact the corporate QA for a copy.
 - 11.1.3.2 The Quality System checklist will be utilized for the overall quality system review.
 - 11.1.3.3 The Chemistry and Microbiology checklists will be utilized for the individual analytical methods review.
 - 11.1.3.4 For microbiology, also review the FDEP Microbiology Audit checklist, which can be found on the designated Quality Assurance (Q) drive of the AEL networked servers.
 - 11.1.3.5 When using the checklists, each method to be assessed shall be listed or highlighted on the checklist so that it is readily apparent which methods were reviewed. A review of the checklists shall ensure that all methods on the scope for that department were addressed during the audit.

- 11.1.3.6 The most recent previous internal and external audits shall be reviewed to ensure continued compliance to the stated corrective action for any findings seen during those audits. The dates of the materials checked, and any relevant notes should be written next to the related finding on a copy of the old audit report and kept with the notes and checklists for the new ongoing internal audit.
- 11.1.3.7 A review of NCFs, any incidence reports, client complaints, or any lab procedural changes shall be reviewed as part of the Internal Audit.
- 11.1.3.8 The auditor shall eye witness the performance of several of the methods and to question the analyst while they are performing the digestion, extraction, or analysis. An understanding of the proper technique for the procedure should be observed such as for example, proper pipette and syringe use, proper spiking technique, and working aseptically.
- 11.1.4 The annual audits will occur according to the internal audit schedule as seen in Figure 11.1. Strict adherence to the listed dates is not required, but internal audits should fall roughly in the date ranges specified. Each lab manager in conjunction with the QA officer will choose a time that gives the best benefit to the lab. The full cycle of department audits shall be completed prior to the regularly scheduled biennial audits, and in conjunction with a location move, major personnel changes, or the addition of scope. The next annual round can be extrapolated for an extended year from figure 11.1.
- 11.1.4.1 For all labs, the internal audit findings must have issued an NCF or NCR form. In other words, root cause, corrections, and follow-ups shall be documented through the NCF program.
- 11.1.5 Any deficiency that is found during these internal audits that could have an effect on the validity of data that has already been reported to the clients will be documented and researched thoroughly. If it is determined that amended reports will be required, the clients involved will be contacted in writing with the reason for the amended reports explaining that a deficiency was found during the internal audit that may have compromised the integrity of the data.
- 11.1.5.1 The clients will receive a detailed explanation of what the error was and how it is being corrected.
- 11.1.5.2 The client shall be notified as soon as practical after the discovery of the issue, not to exceed 15 days. The lab manager

shall be made fully aware whenever a client is to be notified and shall be responsible to ensure that notification has been made and have readily available documentation.

11.1.6 The Annual Internal Audit follows the basic procedure detailed in SOP ADMIN-020.

11.1.7 All deficiencies will be alerted to the Technical Director of the corresponding laboratory and the QA Officer and Technical Director will ensure that all deficiencies are corrected, and the quality system is followed as described in this Manual to ensure future compliance.

11.1.7.1 The audit results will be stored in the custody of the QA Officer and can also be found on the designated Quality Assurance (Q) drive of the AEL networked servers. If there are any deficiencies, they will be handled with corrective actions accompanying the resolutions.

11.1.8 The QA officer or the designated person shall have the technical expertise to perform the audit in the area under review with a full understanding of the procedures and required Quality Control.

11.1.8.1 The QA officer shall have conducted an internal audit side by side with an experienced QA officer prior to conducting one solo.

11.1.8.2 Prior to the audit the QA officer is to have read and compared the base method against the SOP for each test method to ensure agreement in procedure and QC requirements.

11.1.8.3 If the QA officer does not have previous hands on experience performing testing in a department, that QA must perform an IDOC on at least two representative test methods in that department prior to conducting the audit for that department.

11.1.8.4 For a person designated to perform an audit in a department, that person must be independent of the day to day activities in that department, must have either been an analyst for over a year on the methods being audited, or have experience as a supervisor for that department.

11.2 Data Audits

11.2.1 If a client calls with a question about a result or an erroneous report is sent to the client, and if deemed to be of a systematic or severe error, a

full data audit may be warranted, which will be referred to as a data review.

11.2.2 Data audits are an intense review of the data associated with a certain project or group of projects. The goal is to verify that the quality system in place has accounted for all irregularities that may have occurred with the samples and that the data review process in place is operating properly.

11.2.2.1 The process for reviewing analytical data is explained in Section 12.0 of this Manual.

11.2.3 The QA Officer or QA Deputy checks random projects on a monthly basis to ensure these procedures are being followed properly as well.

11.2.3.1 For labs accredited under DoD ELAP, the quality manager or designee reviews a minimum of 10% of all data packages for technical completeness and accuracy on a quarterly basis. This review is considered a part of overall data review and does not need to be completed before the data package is issued to the customer. If data quality issues are discovered during the review, the client shall be notified within fifteen (15) business days of the discovery of the issue. 10% of all projects shall be fully reviewed inception to completion and must include review of sample login, raw data, data entry, and final report. Documentation on review forms is mandated.

11.2.4 If common, routine, or repeated errors are found during the random review, it may instigate a more in-depth review of the area of the quality system affected.

11.2.5 Data Audits will also verify the traceability of measurements according to the procedures outlines in Section 5.0 of this Manual.

11.2.6 Non-conformities, as explained in Section 10.0, can also instigate a data audit if the non-conformity is routine or severe in nature. This determination is the responsibility of the QA Officer.

11.3 Audits by an outside agency

11.3.1 AEL will routinely go through full audits by outside agencies, such as regulatory agencies, accrediting bodies, and clients.

11.3.1.1 An FDOH or FDOH approved on-site assessment is a biennial requirement.

11.3.2 These assessments can be either data audits or a full review of the quality system depending upon who is performing the assessment and the reason for it.

11.3.3 The findings of these assessments are maintained in the custody of the QA Officer, but any findings (deficiencies) that are found will be addressed between the QA Officer and Technical Director.

11.3.3.1 Any deficiency will be corrected, and a corrective action response will be documented accordingly. If so required, these corrective actions will be submitted to the entity performing the assessment.

11.3.3.2 These corrective actions and responses plan to the findings will be completed in a timely fashion, generally no more than 30 days after the assessment. The plan will state timelines for the corrections to be completed.

11.3.3.3 Corrective actions must be implemented by the lab in the fashion and in the timelines submitted to the accrediting body and FDOH.

11.3.4 The findings of the audit will be available for all employees to review and comment upon.

11.3.5 These findings and corrective responses will be made available for all entities that wish to review them as part of an assessment of AEL as well.

11.3.6 Willful avoidance of approved corrective action implementation may result in loss of DoD ELAP accreditation or in DOECAP Priority I findings.

11.3.7 As a result, work may be discontinued until implementation is verified by the DoD ELAP AB or DOECAP Operations Team, as appropriate.

11.4 Annual Management Review

11.4.1 The Technical Director of each laboratory in conjunction with the QA Officer on an annual basis performs the annual management review.

11.4.1.1 The review process is outlined in SOP ADMIN-021.

11.4.2 The management review is a review of the Quality System by the Technical Director of the individual laboratory. It includes a thorough review of the following:

11.4.2.1 The AEL Quality Manual. Since it is the QA Officer's responsibility to keep the Manual current, it is reviewed by another source, other than the QA Officer, to ensure compliance with this requirement.

11.4.2.2 The Quality System in general.

11.4.2.2.1 This can entail going through certain sections of the Quality Manual and verifying the policies in place and being followed throughout their individual laboratories.

11.4.2.2.2 The Quality System checklist is utilized as a guideline for performing this annual review.

11.4.2.3 This review allows the QA Officer and Technical Director to confirm the Quality System in place is effective and functioning properly.

11.4.2.4 The Quality Manual will typically be reviewed following the Annual Management Review to account for any changes that were determined needed to be made during this review and to coincide with any new regulatory changes that must be accounted for in the Quality Manual.

11.4.2.5 The Annual Management review will also review any audits that have occurred by outside agencies. During the review of those, any deficiencies found will be verified as corrected and the correction is still in effect.

11.4.2.6 This review will also go through the non-conformities to ensure all routine or severe errors have been corrected satisfactorily.

11.4.2.7 Any deficiencies found during this review will be discussed and resolved with the QA Officer and/or Lab Director.

11.4.2.8 The Technical Director will also review the PT study summaries to ensure the proper protocol is being followed and the analytes for which they have certification are in compliance with the PT requirements. This is a review of the system in place by the QA Officer for tracking these, as detailed in Section 9.0 of this Manual.

11.5 Data Validation

- 11.5.1 The total data validation is a requirement of the QA Officer or QA Deputy to review on a routine basis.
- 11.5.2 This is performed on a project-by-project basis and is a random review. (See also 11.2.3)
- 11.5.3 This project review is to verify the data meets all objectives of the Quality System, all data is properly traceable, and free of transcription errors.
- 11.5.4 The actual reporting of the data and peer review process before assembling the final report is detailed in Section 12.0

11.6 QA Acceptance and Rejection Criteria

- 11.6.1 The criteria used for acceptance or rejection of quality control data is defaulted to the limits listed in the individual analytical methods.
- 11.6.2 If the methods do not define the limits or if project specific limits provided by the client are not available, then other sources may be consulted, such as;
 - 11.6.2.1 CLP Data Assessment documentation – on file in the QA Officer’s custody.
 - 11.6.2.2 FDEP’s data assessment guidelines, as provided in the FDEP SOPs, which are also on file in the QA Officer’s custody.
 - 11.6.2.3 Other methodologies, such as CLP methods that may be similar, but yet have the limits defined for certain standards.
 - 11.6.2.4 Sound professional judgment, based upon experience by the analyst(s). This is typically in the inorganic testing arena and is the last resort for setting acceptance criteria. This can only be done with consultation between the QA Officer and Technical Director, and never exclusively at an analyst’s discretion.

11.7 Measurement of Uncertainty

- 11.7.1 The measurement of uncertainty is defined by AEL as the acceptance limits for the Laboratory Control Spike (LCS). These limits can be either defined by the method, defined by the FDOH checklists, or in-house limits, determined by tracking the LCS recoveries.

- 11.7.1.1 For tests or methods which do not lend themselves to analyzing a LCS, either the limits stated in the method for precision and accuracy will be utilized or the calibration verification standard limits will be used.
- 11.7.2 The measurements of uncertainty values are detailed in SOP ADMIN-024.
- 11.7.3 The steps utilized and all determining factors to the measurement of uncertainty are detailed in SOP ADMIN-024.
- 11.7.4 A statement can be added on the last page of each analytical report, which states, "*The estimated levels of uncertainty can be provided upon request.*"

Figure 11.1. **AEL TENTATIVE ANNUAL AUDIT SCHEDULE**

AEL Tampa Internal

April 2022 Wet Chemistry
June 2022 Metals
August 2022 Microbiology
September 2022 Organics
October 2022 Quality Systems
Close: December 31st, 2022

Tampa External

Nov 2022

AEL Jacksonville – QA Internal Audit

June 2022 Microbiology
July 2022 Metals
September 2022 Organics
November 2022 Wet Chemistry
December 2022 Quality Systems
Close: December 31st, 2022

Jacksonville External Including DOD

March 2022

AEL Fort Myers – QA Internal Audit

February 2022 Microbiology
April 2022 Wet Chemistry

May 2022 Quality Systems
Close: June 30th, 2022

Fort Myers External

May 2023

AEL Gainesville – QA Internal Audit

March 2022 Microbiology
April 2022 Wet Chemistry
May 2022 Quality Systems
Close: May 31st, 2022

Gainesville External

July 2022

AEL Orlando – QA Internal Audit

February 2022 Microbiology
April 2022 Wet Chemistry
May 2022 Quality Systems
Close: May 31st, 2022

Orlando External

July 2022

AEL Miami – QA Internal Audit

February 2022 Microbiology
March 2022 Metals
May 2022 Wet Chemistry
June 2022 Organics
July 2022 Quality Systems
Close: August 31st, 2022

Miami External Including ISO

October 2022

AEL Tallahassee – QA Internal Audit

March 2022 Microbiology
April 2022 Wet Chemistry
May 2022 Quality Systems

Tallahassee External

August 2022

12.0 Reporting and Analytical Results

12.1 Detection Limits

See also AEL ADMIN SOP-012 MDLs, LODs, PQLs, and LOQs.

The lab reports are generated using the MDL (Method Detection Limit) as the level of detection and the PQL (Practical Quantitation Level) as the level of quantitation that results can be accurately reported. The lab can in certain circumstances report the LOD (Limit of Detection) as the level of detection on the reports, such as in the case of DoD analysis reporting. Note: MDLs are not applicable to all methods, examples being pH and Odor. See Admin SOP-012 for a full list.

12.1.1 AEL defines The MDL as an estimate of the minimum amount of a substance that an analytical process can readily detect. It is also defined as the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the MDL, the false positive rate (Type I error) is 1%. The MDL in the past was often calculated by performing seven QC sample replicates at a low concentration. Moving forward, MDLs run over the course of the year shall be used to calculate the MDL or be used as the MDL verification. By taking the standard deviation of the recoveries of the replicates and multiplying this SD by the appropriate factor for the number of data points (see table 1), a statistically derived MDL is generated. Also, these statistical MDLs are compared to a collection of Method Blank levels calculated to derive a Blanks MDL. The higher of these two derived MDLs shall be the MDL in use for the lab. For tests that do not allow for a calculated MDL, the MDL is often the lowest increment of measurement or a method established value. Detection Level (DL) is defined by DoD as to be equivalent to MDL

12.1.2 Limit of Detection (LOD): This is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte, prep method, clean up method, and matrix-specific and is laboratory dependent. For drinking water, it is also instrument dependent. The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%.

12.1.2.1 AEL further defines the LOD as the concentration level at which the MDL is validated. It is appropriate to report to either the MDL or LOD. For work performed under DoD (Department of Defense) and ELAP (Environmental

Laboratory Accreditation Program), reporting shall be to the LOQ or at the levels listed in the statement of work.

- 12.1.2.2 An MDL must be determined prior to determining the LOD.
- 12.1.3 Practical Quantitation Limit (PQL): Advanced Environmental Laboratories defines the PQL the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. This is most often at the concentration of the lowest level standard on the standard curve.
- 12.1.4 Limits of Quantitation (LOQ): The lowest concentration that produces a quantitative result within specified limits of precision and bias. (For DoD and ELAP projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.) The LOQ is most often at a concentration equal to the PQL but is not synonymous with the PQL. The LOQ is the concentration at which the PQL is verified. When a QC sample at the concentration level of the PQL is taken through all processing steps that a sample would be taken through and passes the LCS criteria +/- 20%, then that concentration is deemed the LOQ.
- 12.1.5 The PQL or LOQ is used as the reporting limit for reporting results. Results reported between the PQL (or LOQ) and the MDL (or LOD) shall be deemed to have greater uncertainty and will be qualified accordingly.
- 12.1.6 The relationship between the PQL and MDL is defined as "the PQL value being greater than that of the MDL value". All data reported below the PQL is to be qualified with the "I" qualifier and no data is to be reported below the MDL level.
- 12.1.7 For results where MDL studies are not applicable, such as odor, the PQL (or LOQ) stated in the method shall be used as the reporting limit.
- 12.1.8 For tests that list working ranges for the method, such as many wetchem tests (solids and CBODs), the minimum value will be utilized as the PQL. See ADMIN SOP-012 for expansion on this subject.
- 12.1.9 The MDL studies are determined according to 40 CFR 136 Appendix B, which is attached as Figure 12.1. Also reference SOP ADMIN-012 for complete instructions for how to perform MDL studies. Below is a summary only.

- 12.1.10 For new methods, where changes are made to the method, or where maintenance is performed on the instrumentation that will affect the instrument sensitivity, a seven-replicate study with the replicates taken through all the same procedures as a normal sample shall be performed. These replicates will be extracted/digested over the course of 3 days and analyzed over 3 days.
- 12.1.11 For already established methods, an MDL will be determined by first collecting MDLs performed on a quarterly basis. After every 13 months, a new MDL is calculated from those collected quarterlies. These values are then evaluated against an evaluation of the method blanks from the previous 6 months to a year. The higher of those two evaluations shall be selected as the new MDL. That new MDL is then compared to the already in use MDL. The existing MDL can be confirmed and left unchanged if the newly calculated MDL is within 50 – 200% of the established MDL if the lab desires to keep it the same. Otherwise, the new MDL will be implemented.
- 12.1.12 MDLs should then be compared with applicable regulatory levels to see if they meet those levels.
- 12.1.13 MDL levels should be reviewed to see that they are at reasonable levels. If once calculated, they are too low for normal analysis, such as they would continuously report noise or erroneously report low level false positives, then the study should be done over with a matrix more typical of real-world samples to account for this.
- 12.1.14 Frequency of MDL studies (other than mandated quarterly studies)
- 12.1.14.1 The MDL studies are repeated when the instrument undergoes maintenance that would significantly affect the performance of the instrument, and thus in turn affects the sensitivity. A change in the procedure (method change) would also require reperformance of the MDL study.
- 12.1.14.1.1 Justifiable reasons for re-determining the MDL.
- 12.1.14.1.1.1 Change the source of the GC/MS detector.
- 12.1.14.1.1.2 Change the optics on the ICP or AA.
- 12.1.14.1.1.3 Change the power tube of the ICP.
- 12.1.14.1.1.4 Install a new detector in a GC.

12.1.14.1.1.5 Any other maintenance or part replacement that would significantly alter the performance of the instrument.

12.1.14.1.2 Maintenance issues that do not justify a new MDL study.

12.1.14.1.2.1 Changing peristaltic pump tubing.

12.1.14.1.2.2 Installing a new column of the same type and dimensions in a GC.

12.1.14.1.2.3 Changing a filament in the GC/MS.

12.1.14.1.2.4 Changing a liner in the GC.

12.1.14.1.2.5 Routine preventative maintenance.

12.1.14.1.2.6 Changing the gas supply as long as it is the same purity.

12.1.15 A validation of the MDL study or a new MDL study will be performed every 13 months for every instrument, method, and matrix. Instruments performing analysis for non-potable water and soils can share combine MDL studies to report all at one MDL. For drinking water instrumentation, the MDLs must be instrument specific.

12.1.16 Some methods, such as some wet chemistry methods (those with LDR requirements), require semi-annual MDL studies.

12.1.17 Some methods, such as some drinking water Semivolatiles methods, require an MDL study with every iDOC. Those drinking water methods in Semivolatiles are EPA 504.1, 508. 524.2, 531.1, 549.2, 552.2.

12.1.18 MDLS for drinking water analytes shall be at 1/2 or less the values for the analyte MCL as listed on the FDEP Drinking Water Report Form.

12.1.19 See **AEL Admin SOP -012** for a more in-depth discussion of MDLs, LODs, PQLs, and LOQS.

12.2 Significant Figures

12.2.1 AEL uses a minimum of two significant figures for reporting all results. If calculations support three, three significant figures can

be reported if requested by client. Performance testing is to be treated as if it were a client requesting 3 significant figures.

12.2.2 The procedure explaining how results are rounded and significant figures are determined is detail in SOP ADMIN-011.

12.2.3 The LIMS (Laboratory Information Management System) that is utilized by AEL handles the final rounding and significant figure rules, so the analysts can enter more digits than will be printed on the final report, to ensure multiple rounding does not occur for the same number.

12.2.4 For all percent recoveries, AEL will report recoveries less than 100% with two significant figures and all recoveries 100% or greater with 3 significant figures.

12.2.5 AEL reports pH to 1 decimal place for all values determined by the electrode, which equates to two significant figures for values below 10.0 and 3 for those equal to or above 10.0. If pH paper is used, the results will only be reported to the applicable sensitivity of the pH paper used.

12.3 Manual integrations of chromatograms

12.3.1 See ADMIN-SOP-038 and QM Section 15 for explanation and rules concerning manual integrations. See TECH-SOP-008 and 009 along with QM section 8 for proper peak identification.

12.3.2 Manually integrating a peak is allowed, but only with proper supervision and approval.

12.3.3 The manual integration is performed consistent with how the instrument was calibrated

12.3.4 Manual integration is not used to adjust the recoveries of quality control samples or surrogates if the software performed proper integration.

12.3.5 The supervisor has the responsibility of verifying a random number of manual integrations within each data pack during the review process to ensure the manual integrations are within the guidelines of AEL policy.

12.3.6 Blatant misuse of the manual integration policy falls under the ethical violations as outlined in Section 1.0 of this manual and will be dealt with according to the policies outlined in that section.

12.3.7 There is a signed document by all organic analysts working with chromatograms stating they will abide by this manual integration policy. This document is retained in the employee file.

12.4 Data Entry, Data Review, and Data Approval in the LIMS system.

See also ADMIN-SOP-010 for Internal Data Review.

12.4.1 The LIMS has a three-step process for approving data. The steps are listed below and then explained in detail in the following subsections. Each step requires performance by a different person.

12.4.1.1 Data Entry is the first step.

12.4.1.2 Data Validation is the second step

12.4.1.3 Data Approval is the third and final step.

12.4.1.3.1 This step is where data can also be rejected if it does not meet quality requirements.

12.4.2 Data Entry into the LIMS is explained in detail in the Horizon Software Manuals

12.4.2.1 The analysts actually performing the analysis perform all data entry.

12.4.2.2 For subcontracted work to laboratories outside the AEL network, the project manager that is responsible for the project will enter the subcontracted results into the LIMS system if appropriate to do so. The entire report from the subcontracted lab will be attached to the AEL report.

12.4.2.3 Once the data is entered into the LIMS, the benchsheets containing the results are printed. The procedure for doing this is explained in SOP ADMIN-015.

12.4.3 Data Validation

12.4.3.1 The supervisor, or other analysts experienced in the analysis, is responsible for reviewing the data entry performed by the analysts.

12.4.3.2 This review is accomplished by:

12.4.3.2.1 First- the supervisor reviews the quality control to verify compliance and make sure the

analytical results are valid within the scope of the analytical SOP and the Quality system.

12.4.3.2.2 Secondly- the bench sheets that were generated from the results entered into the LIMS are verified against the raw data in the data pack to ensure there are no transcription errors.

12.4.3.3 Once these steps are complete, the supervisor (or peer reviewer) makes any necessary changes in the LIMS and validates the data.

12.4.3.4 The supervisor then passes the bench sheets on to the project manager for final review of the entire project.

12.4.3.5 The supervisors are responsible for the accuracy and technical correctness of the data and not the project managers.

12.4.4 Data Approval

12.4.4.1 The project managers complete the data approval step.

12.4.4.2 This is an overall review of the entire project and not a detailed review of the actual data itself. The goal of the final review is:

12.4.4.2.1 Compare the results of the different analyses to make sure the results agree with each other

12.4.4.2.2 Make sure there are no omissions throughout the report, such as dates and times.

12.4.4.2.3 Verify the samples are labeled correctly in accordance with the COC.

12.4.4.2.4 Make sure all appropriate qualifiers are applied and explained in the case narratives.

12.4.4.2.4.1 The case narrative process is explained in detail in SOP ADMIN-028.

12.4.4.3 The project manager is the individual who will sign the report.

12.5 Levels of Reports

12.5.1 AEL provides several levels of reports, as listed below.

12.5.1.1 Level 1. This is a report consisting simply of the analytical results and the method blanks for the associated methods.

12.5.1.1.1 This is the standard report provided by AEL to most clients. The other level reports are only provided on an as needed basis.

12.5.1.2 Level 2. This is a Level 1 report that also includes the batch quality control sample results, such as the laboratory control sample (LCS) and matrix spike (MS) and matrix spike duplicate (MSD).

12.5.1.3 Level 3. This is a Level 2 report that also includes the CLP-like forms (EPA Contract Laboratory Program). The CLP forms encompass most aspects of the analytical run, such as instrument calibration data, tune data, prep logs, analysis logs, and instrument quality control data.

12.5.1.4 Level 4. This is a Level 3 report that also includes the raw data for the analyses involved. The raw data includes the printouts from the instruments, such as chromatograms.

12.6 Report Format

12.6.1 The analytical report is designed to include all the required information listed in Section 5.10, Module 2 of the TNI 2016 Standards.

12.6.2 The report also includes all requirements of the SOP provided by Florida's Department of Environmental Protection (FDEP) in DEP-QA-002/02. This document is in the custody of the QA Officer.

12.6.3 Reporting for DoD work will require having all the elements for reporting as outlined in the DoD QSM rev 5.4.

12.7 Printing of Reports

12.7.1 The project managers print the reports.

12.7.2 The reports are printed or generated as an electronic report after the data has been approved and everything verified as complete and accurate.

12.7.2.1 The electronic copy has been designed to minimize the possibility of misunderstanding or misuse.

12.7.3 The signed report (physically or electronically) is then delivered to the client.

12.7.3.1 The methods for delivering the report to the client are facsimile, email, traditional mailing, or hand delivery.

12.7.4 A copy of the signed, completed report is kept electronically by lab in the designated "Horizon" report folders of the AEL networked servers.

12.8 Signatures on Reports

12.8.1 The signatures on reports can either be electronic or manual.

12.8.2 If the report is being delivered electronically via email, the preferred signature is the electronic version.

12.8.3 The procedure for formatting and completing an electronic signature is contained in SOP ADMIN-026. These signatures are to be secure and password protected per the instructions outlined in this SOP.

12.9 Numbering format for AEL Projects

12.9.1 Each AEL Laboratory will have its own unique system for numbering reports. The format will be consistent for all labs, but unique to the actual laboratory producing the final report.

12.9.2 The format employed will be of the following type LYY#####,

12.9.2.1 An example of a project number is J140001. The actual identifiers are explained below.

12.9.2.2 L is the Lab identifier

12.9.2.2.1 AEL Jacksonville will use a 'J'.

12.9.2.2.2 AEL Tampa will use a 'T'.

12.9.2.2.3 AEL Gainesville will use a 'G'.

12.9.2.2.4 AEL Orlando will use an 'A'

12.9.2.2.5 AEL Miami will use an 'M'

12.9.2.2.6 AEL Tallahassee will use an 'S'

12.9.2.2.7 AEL Fort Myers will use an 'F'

12.9.2.3 YY is the two-digit calendar year identifier, such as 14 for 2014, etc.

12.9.2.4 ##### is a unique sequential number that is assigned by the LIMS, such as 0001.

12.9.2.4.1 All samples associated with a given project utilize the format of the project number followed by -##, where ## equates to a sequential number for each successive sample.

12.9.2.4.1.1 An example of a project with two samples would be J140001-01 and J140001-02.

12.10 Amendments

12.10.1 If for any reason, and report needs to be amended, then the report shall be generated through LIMS using the amended reporting format. This will mark each page as amended and differentiate it from the original report.

12.10.2 All amended reports shall have attached a case narrative explaining why the report was amended. This can be due to adding an extra analyte per client request or a correction of an erroneous result. (Note: If for an erroneous result, then the case narrative and NCF should be constructed in unison.)

12.10.3 Any additions to a report shall be submitted as an amended report with the entire report resent as amended.

12.11 Holding Times and Analysis Start time

12.11.1 Holding times are defined as the time elapsed from the time of sampling to the time of extraction, digestion, or analysis, or from extraction or digestion to analysis, as appropriate.

12.11.2 For those analytes where there is no separate holding time specified for the extract, digestate or other processed sample, the holding period ends when the sample processing begins.

12.11.3 Holding times that are specified in "hours" are met if the sample processing (e.g., extraction, digestion, filtration, etc) or analysis, as applicable, begins within the last hour of the specified holding time, accounting for the time zone in which the sample was collected.

12.11.3.1 Example: For a sample with a 24-hour holding time that was collected at 14:15 on May 20, 2020, the sample is within holding time if the sample is processed before 14:15 on May 21, 2020.

12.11.4 Holding times that are specified in “days” are met if the sample processing begins before 24:00 on the final day, accounting for the time zone in which the sample was collected.

12.11.4.1 For microbiology, the holding time is evaluated as the duration between the sample collection date and time and the date and time of the placement of the processed sample into or on the applicable growth medium.

12.11.4.2 For BOD or CBOD, the holding time is evaluated as the duration between the sample collection date and time and the date and time of the initial DO measurement for the test.

12.11.5 Analysis Start time is synonymous with the start of processing the sample as listed in sections 12.11.1 thru 12.11.4. Only those analyses that are processed by batch only, such as odor, shall be listed with the start time of the batch processing. Otherwise, the start time of the analysis is that of the individual client sample.

12.12 Analytical Batches

12.12.1 AEL utilizes batches to perform the analysis for each individual method.

12.12.2 A Batch is defined as a group of samples that are prepared or analyzed together. There are two types of batches used by AEL:

12.12.2.1 Prep Batches. These are created for samples that undergo a preparation step. The prep batch consists of the group of samples prepared together and the batch quality control samples that accompany them.

12.12.2.2 QC Batches. QC batches are created for groups of samples analyzed together. This can be a prep batch for methods that require a preparation step before analysis, or it can be just a group of samples. Either way, the QC batch will contain all instrument and batch quality control.

12.12.3 Batch Quality Control is defined as the quality control samples that are required to be prepared and/or analyzed with the group of samples. The requirements and acceptance limits are outlined

by the individual analytical methods and incorporated into the SOPs. The SOPs are listed in Section 3.0.

12.12.3.1 The definitions of the batch quality control standards are explained in the SOPs and are summarized below.

12.12.3.2 Batch Quality Control can consist of any of the following:

12.12.3.2.1 Laboratory Control Sample (LCS)

12.12.3.2.2 Laboratory Control Sample Duplicate (LCSD)

12.12.3.2.3 Sample Duplicate (DUP)

12.12.3.2.4 Matrix Spike (MS)

12.12.3.2.5 Matrix Spike Duplicate (MSD)

12.12.3.2.6 Preparation or Method Blanks (PB or MB)

12.12.4 Instrument Quality Control is defined as the quality control samples that are required by the analytical methods to prove the instrument is functioning properly. The required standards and acceptance limits are listed in the individual analytical SOPs.

12.12.4.1 The definitions of the instrument quality control standards are explained in the SOPs and are summarized below.

12.12.4.2 Instrument quality control can consist of any mixture of the following:

12.12.4.2.1 Initial Calibration Verification (ICV)

12.12.4.2.2 Continuing Calibration Verification (CCV)

12.12.4.2.3 Calibration Blanks (ICB or CCB)

12.12.4.2.4 Instrument Tunes

12.12.4.2.5 Instrument Blanks (IB)

12.12.4.2.6 Serial Dilutions (SD)

12.12.4.2.7 Post-spikes (PS)

12.12.4.2.8 Interference Check Standards (ICS)

12.12.4.2.9 Calibration Standards

12.12.4.2.10 Column efficiency standards

12.12.4.2.11 Column performance standards

12.12.5 A batch is defined as a maximum of 20 unknown samples and the associated batch quality control. (Many methods require batches no greater than 10. See each analytical SOP for specific requirements.)

12.12.5.1 The time limit for batch consideration is 24 hours. This means if a batch is in progress or the preparation process has just been completed and more samples are received for that method, then the additional samples can be added to the batch in progress as long as the samples are received within 24 hours of the start of the batch – either prep or analytical.

12.12.5.2 This provision is still limited to a maximum number of 20 unknown samples.

12.12.6 If there are more than 20 samples to be analyzed for the same method, it will be broken into multiple batches.

12.12.7 If there is only one sample to be analyzed, the batch will still consist of all the required batch quality control elements.

12.13 Precision and Accuracy

12.13.1 The precision and accuracy are determined for all batch quality control samples, specifically a MS and MSD or a LCS and LCSD.

12.13.2 The accuracy refers to how well the spike was recovered at its expected concentration, and is expressed in percent recovery (%R)

12.13.2.1 Percent Recovery is determined by the following equation:

$$\%R = \frac{(\text{Spike Result} - \text{Sample Result}) (\text{mg/L}) \times 100}{\text{Spike Amount (mg/L)}}$$

Where;

Spike Result = the result determined that includes the amount of analyte spiked plus the amount of analyte in the sample.

Sample Result = the result determined that is indicative of the amount of analyte in the sample only.

Spike Amount = the concentration of spike that was added during the spike process, effectively the true value of the spike.

12.13.2.2 The acceptance limits for accuracy are defined in the analytical SOPs.

12.13.2.3 The recoveries are reported according to the significant figures listed above in Section 12.2.5.

12.13.3 The precision refers to how well the duplicate spikes or duplicate samples agree with each other and is expressed in relative percent difference (RPD).

12.13.3.1 RPD is calculated using the following equation:

$$\text{RPD} = \frac{\text{ABS}[(\text{Result 1} - \text{Result 2})] \times 100}{\text{AVG}[(\text{Result 1}, \text{Result 2})]}$$

Where;

ABS = absolute value of the difference between Result 1 and Result 2

AVG = the average of Result 1 and Result 2

12.13.3.2 AEL, as a general rule, calculates the RPD for spiked samples (LCS/LCSD or MS/MSD) using the concentrations and not the %R values.

12.13.3.2.1 The RPD can be calculated based upon the %R values if requested by the client.

12.13.3.3 The acceptance limits for precision are defined in the analytical SOPs.

12.13.4 These values are typically reported to the client only for higher levels of reports but are retained in the data pack and reviewed by the analyst and supervisor to ensure all requirements are met sufficiently or else results are qualified accordingly.

12.14 Qualifiers

- 12.14.1 The procedure explaining how to qualify data and a listing of the qualifiers are provided in SOP ADMIN-008.
- 12.14.2 AEL typically utilizes the FDEP qualifiers as described in FAC 62-160 Table 1. A listing of those qualifiers is attached as Table 12.2. For DoD work, the qualifiers listed in the DoD QSM rev 5.4 shall be used where appropriate.
- 12.14.3 It is the responsibility of the analyst and supervisor to ensure that all data is properly qualified when necessary and explain the reasons behind the qualifiers and what possible impacts it may have on the data in the text of the case narrative.
- 12.14.4 It is the responsibility of the Project Manager to verify accuracy of the qualifiers and ensure the case narratives are complete and accurate.

Figure 12.1

Electronic Code of Federal Regulations

e-CFR data is current as of February 11, 2021

Title 40 → Chapter I → Subchapter D → Part 136 → §136.6

Appendix B to Part 136—Definition and Procedure for the Determination of the Method Detection Limit—Revision 2

Definition

The method detection limit (MDL) is defined as the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results.

I. Scope and Application

(1) The MDL procedure is designed to be a straightforward technique for estimation of the detection limit for a broad variety of physical and chemical methods. The procedure requires a complete, specific, and well-defined analytical method. It is essential that all sample processing steps used by the laboratory be included in the determination of the method detection limit.

(2) The MDL procedure is *not* applicable to methods that do not produce results with a continuous distribution, such as, but not limited to, methods for whole effluent toxicity, presence/absence methods, and microbiological methods that involve counting colonies. The MDL procedure also is *not* applicable to measurements such as, but not limited to, biochemical oxygen demand, color, pH, specific conductance, many titration methods, and any method where low-level spiked samples cannot be prepared. Except as described in the addendum, for the purposes of this procedure, “spiked samples” are prepared from a clean reference matrix, such as reagent water, spiked with a known and consistent quantity of the analyte. MDL determinations using spiked samples may not be appropriate for all gravimetric methods (e.g., residue or total suspended solids), but an MDL based on method blanks can be determined in such instances.

II. Procedure

(1) Estimate the initial MDL using one or more of the following:

(a) The mean determined concentration plus three times the standard deviation of a set of method blanks.

(b) The concentration value that corresponds to an instrument signal-to-noise ratio in the range of 3 to 5.

- (c) The concentration equivalent to three times the standard deviation of replicate instrumental measurements of spiked blanks.
- (d) That region of the calibration where there is a significant change in sensitivity, *i.e.*, a break in the slope of the calibration.
- (e) Instrumental limitations.
- (f) Previously determined MDL.

NOTE: It is recognized that the experience of the analyst is important to this process. However, the analyst should include some or all of the above considerations in the initial estimate of the MDL.

(2) Determine the initial MDL.

NOTE: The Initial MDL is used when the laboratory does not have adequate data to perform the Ongoing Annual Verification specified in Section (4), typically when a new method is implemented or if a method was rarely used in the last 24 months.

(a) Select a spiking level, typically 2—10 times the estimated MDL in Section 1. Spiking levels in excess of 10 times the estimated detection limit may be required for analytes with very poor recovery (e.g., for an analyte with 10% recovery, spiked at 100 micrograms/L, with mean recovery of 10 micrograms/L; the calculated MDL may be around 3 micrograms/L. Therefore, in this example, the spiking level would be 33 times the MDL, but spiking lower may result in no recovery at all).

(b) Process a minimum of seven spiked samples and seven method blank samples through all steps of the method. The samples used for the MDL must be prepared in at least three batches on three separate calendar dates and analyzed on three separate calendar dates. (Preparation and analysis may be on the same day.) Existing data may be used, if compliant with the requirements for at least three batches, and generated within the last twenty four months. The most recent available data for method blanks and spiked samples must be used. Statistical outlier removal procedures should not be used to remove data for the initial MDL determination, since the total number of observations is small and the purpose of the MDL procedure is to capture routine method variability. However, documented instances of gross failures (e.g., instrument malfunctions, mislabeled samples, cracked vials) may be excluded from the calculations, provided that at least seven spiked samples and seven method blanks are available. (The rationale for removal of specific outliers must be documented and maintained on file with the results of the MDL determination.)

(i) If there are multiple instruments that will be assigned the same MDL, then the sample analyses must be distributed across all of the instruments.

(ii) A minimum of two spiked samples and two method blank samples prepared and analyzed on different calendar dates is required for each instrument. Each analytical batch may contain one spiked sample and one method blank sample run together. A

spiked sample and a method blank sample may be analyzed in the same batch, but are not required to be.

(iii) The same prepared extract may be analyzed on multiple instruments so long as the minimum requirement of seven preparations in at least three separate batches is maintained.

(c) Evaluate the spiking level: If any result for any individual analyte from the spiked samples does not meet the method qualitative identification criteria or does not provide a numerical result greater than zero, then repeat the spiked samples at a higher concentration. (Qualitative identification criteria are a set of rules or guidelines for establishing the identification or presence of an analyte using a measurement system. Qualitative identification does not ensure that quantitative results for the analyte can be obtained.)

(d) Make all computations as specified in the analytical method and express the final results in the method-specified reporting units.

(i) Calculate the sample standard deviation (S) of the replicate spiked sample measurements and the sample standard deviation of the replicate method blank measurements from all instruments to which the MDL will be applied.

(ii) Compute the MDL_s (the MDL based on spiked samples) as follows:

$$MDL_s = t_{(n-1, 1-\alpha = 0.99)} S_s$$

Where:

MDL_s = the method detection limit based on spiked samples

$t_{(n-1, 1-\alpha = 0.99)}$ = the Student's t-value appropriate for a single-tailed 99th percentile t statistic and a standard deviation estimate with n-1 degrees of freedom. See Addendum Table 1.

S_s = sample standard deviation of the replicate spiked sample analyses.

(iii) Compute the MDL_b (the MDL based on method blanks) as follows:

(A) If none of the method blanks give numerical results for an individual analyte, the MDL_b does not apply. A numerical result includes both positive and negative results, including results below the current MDL, but not results of "ND" (not detected) commonly observed when a peak is not present in chromatographic analysis.

(B) If some (but not all) of the method blanks for an individual analyte give numerical results, set the MDL_b equal to the highest method blank result. If more than 100 method blanks are available, set MDL_b to the level that is no less than the 99th percentile of the method blank results. For "n" method blanks where $n \geq 100$, sort the method blanks in rank order. The $(n * 0.99)$ ranked method blank result (round to the nearest whole number) is the MDL_b . For example, to find MDL_b from a set of 164 method blanks where

the highest ranked method blank results are . . . 1.5, 1.7, 1.9, 5.0, and 10, then $164 \times 0.99 = 162.36$ which rounds to the 162nd method blank result. Therefore, MDL_b is 1.9 for $n = 164$ (10 is the 164th result, 5.0 is the 163rd result, and 1.9 is the 162nd result). Alternatively, you may use spreadsheet algorithms to calculate the 99th percentile to interpolate between the ranks more precisely.

(C) If all of the method blanks for an individual analyte give numerical results, then calculate the MDL_b as:

$$MDL_b = X + t_{(n-1, 1-\alpha = 0.99)} S_b$$

Where:

MDL_b = the MDL based on method blanks

X = mean of the method blank results (use zero in place of the mean if the mean is negative)

$t_{(n-1, 1-\alpha = 0.99)}$ = the Student's t-value appropriate for the single-tailed 99th percentile t statistic and a standard deviation estimate with $n-1$ degrees of freedom. See Addendum Table 1.

S_b = sample standard deviation of the replicate method blank sample analyses.

NOTE: If 100 or more method blanks are available, as an option, MDL_b may be set to the concentration that is greater than or equal to the 99th percentile of the method blank results, as described in Section (2)(d)(iii)(B).

(e) Select the greater of MDL_s or MDL_b as the initial MDL.

(3) Ongoing Data Collection.

(a) During any quarter in which samples are being analyzed, prepare, and analyze a minimum of two spiked samples on each instrument, in separate batches, using the same spiking concentration used in Section 2. If any analytes are repeatedly not detected in the quarterly spiked sample analyses, or do not meet the qualitative identification criteria of the method (see section 2(c) of this procedure), then this is an indication that the spiking level is not high enough and should be adjusted upward. Note that it is not necessary to analyze additional method blanks together with the spiked samples, the method blank population should include all of the routine method blanks analyzed with each batch during the course of sample analysis.

(b) Ensure that at least seven spiked samples and seven method blanks are completed for the annual verification. If only one instrument is in use, a minimum of seven spikes are still required, but they may be drawn from the last two years of data collection.

(c) At least once per year, re-evaluate the spiking level.

(i) If more than 5% of the spiked samples do not return positive numerical results that meet all method qualitative identification criteria, then the spiking level must be increased, and the initial MDL re-determined following the procedure in section 2.

(ii) [Reserved]

(d) If the method is altered in a way that can be reasonably expected to change its sensitivity, then re-determine the initial MDL according to section 2, and restart the ongoing data collection.

(e) If a new instrument is added to a group of instruments whose data are being pooled to create a single MDL, analyze a minimum of two spiked replicates and two method blank replicates on the new instrument. If both method blank results are below the existing MDL, then the existing MDL_b is validated. Combine the new spiked sample results to the existing spiked sample results and recalculate the MDL_s as in Section 4. If the recalculated MDL_s does not vary by more than the factor specified in section 4(f) of this procedure, then the existing MDL_s is validated. If either of these two conditions is not met, then calculate a new MDL following the instructions in section 2.

(4) Ongoing Annual Verification.

(a) At least once every thirteen months, re-calculate MDL_s and MDL_b from the collected spiked samples and method blank results using the equations in section 2.

(b) Include data generated within the last twenty four months, but only data with the same spiking level. Only documented instances of gross failures (e.g., instrument malfunctions, mislabeled samples, cracked vials) may be excluded from the calculations. (The rationale for removal of specific outliers must be documented and maintained on file with the results of the MDL determination.) If the laboratory believes the sensitivity of the method has changed significantly, then the most recent data available may be used, maintaining compliance with the requirement for at least seven replicates in three separate batches on three separate days (see section 2b).

(c) Include the initial MDL spiked samples if the data were generated within twenty four months.

(d) Only use data associated with acceptable calibrations and batch QC. Include all routine data, with the exception of batches that are rejected and the associated samples reanalyzed. If the method has been altered in a way that can be reasonably expected to change its sensitivity, then use only data collected after the change.

(e) Ideally, use all method blank results from the last 24 months for the MDL_b calculation. The laboratory has the option to use only the last six months of method blank data or the fifty most recent method blanks, whichever criteria yields the greater number of method blanks.

(f) The verified MDL is the greater of the MDL_s or MDL_b. If the verified MDL is within 0.5 to 2.0 times the existing MDL, and fewer than 3% of the method blank results (for the

individual analyte) have numerical results above the existing MDL, then the existing MDL may optionally be left unchanged. Otherwise, adjust the MDL to the new verification MDL. (The range of 0.5 to 2.0 approximates the 95th percentile confidence interval for the initial MDL determination with six degrees of freedom.)

Addendum to Section II: Determination of the MDL for a Specific Matrix

The MDL may be determined in a specific sample matrix as well as in reagent water.

- (1) Analyze the sample matrix to determine the native (background) concentration of the analyte(s) of interest.
- (2) If the response for the native concentration is at a signal-to-noise ratio of approximately 5-20, determine the matrix-specific MDL according to Section 2 but without spiking additional analyte.
- (3) Calculate MDL_b using the method blanks, not the sample matrix.
- (4) If the signal-to-noise ratio is less than 5, then the analyte(s) should be spiked into the sample matrix to obtain a concentration that will give results with a signal-to-noise ratio of approximately 10-20.
- (5) If the analytes(s) of interest have signal-to-noise ratio(s) greater than approximately 20, then the resulting MDL is likely to be biased high.

Table 1—Single-Tailed 99th Percentile t Statistic

| Number of replicates | Degrees of freedom (n-1) | t_(n-1, 0.99) |
|-----------------------------|---------------------------------|--------------------------------|
| 7 | 6 | 3.143 |
| 8 | 7 | 2.998 |
| 9 | 8 | 2.896 |
| 10 | 9 | 2.821 |
| 11 | 10 | 2.764 |
| 12 | 11 | 2.718 |
| 13 | 12 | 2.681 |
| 14 | 13 | 2.650 |
| 15 | 14 | 2.624 |
| 16 | 15 | 2.602 |
| 17 | 16 | 2.583 |
| 18 | 17 | 2.567 |
| 19 | 18 | 2.552 |

| | | |
|------------|-----------|--------------|
| 20 | 19 | 2.539 |
| 21 | 20 | 2.528 |
| 22 | 21 | 2.518 |
| 23 | 22 | 2.508 |
| 24 | 23 | 2.500 |
| 25 | 24 | 2.492 |
| 26 | 25 | 2.485 |
| 27 | 26 | 2.479 |
| 28 | 27 | 2.473 |
| 29 | 28 | 2.467 |
| 30 | 29 | 2.462 |
| 31 | 30 | 2.457 |
| 39 | 40 | 2.423 |
| 50 | 49 | 2.405 |
| 59 | 60 | 2.390 |
| 64 | 63 | 2.387 |
| 80 | 79 | 2.374 |
| 96 | 95 | 2.366 |
| 100 | 99 | 2.365 |

III. Documentation

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units. Data and calculations used to establish the MDL must be able to be reconstructed upon request. The sample matrix used to determine the MDL must also be identified with MDL value. Document the mean spiked and recovered analyte levels with the MDL. The rationale for removal of outlier results, if any, must be documented and maintained on file with the results of the MDL determination.

[82 FR 40939, Aug. 28, 2017]

Table 12.2

DATA QUALIFIER CODES (From 62-160, Table 1)

The following qualifier codes shall be used by laboratories when reporting data values that either meet the specified descriptions outlined below or do not meet the quality control criteria of the laboratory. They are categorized on these 2 pages as Acceptable, Not Acceptable, or May Be Acceptable for compliance.

| The following codes (B,D,E,I,K,L,M,U,V,!) ARE ACCEPTABLE for use with results submitted for compliance with 62-550 and 62-555. | |
|---|---|
| SYMBOL | MEANING |
| B | Results based upon colony counts outside the acceptable range. Applies to microbiological tests and specifically to membrane filter colony counts. It is to be used if the colony count is generated from a plate in which the total number of coliform colonies is outside the method indicated ideal range. This code is not to be used if a 100 mL sample has been filtered and the colony count is less than the lower value of the ideal range. |
| D | Measurement was made in the field (i.e., in situ). This code applies to any value (except field measurements of pH, specific conductance, dissolved oxygen, temperature, total residual chlorine, transparency, turbidity or salinity) that was obtained under field conditions using approved analytical methods. If the parameter code specifies a field measurement (e.g., "Field pH"), this code is not required. |
| E | Indicates that extra samples were taken at composite stations. |
| I | The reported value is greater than or equal to the laboratory method detection limit but less than the laboratory practical quantitation limit. |
| K | Off-scale low. Actual value is known to be less than the value given. This code shall be used if the value is less than the lowest calibration standard and the calibration curve is known to be non-linear; or the value is known to be less than the reported value based on sample size, dilution. Shall not be used to report values that are less than the laboratory practical quantitation limit or laboratory method detection limit. |
| L | Off-scale high. Actual value is known to be greater than value given. To be used when the concentration of the analyte is above the acceptable level for quantitation (exceeds the linear range or highest calibration standard) and the calibration curve is known to exhibit a negative deflection. |
| M | When reporting chemical analyses: presence of material is verified but not quantified; the actual value is less than the value given. The reported value shall be the laboratory practical quantitation limit. This code shall be used if the level is too low to permit accurate quantification, but the estimated concentration is greater than or equal to the method detection limit. If the value is less than the method detection limit use "T" below. |
| U | Indicates that the compound was analyzed for but not detected. This symbol shall be used to indicate that the specified component was not detected. The value associated with the qualifier shall be the laboratory method detection limit. Unless requested by the client, less than the method detection limit values shall not be reported (see "T" below). |
| V | Indicates that the analyte was detected at or above the method detection limit in both the sample and the associated method blank and the value of 10 times the blank value |

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|---|---|
| | was equal to or greater than the associated sample value. Note: unless specified by the method, the value in the blank shall not be subtracted from associated samples. |
| ! | Data deviate from historically established concentration ranges. |
| The following codes (A,F,H,N,O,T,Z,?,*) are NOT ACCEPTABLE for use with results submitted for compliance with 62-550 and 62-555. | |
| A | Value reported is the arithmetic mean (average) of two or more determinations. This code shall be used if the reported value is the average of results for two or more discrete and separate samples. These samples shall have been processed and analyzed independently. Do not use this code if the data are the result of replicate analysis on the same sample aliquot, extract or digestate. |
| F | When reporting species: F indicates the female sex. |
| H | Value based on field kit determination; results may not be accurate. This code shall be used if a field screening test (i.e., field gas chromatograph data, immunoassay, vendor-supplied field kit, etc.) was used to generate the value and the field kit or method has not been recognized by the Department as equivalent to laboratory methods. |

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|---|---|
| N | Presumptive evidence of presence of material. This qualifier shall be used if the component has been tentatively identified based on mass spectral library search; or there is an indication that the analyte is present, but quality control requirements for confirmation were not met (i.e., presence of analyte was not confirmed by alternative procedures). |
| O | Sampled, but analysis lost or not performed. |
| T | Value reported is less than the laboratory method detection limit. The value is reported for informational purposes only and shall not be used in statistical analysis. |
| Z | Too many colonies were present for accurate counting. Historically, this condition has been reported as “too numerous to count” (TNTC). The “Z” qualifier code shall be reported when the total number of colonies of all types is more than 200 in all dilutions of the sample. When applicable to the observed test results, a numeric value for the colony count for the microorganism tested shall be estimated from the highest dilution factor (smallest sample volume) used for the test and reported with the qualifier code. |
| ? | Data are rejected and should not be used. Some or all of the quality control data for the analyte were outside criteria, and the presence or absence of the analyte cannot be determined from the data. |
| * | Not reported due to interference. |

The following codes (J,Q,R,Y) **MAY OR MAY NOT BE ACCEPTABLE** for use with results submitted for compliance with 62-550 and 62-555, depending on the parameter(s) and/or the circumstances. Results with these codes will be evaluated on a case by case basis.

| SYMBOL | MEANING |
|--------|--|
| J | Estimated value. A “J” value shall be accompanied by a detailed explanation for designating the value as estimated. Where possible, the lab shall report whether the actual value is estimated to be less than or greater than the reported value. A “J” value shall not be used as a substitute for K, L, M, T, V, or Y, however, if additional reasons exist for identifying the value as an estimate (e.g., matrix spiked failed to meet acceptance criteria), the “J” code may be added to a K, L, M, T, V, or Y. Examples of situations in which a “J” code must be reported include: instances where a quality control item associated with the reported value failed to meet the established quality control criteria (the specific failure must be identified); instances when the sample matrix interfered with the ability to make any accurate determination; instances when data are questionable because of improper laboratory or field protocols (e.g., composite sample was collected instead of a grab sample); instances when the analyte was detected at or above the method detection limit in a blank other than the method |

| | |
|---|---|
| | blank (such as calibration blank or field-generated blanks and the value of 10 times the blank value was equal to or greater than the associated sample value); or instances when the field or laboratory calibrations or calibration verifications did not meet calibration acceptance criteria. |
| Q | Sample held beyond the accepted holding time. This code shall be used if the value is derived from a sample that was prepared or analyzed after the approved holding time restrictions for sample preparation or analysis. |
| R | Significant rain in the past 48 hours. (Significant rain typically involves rain in excess of 1/2 inch within the past 48 hours.) This code shall be used when the rainfall might contribute to a lower than normal value. |
| Y | The laboratory analysis was from an improperly preserved sample. The data may not be accurate. |

13.0 Field Services

- 13.1 **AEL's sampling plan** in general consists of implementing the procedures as outlined in the Florida Department of Environmental Protection (FDEP) Standard Operating Procedures for Field Activities 2017 (effective April 2018). A copy of these SOPs is assigned to each sampling vehicle so as to have them present for reference by sampling personnel at the time of sampling. When the client requires a specific sampling plan, those plans are followed. If the client's sampling plan deviates from the FDEP SOPs or limitations during sampling prevents following FDEP SOPs, this will be discussed with the client for either correction to conform to the FDEP SOPs or if the client elects not to conform, then the deviations are noted in the final report on the case narrative and in all documents containing environmental test results. The final results may also be qualified, dependent on the nature of the deviations. These deviations shall be documented in detail at the time of sampling in any field logbooks or other documentation and at the time of login, on non-conformation forms.
- 13.2 AEL performs a limited amount of field services and courier services, as well as subcontracts out some of the field services requested.
- 13.3 All field services are completed in accordance with FDEP SOPs. A listing of the SOPs is attached to this section as Table 13.1.
- 13.4 All field services performed by or for AEL will follow these SOPs explicitly.
- 13.5 All field services personnel will read and sign a copy of the AEL's Field Department Performance Protocols, which outline conduct, attire, safety measures, PPE, rules for driving, situations for notification, accident reporting, disciplinary action for misconduct, and commitment to professionalism.
- 13.6 Copies of the SOPs are on file in the QA Officer's custody and every member of the AEL field personnel will have this documentation on hand when performing sampling.
- 13.7 Any field services that are subcontracted to an outside source will be expected to follow these procedures, and if it can be determined the procedures were not followed properly, the data will be qualified and resampling may be required, depending upon the circumstances.
- 13.8 All AEL couriers are required to follow all documentation procedures as described in AEL SOPs as well as the standard protocol outlined in Section 6.0 for Sample Acceptance in this Quality Manual. This is especially important for couriating involving sample pickups directly from the field samplers, either internal or external.

- 13.9 AEL utilizes the policy for all field methods, as outlined by FAC 62-160.300, included as Figure 13.1, for determining certification requirements and testing procedures.
- 13.10 All AEL employed field sampling service personnel will attend method update seminars provided by FDEP or TREEO to maintain current training on any revisions to the field sampling procedures or be trained by someone who has attended seminar(s). (Often referred to as train the trainer.)
- 13.11 Any AEL employed field service personnel who will perform field services requiring 40-hour OSHA training will maintain current status with that requirement.
- 13.11.1 These certificates will be retained in the Employee Training Files, located in the custody of the QA Officer or QA Deputy.
- 13.12 All AEL field measurements will be documented in a bound logbook or by electronic means, such as the portable Acer laptops. Documents are to contain all the necessary information, as listed in the FDEP SOPs mentioned above and listed in table 13.1 below. Collected data is to include those items as found on the example worksheet in Figure 13.3 and in accordance with the guidelines in Figure 13.2.
- 13.13 It is the responsibility of the field personnel to ensure all instruments and equipment are functioning properly and accurately before doing any field testing.
- 13.13.1 All maintenance performed on the equipment and instruments will be documented in either a paper maintenance logbook for field equipment or by electronic means.
- 13.13.2 All instrument calibrations performed will be documented in the field notes logbook.
- 13.13.2.1 Any instrument that does not pass its acceptance criteria for proper calibration will not be used for testing and sampling will not be completed until the instrument is functioning properly.
- 13.14 High-profile Sampling
- 13.14.1 In an effort to provide the most accurate and reliable sampling data to the clients, AEL will decline to sample for projects that are beyond the scope of services typically provided.

13.14.2 If a Project Manager or Lab Manager is contacted for a sampling project that falls into one of the categories listed below, they will contact the Sales and Marketing Director to determine if we should accept the project or refer them to someone more qualified to perform this sampling, such as a consultant.

13.14.2.1 Clean sampling for trace metals.

13.14.2.2 Underground storage tank removal.

13.14.2.3 Site Assessment to determine level of contamination.

13.14.2.4 Sampling stemming from a private or corporate complaint concerning potential contamination.

13.14.2.5 Asbestos Sampling.

13.14.2.6 Lead in Paint sampling.

13.14.2.7 Surface waters with un-determined sampling point – i.e., the scope of contamination is not yet defined.

13.14.3 By referring these types of sampling events to consultants or certified sampling personnel for the specific project being requested, AEL will provide the most accurate results to the client and will protect its assets from any potential lawsuits stemming from improper sampling due to lack of understanding, training, or knowledge.

13.14.4 AEL treats its responsibility for sampling seriously and will make every attempt to provide only accurate information to the client. Any questions stemming from or pertaining to sampling that are above the knowledge base or comfort zone of AEL will be redirected to either FDEP or a consultant for answering.

Figure 13.1

(3) Laboratory certification by the DOH ELCP is not required for the following test procedures when conducted for the purposes of drinking water compliance:

- (a) Alkalinity;
- (b) Bromide;
- (c) Calcium;
- (d) Chlorite (only at entrances to distribution systems);
- (e) Specific conductance;
- (f) Disinfectant residual (includes residual chlorine);
- (g) Orthophosphate;
- (h) pH;
- (i) Silica;
- (j) Specific ultraviolet absorbance;
- (k) Temperature;
- (l) Total organic carbon; or
- (m) Turbidity.

In cases where the Department has a specific field-testing method standard operating procedure (e.g., FT 1100 for pH), the laboratory shall follow the Department's procedures. For all other analytes, a laboratory shall only use test methods that are acceptable for drinking water compliance.

(4) Except for drinking water compliance testing (see subsection 62-160.300(3), F.A.C.), laboratories are not required to be certified by the DOH ELCP when conducting the following test procedures:

- (a) pH;
- (b) Dissolved oxygen;
- (c) Specific conductivity;
- (d) Temperature;
- (e) Total residual chlorine (including free available chlorine);
- (f) Transparency or light penetration;
- (g) Salinity;
- (h) Oxidation/reduction potential;
- (i) Turbidity (when performed at the sampling location);
- (j) Explosive gases (when monitoring for the Lower Explosive Limit);
- (k) Sulfite (when performed at the sampling location);
- (l) Sediment oxygen demand; and
- (m) Any other test with a specified holding time of fifteen minutes or less when performed at the sampling location.

However, these laboratories shall follow the applicable standard operating procedures in DEP-SOP-001/01 (January 1, 2002) when conducting the analyses specified in paragraphs 62-160.300(4)(a) through (m), F.A.C.

Figure 13.2

Documentation

Field events shall be documented and shall include the following items as appropriate for the interpretation of test results. When necessary, this information shall be provided to the data user:

- a) Sampling/field measurement organization, including address, phone number, and email address
- b) Printed name and signature of technician, plus names of all members of the sampling team
- c) Sample type (grab, composite, etc.), including an identification of the matrix sampled; (aqueous, solids, etc.)
- d) Sample identification number including a unique field identification code for each sample Container
- e) Reason for sampling/measurement
- f) Date and time of sampling/measurement
- g) Location of sampling, including any diagrams, sketches, or photographs; name of sampling station, and/or latitude, longitude, and altitude when sample point is not otherwise identified
- h) For water sampling: the water level measure, sample depth, and water discharge rate measure if appropriate/required
- i) Reference to the sampling plan and procedures used, including field blanks, spikes, duplicates, and if applicable, any confirmation samples; field instrument calibration, span, drift, and calibration standards; sampling system bias and response time; and field test standards and reagents as required by the standard/test method
- j) Sample preservation, transportation, and storage, including a description of sample containers and chain of custody
- k) Details of any conditions during sampling that may affect the interpretation of the test results
- l) Any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned
- m) The organization collecting samples shall certify that samples and field measurements were collected in accordance with FDEP SOPs or provide reasons and/or justification if they were not.

Table 13.1

| Series | Description |
|---------|---------------------------------|
| FA 1000 | Administrative |
| FC 1000 | Field Decontamination |
| FD 1000 | Documentation |
| FM 1000 | Field Mobilization |
| FQ 1000 | Quality Control |
| FS 1000 | General Sampling |
| FS 2000 | General Water Sampling |
| FS 2100 | Surface Water Sampling |
| FS 2200 | Groundwater Sampling |
| FS 2300 | Drinking Water Sampling |
| FS 2400 | Wastewater Sampling |
| FS 3000 | Soil Sampling |
| FS 4000 | Sediment Sampling |
| FS 5000 | Waste Sampling |
| FS 6000 | Tissue Sampling |
| FS 7000 | Biological Communities |
| FS 8100 | Contaminated Surfaces Sampling |
| FS 8200 | Clean Sampling for Trace Metals |
| FT 1000 | Field Testing General |
| FT 1100 | Field pH |
| FT 1200 | Field Conductance |
| FT 1300 | Field Salinity |
| FT 1400 | Field Temperature |
| FT 1500 | Field Dissolved Oxygen |
| FT 1600 | Field Turbidity |
| FT 1700 | Field Light Penetration |
| FT 1800 | Field Flowmeters |
| FT 1900 | Field Continuous Monitoring |
| FT 2000 | Field Chlorine |
| FT 2100 | Field Oxidation-Reduction |
| FT 2200 | Field Sulfite |
| FT 2300 | Field Sediment Oxygen Demand |
| FT 2400 | Field Gases |
| FT 3000 | Habitat Sampling |
| LD 1000 | Laboratory Documentation |
| LQ 1000 | Laboratory Quality Control |
| LT 7000 | Biological Indices |

Table 14 Methods, Tests, Calibration Points and Frequency

14.0 Methods, Tests, Calibration Points and Frequency

14.1 The attached Table 14.1 lists the methods, test names, number of calibration points used and the frequency of the calibration used by AEL.

14.2 The number of calibration points meets or exceeds all the requirements listed in the applicable analytical methods or NELAC/TNI standards and are fully defined in the analytical SOPs.

14.2.1 Any method that does not have the number of points clearly defined by the method, AEL uses sound analytical judgment in determining the number of points required to produce quality analytical data.

14.3 See also Section 8.0 for further calibration instructions.

| Lab | Matrix | Method | Test Name | # of AEL Calibration | Method Calibration | Type of Regression | Calibration Frequency |
|---------|----------------|------------------|---|----------------------|--------------------|--------------------|-----------------------|
| AEL Jax | Drinking Water | SM5710B/E524.2 | THM Formation Potential | 5 | 3 | linear/non-linear | 6 months or QC fails |
| AEL Jax | Drinking Water | E110.2/SM2120B | Color | 12 | 12 | N/A | N/A |
| AEL Jax | Drinking Water | E120.1/SM2510B | Conductivity | 3 | 5 | N/A | Daily |
| AEL Jax | Drinking Water | E150.1/SM4500H+B | pH | 3 | 2 | N/A | Daily |
| AEL Jax | Drinking Water | E160.1/SM2540C | TDS | N/A | N/A | N/A | N/A |
| AEL Jax | Drinking Water | E180.1 | Turbidity | 6 | Undefined | Linear 0.995 or > | Daily |
| AEL Jax | Drinking Water | E200.7 | Metals by ICP-AES | 2 | 2 | Linear 1.0 | Daily |
| AEL Jax | Drinking Water | E200.8 | Metals by ICP/MS | 2 | 2 | Linear 1.0 | Daily |
| AEL Jax | Drinking Water | E245.1 | Mercury by CVAA | 5 | 5 | Linear 0.995 or > | Daily |
| AEL Jax | Drinking Water | E1631-E | Low Level Mercury | 6 | 6 | Linear 0.995 or > | 6 months or QC fails |
| AEL Jax | Drinking Water | E310.1/SM2320B | Alkalinity | N/A | N/A | N/A | N/A |
| AEL Jax | Drinking Water | E415.1/SM5310B | TOC | 6 | N/A | linear | 6 months or QC fails |
| AEL Jax | Drinking Water | E504.1 | EDB and DBCP by GC/ECD | 6 | 5 | 2nd order | 6 months or QC fails |
| AEL Jax | Drinking Water | E508 | Drinking Water Pesticides by GC/ECD | 5 | 5 | Not specified | 6 months or QC fails |
| AEL Jax | Drinking Water | E515.3 | Drinking Water Herbicides by GC/ECD | 5 | 5 | 2nd order | 6 months or QC fails |
| AEL Jax | Drinking Water | E522 | Drinking Water by GC/MS SIMs 1,4 Dioxane | 5 | 5 | linear | 6 months or QC fails |
| AEL Jax | Drinking Water | E524.2 | Drinking Water VOCs by GC/MS | 7 | 5 | linear | 6 months or QC fails |
| AEL Jax | Drinking Water | E525.2 | Drinking Water SOCs by GC/MS | 5 | 5 | rsd <30% | 6 months or QC fails |
| AEL Jax | Drinking Water | E531.1 | Carbamates by HPLC | 5 | 5 | 2nd order | 6 months or QC fails |
| AEL Jax | Drinking Water | E533 | PFAS by LC/LC/MS | 9 | 5 | Quadratic | 6 months or QC fails |
| AEL Jax | Drinking Water | E547 | Glyphosate by HPLC | 3 | 3 | Not specified | 6 months or QC fails |
| AEL Jax | Drinking Water | E548.1 | Endothall by GC/MS | 4 | 4 | rsd <30% | 6 months or QC fails |
| AEL Jax | Drinking Water | E549.2 | Diquat by HPLC | 3 | 3 | Not specified | 6 months or QC fails |
| AEL Jax | Drinking Water | E552.2 | Haloacetic Acids by GC/ECD | 6 | 5 | 2nd order | 6 months or QC fails |
| AEL Jax | Drinking Water | SM5710B/E552.2 | Haloacetic Acids Formation Potential | 6 | 5 | 2nd order | 6 months or QC fails |
| AEL Jax | Drinking Water | SM2150B | Odor | N/A | N/A | N/A | N/A |
| AEL Jax | Drinking Water | SM2340B | Hardness by Calculation | N/A | N/A | N/A | N/A |
| AEL Jax | Drinking Water | E300.0 | Anions (CL,F,NO2,NO3,NO2+NO3,OP,SO4)by IC | 5 | 3 | 0.995 | 6 months or QC fails |
| AEL Jax | Drinking Water | SM4500-P-E | Anion OP by Spectrophotometer | 5 | 3 | 0.995 | 6 months or QC fails |
| AEL Jax | Drinking Water | SM9215B | HPC | N/A | N/A | N/A | N/A |

Table 14 Methods, Tests, Calibration Points and Frequency

| Lab | Matrix | Method | Test Name | # of AEL Calibration | Method Calibration | Type of Regression | Calibration Frequency |
|---------|----------------|--------------------------------|---|----------------------|--------------------|--------------------|-----------------------|
| AEL Jax | Drinking Water | SM9221F | Escherichia coli (Confirmation)by Tube Fermentation | N/A | N/A | N/A | N/A |
| AEL Jax | Drinking Water | SM9222B | Total Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Jax | Drinking Water | SM9223B | Total Coliform by MMO-MUG | N/A | N/A | N/A | N/A |
| AEL Jax | Drinking Water | SM9223B | Escherichia coli by MMO-MUG | N/A | N/A | N/A | N/A |
| AEL Jax | Soil | FL-PRO | Total Recoverable Petroleum Hydrocarbons | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | AEL SOP SVOC-022 | Diesel Range Organics | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | TPHCWG Direct Method | Total Recoverable Petroleum Hydrocarbons | 5 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | SM2540G | Total Solids in Soil | N/A | N/A | N/A | N/A |
| AEL Jax | Soil | SW1020 and SW1030 | Flashpoint/Ignitability | N/A | N/A | N/A | Daily |
| AEL Jax | Soil | SW6010B | Metals by ICP-AES | 2 | 2 | Linear 1.0 | Daily |
| AEL Jax | Soil | SW6020 | Metals by ICP/MS | 2 | 2 | Linear 1.0 | Daily |
| AEL Jax | Soil | SW7471A | Mercury by CVAA | 5 | 6 | Linear 0.995 or > | Daily |
| AEL Jax | Soil | SW8015B | Volatiles Organics by GC/FID | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | AEL SOP VOC-009 | Volatiles Organics by GC/FID | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | SW8081A&B | Organochlorine Pesticides by GC/ECD | 6 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | SW8082A | Aroclors by GC/ECD | 5 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | SW8260B&C | Volatile Organics by GC/MS | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | AEL SOP VOC-003 | Volatile Organics by GC/MS | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | SW8270C&D | Semi-Volatile Orgniacs by GC/MS | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | AEL SOP SVOC-006 | Semi-Volatile Orgniacs by GC/MS | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | SW8141 & 8141B | Organophosphorus Compounds by GC/NPD | 5 | 5 | 2nd order per 8000 | Daily |
| AEL Jax | Soil | SW8151A | Chlorinated Herbicides by GC/ECD | 5 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | SW8270C-SIM | PAHs by GC/MS | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | E9056 | Anions (CL,F,NO2,NO3,NO2+NO3,OP,SO4)by IC | 5 | 3 | 0.995 | 6 months or QC fails |
| AEL Jax | Soil | SW9040B | pH | 3 | 2 | N/A | Daily |
| AEL Jax | Soil | SW9045C | pH | 3 | 2 | N/A | Daily |
| AEL Jax | Soil | E9060A | TOC | 6 | N/A | linear | 6 months or QC fails |
| AEL Jax | Soil | SM2540G | Total Residue (%Solids) | N/A | N/A | N/A | N/A |
| AEL Jax | Soil | SW9095A | Paint Filter | N/A | N/A | N/A | N/A |
| AEL Jax | Soil | SW1311 | TCLP Extraction | N/A | N/A | N/A | N/A |
| AEL Jax | Soil | SW1312 | SPLP Extraction | N/A | N/A | N/A | N/A |
| AEL Jax | Soil | 8015C | Gasoline Range Organics (GRO) | 6 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | MADEP-EPH (MA-EPH) | EPH | 6 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | MADEP-EPH (MA-VPH) | VPH | 6 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Soil | E9023 | EOX | N/A | N/A | N/A | N/A |
| AEL Jax | Soil | E533 Mod. , DoD QSM Table B-15 | PFAS by LC/LC/MS | 9 | 5 | Quadratic | 6 months or QC fails |
| AEL Jax | Water | SM5710B/E502.2 | THM Formation Potential | 5 | 3 | linear/non-linear | 6 months or QC fails |
| AEL Jax | Water | E110.2/SM2120B | Color | N/A | N/A | N/A | N/A |
| AEL Jax | Water | E150.1/SM4500H+B | pH | 3 | N/A | N/A | Daily |
| AEL Jax | Water | E160.1/SM2540C | TDS | N/A | N/A | N/A | N/A |

Table 14 Methods, Tests, Calibration Points and Frequency

| Lab | Matrix | Method | Test Name | # of AEL Calibration | Method Calibration | Type of Regression | Calibration Frequency |
|---------|--------|------------------------|---|----------------------|---------------------|--------------------|-----------------------|
| AEL Jax | Water | E160.2/SM2540D | TSS | N/A | N/A | N/A | N/A |
| AEL Jax | Water | E160.3/SM2540B/G | Total Solids | N/A | N/A | N/A | N/A |
| AEL Jax | Water | E160.5/SM2540F | Total Settleable Solids | N/A | N/A | N/A | N/A |
| AEL Jax | Water | E1664A/B-SM5520B | Oil and Grease | N/A | N/A | N/A | N/A |
| AEL Jax | Water | E180.1/SM2130B | Turbidity | 6 | N/A | Linear 0.995 or > | Daily |
| AEL Jax | Water | SM2520B | Salinity | High and Low | N/A | N/A | Daily |
| AEL Jax | Water | E200.7 | Metals By ICP-AES | 2 | 2 | Linear 1.0 | Daily |
| AEL Jax | Water | E200.8 | Metals By ICP/MS | 2 | 2 | Linear 1.0 | Daily |
| AEL Jax | Water | E245.1 | Mercury by CVAA | 5 | 6 | Linear 0.995 or > | Daily |
| AEL Jax | Water | E1631-E | Low Level Mercury | 5 | 5 | Linear 0.995 or > | 6 months or QC fails |
| AEL Jax | Water | E310.1 | Alkalinity | N/A | N/A | N/A | N/A |
| AEL Jax | Water | E405.1 | BOD | N/A | N/A | N/A | Daily |
| AEL Jax | Water | E410.4 | COD | 6 | 9 | Linear 0.995 or > | 6 months or QC fails |
| AEL Jax | Water | E410.1 | COD | NA | N/A | N/A | NA |
| AEL Jax | Water | E415.1/SM5310B | TOC | 6 | N/A | linear | 6 months or QC fails |
| AEL Jax | Water | E602 | Volatile Aromatics by GC/PID | 7 | 5 | linear/non-linear | 6 months or QC fails |
| AEL Jax | Water | E608 | Organochlorine Pesticides by GC/ECD | 7 | 3 | Not specified | 6 months or QC fails |
| AEL Jax | Water | E608.2 | Organochlorine Pesticides by GC/ECD | 7 | 3 | Not specified | 6 months or QC fails |
| AEL Jax | Water | E624 | Volatile Organics by GC/MS | 7 | 5 | linear | 6 months or QC fails |
| AEL Jax | Water | E625 | Semi-volatile Organics by GC/MS | 7 | 3 | Not specified | 6 months or QC fails |
| AEL Jax | Water | FL-PRO | Total Recoverable Petroleum Hydrocarbons | 7 | 5 | 2nd order | 6 months or QC fails |
| AEL Jax | Water | AEL SOP SVOC-022 | DRO by FloPro Modified | 7 | 5 | 2nd order | 6 months or QC fails |
| AEL Jax | Water | SM4500-P-F | Ortho-phosphates | 6 | 6 | Linear 0.995 or > | Daily |
| AEL Jax | Water | E300.0/9056 | Anions (CL,F,NO2,NO3,NO2+NO3,OP,SO4)by IC | 5 | 3 | 0.995 | 6 months or QC fails |
| AEL Jax | Water | SM2320B | Alkalinity | N/A | N/A | N/A | N/A |
| AEL Jax | Water | SM2340B | Hardness by Calculation | N/A | N/A | N/A | N/A |
| AEL Jax | Water | SM2540G | Total Solids | N/A | N/A | N/A | N/A |
| AEL Jax | Water | SM3500CrD/EPA 7196 | Hexavalent Chromium | 4 | Method Not Specific | Linear 0.995 or > | 6 months or QC fails |
| AEL Jax | Water | SM4500CI-G | Chlorine | N/A | N/A | N/A | N/A |
| AEL Jax | Water | SM4500O-G | Dissolved Oxygen | N/A | N/A | N/A | Daily |
| AEL Jax | Water | SM5210B | CBOD | N/A | N/A | N/A | Daily |
| AEL Jax | Water | SM5210B | BOD | N/A | N/A | N/A | Daily |
| AEL Jax | Water | SM9215B | HPC | N/A | N/A | N/A | N/A |
| AEL Jax | Water | SM9222B | Total Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Jax | Water | SM9222D | Fecal Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Jax | Water | E1600 | Enterococci by MF | N/A | N/A | N/A | N/A |
| AEL Jax | Water | Colilert 18-Quantitray | Fecal | N/A | N/A | N/A | N/A |
| AEL Jax | Water | SM9223B Quantitray | Total Coliform and E. Coli | N/A | N/A | N/A | N/A |
| AEL Jax | Water | Enterolert | Enterococci by Quanti-tray | N/A | N/A | N/A | N/A |
| AEL Jax | Water | E1603 | E. Coli by MF | N/A | N/A | N/A | N/A |

Table 14 Methods, Tests, Calibration Points and Frequency

| Lab | Matrix | Method | Test Name | # of AEL Calibration | Method Calibration | Type of Regression | Calibration Frequency |
|------------|----------------|--------------------------------|---|----------------------|--------------------|--------------------|-----------------------|
| AEL Jax | Water | SM9223B | Total Coliform and E. coli by MMO-MUG | N/A | N/A | N/A | N/A |
| AEL Jax | Water | SW1010 | Flashpoint/Ignitability | N/A | N/A | N/A | Daily |
| AEL Jax | Water | SW6010B | Metals by ICP-AES | 2 | 2 | Linear 1.0 | Daily |
| AEL Jax | Water | SW6020 | Metals by ICP/MS | 2 | 2 | Linear 1.0 | Daily |
| AEL Jax | Water | SW8015B | Volatile Organics by GC/FID | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Water | AEL SOP VOC-009 | Volatile Organics by GC/FID | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Water | SW8021B | Volatile Organics by GC/PID | 7 | 5 | linear/non-linear | 6 months or QC fails |
| AEL Jax | Water | SW8011 | EDB/ DBCP by GC/ECD | 6 | 5 | 2nd order | 6 months or QC fails |
| AEL Jax | Water | SW8081A&B | Organochlorine Pesticides by GC/ECD | 6 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Water | SW8082A | Aroclors by GC/ECD | 5 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Water | SW8141 & 8141B | Organophosphorus Compounds by GC/NPD | 5 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Water | SW8151A | Chlorinated Herbicides by GC/ECD | 5 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Water | SW8260B&C | Volatile Organics by GC/MS | 7 | 5 | linear | 6 months or QC fails |
| AEL Jax | Water | AEL SOP VOC-003 | Volatile Organics by GC/MS | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Water | SW8270C&D | Semi-Volatile Organics by GC/MS | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Water | AEL SOP SVOC-006 | Semi-Volatile Organics by GC/MS | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Water | SW8270C-SIM | PAHs by GC/MS | 7 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Water | SW9040B | pH (corrosivity) | 3 | 2 | N/A | Daily |
| AEL Jax | Water | E120.1 | Conductivity | 5 | Undefined | N/A | Daily |
| AEL Jax | Water | SW9050A/SM2510B | Conductivity | 5 | Undefined | N/A | Daily |
| AEL Jax | Water | SW9095A | Paint Filter | N/A | N/A | N/A | N/A |
| AEL Jax | Water | SM2710B | S.O.U.R. by calculation | N/A | N/A | N/A | N/A |
| AEL Jax | Water | TKN-NH3 | Organic Nitrogen by Calculation | N/A | N/A | N/A | N/A |
| AEL Jax | Water | SM2330B | Corrosivity (langlier index) | N/A | N/A | N/A | N/A |
| AEL Jax | Water | SM2340B | Hardness by Calculation | N/A | N/A | N/A | N/A |
| AEL Jax | Water | 8015C | Gasoline Range Organics (GRO) | 6 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Water | MADEP-EPH (MA-EPH) | EPH | 6 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Water | MADEP-EPH (MA-VPH) | VPH | 6 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Water | RSK-175 | Headspace Analysis | 6 | 5 | 2nd order per 8000 | 6 months or QC fails |
| AEL Jax | Water | SW1020 | Flashpoint/Ignitability | N/A | N/A | N/A | Daily |
| AEL Jax | Water | E9020B | TOX | N/A | N/A | N/A | N/A |
| AEL Jax | Water | E1650C | AOX | N/A | N/A | N/A | N/A |
| AEL Jax | Water | E533 Mod. , DoD QSM Table B-15 | PFAS by LC/LC/MS | 9 | 5 | Quadratic | 6 months or QC fails |
| AEL Jax | Water | SM4500SD | Sulfide | 7 | 7 | Linear | 6 months or QC fails |
| AEL Gville | Drinking Water | E300.0 | Anions (CL,F,NO2,NO3,NO2+NO3,OP,SO4)by IC | 5 | 3 | 0.995 | 6 months or QC fails |
| AEL Gville | Drinking Water | SM2120B | Color | N/A | N/A | N/A | N/A |
| AEL Gville | Drinking Water | E353.2/SM4500-NO3-F | Nitrite/Nitrate | 6 | 4 | NA | 6 months or QC fails |
| AEL Gville | Drinking Water | E335.2/SM4500CN-E | Cyanide | 6 | 6 | 0.995 | Daily |
| AEL Gville | Drinking Water | SM9223B | Total Coliform and E.coli by MMO-MUG | N/A | N/A | N/A | N/A |
| AEL Gville | Drinking Water | SM9223B/Q-Tray | Total Coliform and E.coli by Quanti-Tray | N/A | N/A | N/A | N/A |

Table 14 Methods, Tests, Calibration Points and Frequency

| Lab | Matrix | Method | Test Name | # of AEL Calibration | Method Calibration | Type of Regression | Calibration Frequency |
|------------|----------------|---------------------|---|----------------------|--------------------|--------------------|-----------------------|
| AEL Gville | Drinking Water | SIMPLATE | Heterotrophic Plate Count (HPC) | N/A | N/A | N/A | N/A |
| AEL Gville | Drinking Water | SM2150B | Odor | N/A | N/A | N/A | N/A |
| AEL Gville | Drinking Water | E150.1/SM4500H+B | pH | 3 | NA | NA | Daily |
| AEL Gville | Drinking Water | E425.1/SM5540C | Surfactants | 10 | 10 | Linear | 6 months or QC fails |
| AEL Gville | Drinking Water | E160.1/SM2540C | TDS | N/A | NA | NA | N/A |
| AEL Gville | Drinking Water | E415.1/SM5310B | TOC | 5 | N/A | 0.995 | Weekly |
| AEL Gville | Soil | SM9221E | Fecal Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Gville | Soil | E9040 | pH | 3 | 3 | N/A | Daily |
| AEL Gville | Soil | E9045 | pH | 3 | 3 | N/A | Daily |
| AEL Gville | Soil | E9010/9014 | Cyanide | 7 | 6 | 0.995 | 6 months or QC fails |
| AEL Gville | Soil | E160.3/SM2540G | Total Solids | N/A | N/A | N/A | N/A |
| AEL Gville | Water | E310.1/SM2320B | Alkalinity | 2 | N/A | N/A | Daily |
| AEL Gville | Water | E 350.1 | Ammonia | 7 | N/A | 0.995 | Daily |
| AEL Gville | Water | E300.0 | Anions (CL,F,NO2,NO3,NO2+NO3,OP,SO4)by IC | 5 | 3 | 0.995 | 6 months or QC fails |
| AEL Gville | Water | E405.1/SM 5210B | BOD | N/A | N/A | N/A | N/A |
| AEL Gville | Water | SM5210B | CBOD | N/A | N/A | N/A | N/A |
| AEL Gville | Water | SM 10200 H | Chlorophylls | N/A | N/A | N/A | N/A |
| AEL Gville | Water | E410.4 | COD | 7 | 7 | 0.995 | 6 months or QC fails |
| AEL Gville | Water | E110.2/SM2120B | Color | N/A | N/A | N/A | N/A |
| AEL Gville | Water | SM2120C | Color | N/A | N/A | N/A | N/A |
| AEL Gville | Water | E120.1 | Conductivity | 2 | N/A | N/A | Daily |
| AEL Gville | Water | E335.2/SM4500CN-E | Cyanide | 6 | 6 | 0.995 | Daily |
| AEL Gville | Water | E9010/9014 | Cyanide (Total) | 6 | 6 | 0.995 | Daily |
| AEL Gville | Water | SM9223B/Q-Tray | E.coli | N/A | N/A | N/A | N/A |
| AEL Gville | Water | SM9221E | Fecal Coliform by MPN | N/A | N/A | N/A | N/A |
| AEL Gville | Water | Colilert-18 | Fecal Coliform by Quanti-Tray | N/A | N/A | N/A | N/A |
| AEL Gville | Water | SM 3500Cr B | Hexavalent Chromium | 8 | N/A | N/A | Daily |
| AEL Gville | Water | SIMPLATE | Heterotrophic Plate Count (HPC) | N/A | N/A | N/A | N/A |
| AEL Gville | Water | E353.2/SM4500-NO3-F | Nitrite/Nitrate | 6 | 4 | NA | 6 months or QC fails |
| AEL Gville | Water | TKN - Ammonia | Organic Nitrogen | N/A | N/A | N/A | N/A |
| AEL Gville | Water | SM4500P-E | Ortho-Phosphate | 9 | NA | NA | 6 months or QC fails |
| AEL Gville | Water | E150.1/SM4500H+B | pH | 3 | N/A | N/A | Daily |
| AEL Gville | Water | SM 2520B | Salinity | 7 | N/A | 0.995 | Daily |
| AEL Gville | Water | E425.1/SM5540C | Surfactants (MBAS) | 10 | 10 | Linear | 6 months or QC fails |
| AEL Gville | Water | E160.1/SM2540C | TDS | N/A | N/A | N/A | N/A |
| AEL Gville | Water | E351.2 | TKN | 6 | 3 | 0.995 | Daily |
| AEL Gville | Water | E415.1/SM5310B | TOC | 5 | N/A | 0.995 | Weekly |
| AEL Gville | Water | SM9223B/Q-Tray | Total Coliform by Quanti-Tray | N/A | N/A | N/A | N/A |
| AEL Gville | Water | TKN + Nox | Total Nitrogen | N/A | N/A | N/A | N/A |
| AEL Gville | Water | EPA 365.1 | Total Phosphorus | 6 | 3 | 0.995 | Daily |

Table 14 Methods, Tests, Calibration Points and Frequency

| Lab | Matrix | Method | Test Name | # of AEL Calibration | Method Calibration | Type of Regression | Calibration Frequency |
|-------------|----------------|------------------|---|----------------------|--------------------|------------------------------|-----------------------|
| AEL Gville | Water | EPA 365.3/365.4 | Total Phosphorus | 6 | 3 | 0.995 | Daily |
| AEL Gville | Water | E160.3/SM 2540B | Total Solids | N/A | N/A | N/A | N/A |
| AEL Gville | Water | E160.2/SM 2540D | TSS | N/A | N/A | N/A | N/A |
| AEL Gville | Water | E180.1 | Turbidity | 4 | 4 | 0.995 | Daily |
| AEL Gville | Water | DEP SOP 10/3/83 | Unionized Ammonia | N/A | N/A | N/A | N/A |
| AEL Gville | Water | E160.4/SM 2540E | Volatile Solids | N/A | N/A | N/A | N/A |
| AEL Orlando | Drinking Water | E110.2/SM2120B | Color | N/A | N/A | N/A | N/A |
| AEL Orlando | Drinking Water | SM2510B | Specific Conductivity | 3 | 3 | N/A | Daily |
| AEL Orlando | Drinking Water | E140.1/SM2150B | Odor | N/A | N/A | N/A | N/A |
| AEL Orlando | Drinking Water | E150.1/SM4500H+B | pH | 3 | 3 | N/A | Daily |
| AEL Orlando | Drinking Water | E300.0 | Anions (CL,F,NO2,NO3,NO2+NO3,OP,SO4)by IC | 5 | 3 | 0.995 | 6 months or QC fails |
| AEL Orlando | Drinking Water | SM9215B | HPC | N/A | N/A | N/A | N/A |
| AEL Orlando | Drinking Water | SM9223B | Escherichia coli by MMO-MUG | N/A | N/A | N/A | N/A |
| AEL Orlando | Drinking Water | SM9221F | Escherichia coli (Confirmation)by Tube Fermentation | N/A | N/A | N/A | N/A |
| AEL Orlando | Drinking Water | SM9222B | Total Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Orlando | Drinking Water | SM2540C | TDS | N/A | N/A | N/A | N/A |
| AEL Orlando | Drinking Water | SM9223B | Total Coliform by MMO-MUG | N/A | N/A | N/A | N/A |
| AEL Orlando | Water | E150.1/E9040 | pH | 3 | 2 | N/A | Daily |
| AEL Orlando | Water | SM4500H+B | pH | 3 | 2 | N/A | Daily |
| AEL Orlando | Water | E300.0 | Anions (CL,F,NO2,NO3,NO2+NO3,OP,SO4)by IC | 5 | 3 | 0.995 | 6 months or QC fails |
| AEL Orlando | Water | E405.1 | BOD | 1 | N/A | N/A | Daily |
| AEL Orlando | Water | SM2510B | Specific Conductivity | 3 | 3 | N/A | Daily |
| AEL Orlando | Water | SM2540D | TSS | N/A | N/A | N/A | N/A |
| AEL Orlando | Water | SM5210B | BOD | 1 | N/A | N/A | Daily |
| AEL Orlando | Water | SM5210B | CBOD | 1 | N/A | N/A | Daily |
| AEL Orlando | Water | SM9222B | Total Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Orlando | Water | SM9223B/Q-Tray | E.coli | N/A | N/A | N/A | N/A |
| AEL Orlando | Water | Colilert-18 | Fecal Coliform by Quanti-Tray | N/A | N/A | N/A | N/A |
| AEL Orlando | Water | SM2540C | TDS | N/A | N/A | N/A | N/A |
| AEL Tampa | Drinking Water | E110.2/SM2120B/C | Color | 9 | 9 | linear/non-linear | Annually or QC fails |
| AEL Tampa | Drinking Water | SM2510B | Specific Conductivity | 3 | 3 | N/A | Daily |
| AEL Tampa | Drinking Water | SM2340B | Hardness by Calculation | N/A | N/A | N/A | N/A |
| AEL Tampa | Drinking Water | SM2340C | Total Hardness (Titration) | N/A | N/A | N/A | N/A |
| AEL Tampa | Drinking Water | E150.1/SM4500H+B | pH | 3 | 3 | N/A | Daily |
| AEL Tampa | Drinking Water | E160.1/SM2540C | TDS | N/A | N/A | N/A | N/A |
| AEL Tampa | Drinking Water | E180.1 | Turbidity | 5 | 5 | Instrument sets P/F criteria | 6 months or QC fails |
| AEL Tampa | Drinking Water | E300.0 | Anions by IC | 7 | 6 | Linear | 6 months or QC fails |
| AEL Tampa | Drinking Water | E300.1 | Anions by IC | 7 | 6 | Linear | 6 months or QC fails |
| AEL Tampa | Drinking Water | SM2310B | Acidity | N/A | N/A | NA | N/A |
| AEL Tampa | Drinking Water | SM2320B | Alkalinity | N/A | N/A | NA | N/A |

Table 14 Methods, Tests, Calibration Points and Frequency

| Lab | Matrix | Method | Test Name | # of AEL Calibration | Method Calibration | Type of Regression | Calibration Frequency |
|-----------|----------------|----------------|---|----------------------|--------------------|--------------------|-----------------------|
| AEL Tampa | Drinking Water | E350.1 | Ammonia | 10 | 6 | 1st Order | 6 months or QC fails |
| AEL Tampa | Drinking Water | E365.1 | Ortho-phosphates | 7 | 6 | 2nd Order | 6 months or QC fails |
| AEL Tampa | Drinking Water | E365.4 | Total Phosphorus | 8 | 6 | 3rd Order | 6 months or QC fails |
| AEL Tampa | Drinking Water | E375.4/E300.0 | Sulfate | 8 | 7 | Linear | 6 months or QC fails |
| AEL Tampa | Drinking Water | SM4500SO4-E | Sulfate | 8 | 7 | Linear | 6 months or QC fails |
| AEL Tampa | Drinking Water | SM5310B | Dissolved Organic Carbon | 12 (3 Ranges) | 12 (3 Ranges) | NA | 6 months or QC fails |
| AEL Tampa | Drinking Water | SM5310B | Total Organic Carbon | 12 (3 Ranges) | 12 (3 Ranges) | Linear | 6 months or QC fails |
| AEL Tampa | Drinking Water | E425.1/SM5540C | MBAS | 10 | 10 | Linear | 6 months or QC fails |
| AEL Tampa | Drinking Water | SM2150B | Odor | N/A | N/A | N/A | N/A |
| AEL Tampa | Drinking Water | SM4500CL-E | Chloride | 9 | 6 | 3rd Order | 6 months or QC fails |
| AEL Tampa | Drinking Water | SM4500CI-G | Chlorine | NONE | NONE | NA | N/A |
| AEL Tampa | Drinking Water | SM4500CN-E | Cyanide | 7 | 6 | Linear | 6 months or QC fails |
| AEL Tampa | Drinking Water | SM4500CN-G | Cyanide, Amenable | 7 | 6 | Linear | 6 months or QC fails |
| AEL Tampa | Drinking Water | SM4500F-C | Fluoride | 4 | 5 | Linear | Daily |
| AEL Tampa | Drinking Water | SM4500O-G | Dissolved Oxygen | N/A | N/A | NA | N/A |
| AEL Tampa | Drinking Water | SM4500NO3-F | Nitrite/Nitrate | 7/7 | 6 | 2nd Order | 6 months or QC fails |
| AEL Tampa | Drinking Water | SM4500SD | Sulfide | 7 | 7 | Linear | 6 months or QC fails |
| AEL Tampa | Drinking Water | SM9215B | HPC | N/A | N/A | N/A | N/A |
| AEL Tampa | Drinking Water | SM9215E | HPC (SimPlate) | N/A | N/A | N/A | N/A |
| AEL Tampa | Drinking Water | SM9221F | Escherichia coli (Confirmation)by Tube Fermentation | N/A | N/A | N/A | N/A |
| AEL Tampa | Drinking Water | SM9222B | Total Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Tampa | Drinking Water | SM9222D | Fecal Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Tampa | Drinking Water | SM9223B | Total Coliform by MMO-MUG | N/A | N/A | N/A | N/A |
| AEL Tampa | Drinking Water | SM9223B | Escherichia coli by MMO-MUG | N/A | N/A | N/A | N/A |
| AEL Tampa | Drinking Water | SM3113B | Metals by GFAA | 4 | 4 | Linear 0.995 or > | Daily |
| AEL Tampa | Drinking Water | E245.1 | Mercury by CVAA | 5 | 6 | Linear 0.995 or > | Daily |
| AEL Tampa | Drinking Water | 200.7 | Metals by ICP-AES | 4 | 4 | Linear 0.995 or > | Daily |
| AEL Tampa | Drinking Water | E552.2 | Haloacetic Acids by GC/ECD | 6 | 5 | 2nd order | 6 months or QC fails |
| AEL Tampa | Drinking Water | E524.2 | Trihalomethanes by GC/ECD | 6 | 5 | 2nd order | 1 year or QC fails |
| AEL Tampa | Drinking Water | E504.1 | EDB and DBCP by GC/ECD | 6 | 5 | 2nd order | 6 months or QC fails |
| AEL Tampa | Drinking Water | SM5910B | UV 254 | 1 | N/A | N/A | N/A |
| AEL Tampa | Soil | E160.3/SM2540B | Total Solids | N/A | N/A | N/A | N/A |
| AEL Tampa | Soil | E351.2 | Total Kjeldahl Nitrogen | 8 | 6 | 1st Order | 6 months or QC fails |
| AEL Tampa | Soil | E365.4 | Total Phosphorus | 8 | 6 | 3rd Order | 6 months or QC fails |
| AEL Tampa | Soil | SM2540G | Total Solids in soil | N/A | N/A | N/A | N/A |
| AEL Tampa | Soil | SM4500NO3-F | Nitrite/Nitrate | 7/7 | 6 | 1st Order | 6 months or QC fails |
| AEL Tampa | Soil | SM9221E | Fecal Coliform MPN | N/A | N/A | N/A | N/A |
| AEL Tampa | Soil | SM9221B | Total Coliform by MPN | N/A | N/A | N/A | N/A |
| AEL Tampa | Soil | SM9222D | Fecal Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Tampa | Soil | SM9222B | Total Coliform by MF | N/A | N/A | N/A | N/A |

Table 14 Methods, Tests, Calibration Points and Frequency

| Lab | Matrix | Method | Test Name | # of AEL Calibration | Method Calibration | Type of Regression | Calibration Frequency |
|-----------|--------|------------------|----------------------------------|----------------------|--------------------|--------------------|-----------------------|
| AEL Tampa | Soil | SM4500-NO3 F | Nitrate/Nitrite | 7/7 | 6 | 2nd Order | 6 months or QC fails |
| AEL Tampa | Soil | SM4500-NO3 F | Nitrate+ Nitrite | 7/7 | 6 | 2nd Order | 6 months or QC fails |
| AEL Tampa | Soil | SW9045C | pH | 3 | 3 | Linear | Daily |
| AEL Tampa | Soil | SW9060 | Total Organic Carbon | 12 (3 Ranges) | 12 (3 Ranges) | Linear | 6 months or QC fails |
| AEL Tampa | Soil | SW6010B | Metals by ICP-AES | 2 | 2 | Linear 1.0 | Daily |
| AEL Tampa | Soil | SW7471 | Mercury by CVAAs | 5 | 6 | Linear 0.995 or > | Daily |
| AEL Tampa | Soil | FL-PRO | TRPH | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Tampa | Soil | SW8081A | Pesticides by GC/ECD | 6 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Tampa | Soil | SW8082 | Aroclors by GC/ECD | 5 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Tampa | Soil | SW8260B | Volatile Organics by GC/MS | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Tampa | Soil | SW8270C | Semi-Vol Orgniacs by GC/MS | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Tampa | Soil | SW8270C-SIM | PAHs by GC/MS | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Tampa | Water | E110.2/SM2120B/C | Color | 9 | 9 | linear/non-linear | Annually or QC fails |
| AEL Tampa | Water | E120.1 | Specific Conductivity | 3 | 3 | Linear | Daily |
| AEL Tampa | Water | E150.1/SM4500H+B | pH | 3 | 3 | Linear | Daily |
| AEL Tampa | Water | E160.1/SM2540C | TDS | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | E160.2/SM2540D | TSS | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | E160.3/SM2540B | Total Solids | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | E160.4/SM2540E | Total Volatile Solids | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | E180.1 | Turbidity | 2 | NA | NA | Daily |
| AEL Tampa | Water | E300.0 | Anions by IC | 7 | 6 | Linear | 6 months or QC fails |
| AEL Tampa | Water | E300.1 | Anions by IC (Chlorate/Chlorite) | 7 | 6 | Linear | 6 months or QC fails |
| AEL Tampa | Water | E310.1/SM2320B | Alkalinity | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | E325.1 | Chloride | N/A | NONE | NA | Daily |
| AEL Tampa | Water | E350.1 | Ammonia | 10 | 6 | 1st Order | 6 months or QC fails |
| AEL Tampa | Water | E351.2 | Total Kjeldahl Nitrogen | 8 | 6 | 1st Order | 6 months or QC fails |
| AEL Tampa | Water | E365.1 | Ortho-phosphates, Total P | 7 | 6 | 2nd Order | 6 months or QC fails |
| AEL Tampa | Water | E365.4 | Total Phosphorus | 8 | 6 | 3rd Order | 6 months or QC fails |
| AEL Tampa | Water | E375.4 | Sulfate | 8 | 7 | Linear | 6 months or QC fails |
| AEL Tampa | Water | E405.1 | BOD | N/A | N/A | NA | N/A |
| AEL Tampa | Water | E410.4 | COD | 9 | 9 | Linear | 6 months or QC fails |
| AEL Tampa | Water | E415.1 | Total Organic Carbon | 12 (3 Ranges) | 12 (3 Ranges) | NA | 6 months or QC fails |
| AEL Tampa | Water | E415.1 | Dissolved Organic Carbon | 12 (3 Ranges) | 12 (3 Ranges) | NA | 6 months or QC fails |
| AEL Tampa | Water | E420.2/E420.4 | Total Phenolics | 7 | 6 | 1st Order | 6 months or QC fails |
| AEL Tampa | Water | E425.1/SM5540C | MBAS | 10 | 10 | Linear | 6 months or QC fails |
| AEL Tampa | Water | SM2120B | Color | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | SM2150B | Odor | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | SM2310B | Acidity | N/A | N/A | NA | N/A |
| AEL Tampa | Water | SM2320B | Alkalinity | N/A | N/A | NA | N/A |
| AEL Tampa | Water | SM2340C | Total Hardness by EDTA | N/A | N/A | N/A | N/A |

Table 14 Methods, Tests, Calibration Points and Frequency

| Lab | Matrix | Method | Test Name | # of AEL Calibration | Method Calibration | Type of Regression | Calibration Frequency |
|-----------|--------|------------------------|--|----------------------|--------------------|--------------------|-----------------------|
| AEL Tampa | Water | SM2340B | Total Hardness by Calculation (from 200.7) | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | SM2510B | Conductivity | 3 | 2 | Linear | 6 months or QC fails |
| AEL Tampa | Water | SM2540G | Total Solids in soil | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | SM3500CrD | Hexavalent Chromium | 8 | 6 | Linear | 6 months or QC fails |
| AEL Tampa | Water | SM4500CL-E | Chloride | 9 | 6 | 3rd Order | 6 months or QC fails |
| AEL Tampa | Water | SM4500CL-G | Chlorine | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | SM4500CN-E | Cyanide | 7 | 6 | Linear | 6 months or QC fails |
| AEL Tampa | Water | SM4500CN-G | Amenable Cyanide | 7 | 6 | Linear | 6 months or QC fails |
| AEL Tampa | Water | SM4500F-C | Fluoride | 4 | 5 | Linear | Daily |
| AEL Tampa | Water | SM4500O-G | Dissolved Oxygen | N/A | N/A | NA | N/A |
| AEL Tampa | Water | SM4500NO3-F | Nitrite/Nitrate | 7/7 | 6 | 2nd Order | 6 months or QC fails |
| AEL Tampa | Water | 351.2+SM4500NO3-F | Total Nitrogen by Calculation | N/A | N/A | NA | N/A |
| AEL Tampa | Water | SM4500SD | Sulfide | 7 | 7 | Linear | 6 months or QC fails |
| AEL Tampa | Water | SM5210B | BOD | 1 | N/A | NA | N/A |
| AEL Tampa | Water | SM5210B | CBOD | 1 | N/A | NA | N/A |
| AEL Tampa | Water | SM5310B | TOC | 12 (3 Ranges) | 12 (3 Ranges) | Linear | 6 months or QC fails |
| AEL Tampa | Water | SM2520B | Salinity | 1 | N/A | NA | N/A |
| AEL Tampa | Water | SM9215B | HPC | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | SM9215E | HPC (SimPlate) | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | SM9221B | Total Coliform MPN | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | SM9221E | Fecal Coliform MPN | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | SM9222B | Total Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | SM9222D | Fecal Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | E1603 | E.coli | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | SM9230C | Enterococci | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | SM9223B Quanti-tray | Total Coliform and E. Coli | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | Enterolert | Enterococci by Quanti-tray | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | Colilert 18-Quantitray | Fecal | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | SW9050A | Conductivity | 3 | 2 | Linear | Daily |
| AEL Tampa | Water | DEP SEP 10/03/83 | Un-ionized Ammonia | N/A | N/A | N/A | N/A |
| AEL Tampa | Water | E200.7/SW6010B | Metals by ICP-AES | 2 | 2 | Linear 1.0 | Daily |
| AEL Tampa | Water | E245.1/SW7470 | Mercury by CVAA | 5 | 6 | Linear 0.995 or > | Daily |
| AEL Tampa | Water | FL-PRO | TRPH | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Tampa | Water | SW8081A | Pesticides by GC/ECD | 6 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Tampa | Water | SW8082 | Aroclors by GC/ECD | 5 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Tampa | Water | SW8260B | Volatile Organics by GC/MS | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Tampa | Water | SW8270C | Semi-Vol Orgniacs by GC/MS | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Tampa | Water | SW8270C-SIM | PAHs by GC/MS | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Tampa | Water | 608/608.2 | Pesticides-PCBs by GC/ECD | 5 | 3 | 2nd order per 8000 | 1 year or QC fails |
| AEL Tampa | Water | 624 | Volatile Organics by GC/MS | 7 | 3 | 2nd order per 8000 | 1 year or QC fails |

Table 14 Methods, Tests, Calibration Points and Frequency

| Lab | Matrix | Method | Test Name | # of AEL Calibration | Method Calibration | Type of Regression | Calibration Frequency |
|-----------|----------------|------------------------|---------------------------------------|----------------------|--------------------|--------------------|-----------------------|
| AEL Tampa | Water | 625 | Semi-Vol Orgniacs by GC/MS | 7 | 3 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Drinking Water | SM3113B | Metals Pb by GFAA | 3 | 4 | Linear 0.995 or > | Daily |
| AEL Miami | Drinking Water | E180.1/SM2130B | Turbidity | 2 | NA | NA | Daily |
| AEL Miami | Drinking Water | E300.0 | Anions by IC | 7 | 6 | Linear | 6 months or QC fails |
| AEL Miami | Drinking Water | SM2320B | Alkalinity | N/A | N/A | NA | N/A |
| AEL Miami | Drinking Water | E300.0 | Ortho-phosphates | 7 | 6 | 2nd Order | 6 months or QC fails |
| AEL Miami | Drinking Water | E300.0 | Sulfate | 8 | 7 | Linear | 6 months or QC fails |
| AEL Miami | Drinking Water | SM9215B | HPC | N/A | N/A | N/A | N/A |
| AEL Miami | Drinking Water | SM9222B | Total Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Miami | Drinking Water | SM9221F | E. coli by Multiple Tube Fermentation | N/A | N/A | N/A | N/A |
| AEL Miami | Drinking Water | SM9223B | Total Coliform by MMO-MUG | N/A | N/A | N/A | N/A |
| AEL Miami | Drinking Water | SM9223B | Escherichia coli by MMO-MUG | N/A | N/A | N/A | N/A |
| AEL Miami | Drinking Water | E524.2 | Drinking Water VOCs by GC/MS | 7 | 5 | linear | 6 months or QC fails |
| AEL Miami | Drinking Water | E504.1 | EDB and DBCP by GC/ECD | 6 | 5 | 2nd order | 6 months or QC fails |
| AEL Miami | Drinking Water | E200.7 | Metals by ICP-AES | 2 | 2 | Linear 1.0 | Daily |
| AEL Miami | Drinking Water | SM4500CI-G | Chlorine | NONE | NONE | NA | N/A |
| AEL Miami | Drinking Water | SM2510B | Specific Conductivity | 3 | 3 | N/A | Daily |
| AEL Miami | Drinking Water | E150.1/SM4500H+B | pH | 3 | 3 | N/A | Daily |
| AEL Miami | Drinking Water | SM2330B | Corrositivity | 3 | 3 | N/A | Daily |
| AEL Miami | Drinking Water | SM2540C | TDS | N/A | N/A | N/A | N/A |
| AEL Miami | Drinking Water | SM2340B | Hardness by calc | N/A | N/A | N/A | N/A |
| AEL Miami | Soil | SW6010B | Metals by ICP-AES | 2 | 2 | Linear 1.0 | Daily |
| AEL Miami | Soil | FL-PRO | TRPH | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Soil | SW8260B | Volatile Organics by GC/MS | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Soil | SW8270C | Semi-Vol Orgniacs by GC/MS | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Soil | SW1311 | TCLP Extraction | N/A | N/A | N/A | N/A |
| AEL Miami | Soil | SW1312 | SPLP Extraction | N/A | N/A | N/A | N/A |
| AEL Miami | Soil | SW8270C-SIM | PAHs by GC/MS | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Soil | SM2540G | Total Residue/Total Solids | N/A | N/A | N/A | N/A |
| AEL Miami | Soil | E351.2 | Total Kjeldahl Nitrogen | 8 | 6 | 1st Order | 6 months or QC fails |
| AEL Miami | Soil | E365.4 | Total Phosphorus | 8 | 6 | 3rd Order | 6 months or QC fails |
| AEL Miami | Soil | E350.1 | Ammonia | 10 | 6 | 1st Order | 6 months or QC fails |
| AEL Miami | Soil | SW8081A | Pesticides by GC/ECD | 6 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Soil | SW8082 | Aroclors by GC/ECD | 5 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Soil | SM9221E | Fecal Coliform MPN | N/A | N/A | N/A | N/A |
| AEL Miami | Water | E150.1/SM4500H+B/9040C | pH | 3 | 2 | Linear | Daily |
| AEL Miami | Water | E160.1/SM2540C | TDS | N/A | N/A | N/A | N/A |
| AEL Miami | Water | E160.2/SM2540D | TSS | N/A | N/A | N/A | N/A |
| AEL Miami | Water | E160.3/SM2540B | Total Solids | N/A | N/A | N/A | N/A |
| AEL Miami | Water | E180.1/SM2130B | Turbidity | 2 | NA | NA | Daily |

Table 14 Methods, Tests, Calibration Points and Frequency

| Lab | Matrix | Method | Test Name | # of AEL Calibration | Method Calibration | Type of Regression | Calibration Frequency |
|-----------|--------|------------------|----------------------------|----------------------|--------------------|--------------------|-----------------------|
| AEL Miami | Water | E300.0/9056A | Anions by IC | 7 | 6 | Linear | 6 months or QC fails |
| AEL Miami | Water | E310.1/SM2320B | Alkalinity | N/A | N/A | N/A | N/A |
| AEL Miami | Water | E300.0 | Chloride | 7 | 6 | Linear | 6 months or QC fails |
| AEL Miami | Water | E350.1 | Ammonia | 10 | 6 | 1st Order | 6 months or QC fails |
| AEL Miami | Water | E365.1/SM4500P-E | Ortho-phosphates, Total P | 7 | 6 | 2nd Order | 6 months or QC fails |
| AEL Miami | Water | E375.4 | Sulfate | 8 | 7 | Linear | 6 months or QC fails |
| AEL Miami | Water | E415.1 | Total Organic Carbon | 12 (3 Ranges) | 12 (3 Ranges) | NA | 6 months or QC fails |
| AEL Miami | Water | E420.2/E420.4 | Total Phenolics | 7 | 6 | 1st Order | 6 months or QC fails |
| AEL Miami | Water | SM2150B | Odor | N/A | N/A | N/A | N/A |
| AEL Miami | Water | E310.1/ SM2320B | Alkalinity | N/A | N/A | NA | N/A |
| AEL Miami | Water | SM2510B | Conductivity | 3 | 2 | Linear | 6 months or QC fails |
| AEL Miami | Water | E300.0 | Fluoride | 7 | 6 | Linear | 6 months or QC fails |
| AEL Miami | Water | SM5210B /E405.1 | BOD | 1 | N/A | NA | N/A |
| AEL Miami | Water | SM5210B | CBOD | 1 | N/A | NA | N/A |
| AEL Miami | Water | SM9222B | Total Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Miami | Water | SM9221E | Fecal Coliform by MPN | N/A | N/A | N/A | N/A |
| AEL Miami | Water | SM9222D | Fecal Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Miami | Water | SM9230B | Fecal Streptococci | N/A | N/A | N/A | N/A |
| AEL Miami | Water | SM2510B | Conductivity | 3 | 2 | Linear | Daily |
| AEL Miami | Water | DEP SEP 10/03/83 | Un-ionized Ammonia | N/A | N/A | N/A | N/A |
| AEL Miami | Water | E200.7/SW6010B | Metals by ICP-AES | 2 | 2 | Linear 1.0 | Daily |
| AEL Miami | Water | E200.7 | Hardness by calc | N/A | N/A | N/A | N/A |
| AEL Miami | Water | SM2340B | Total Hardness by calc | N/A | N/A | N/A | N/A |
| AEL Miami | Water | SM2340B | Ccalcium Hardness by calc | N/A | N/A | N/A | N/A |
| AEL Miami | Water | FL-PRO | TRPH | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Water | SW8260B | Volatile Organics by GC/MS | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Water | SW8270C | Semi-Vol Orgniacs by GC/MS | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Water | SW8270C-SIM | PAHs by GC/MS | 7 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Water | 624 | Volatile Organics by GC/MS | 7 | 3 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Water | 625 | Semi-Vol Orgniacs by GC/MS | 7 | 3 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Water | E410.4 | COD | 9 | 9 | Linear | 6 months or QC fails |
| AEL Miami | Water | SM4500CI-G | Chlorine | NONE | NONE | NA | N/A |
| AEL Miami | Water | E351.2 | Total Kjeldahl Nitrogen | 8 | 6 | 1st Order | 6 months or QC fails |
| AEL Miami | Water | SM3500CrD | Hexavalent Chromium | 8 | 6 | Linear | 6 months or QC fails |
| AEL Miami | Water | E365.4 | Total Phosphorus | 8 | 6 | 3rd Order | 6 months or QC fails |
| AEL Miami | Water | SM2530B | Salinity | NONE | NONE | NA | N/A |
| AEL Miami | Water | SM2330B | Corrositivity | 3 | 3 | N/A | Daily |
| AEL Miami | Water | SW8081A | Pesticides by GC/ECD | 6 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Water | SW8082 | Aroclors by GC/ECD | 5 | 5 | 2nd order per 8000 | 1 year or QC fails |
| AEL Miami | Water | 608/608.2 | Pesticides-PCBs by GC/ECD | 5 | 3 | 2nd order per 8000 | 1 year or QC fails |

Table 14 Methods, Tests, Calibration Points and Frequency

| Lab | Matrix | Method | Test Name | # of AEL Calibration | Method Calibration | Type of Regression | Calibration Frequency |
|-----------------|----------------|------------------------|--|----------------------|--------------------|--------------------|-----------------------|
| AEL Miami | Water | SM2540g | Total Residue | N/A | N/A | N/A | N/A |
| AEL Tallahassee | Drinking Water | E110.2/SM2120B | Color | N/A | N/A | N/A | N/A |
| AEL Tallahassee | Drinking Water | E140.1/SM2150B | Odor | N/A | N/A | N/A | N/A |
| AEL Tallahassee | Drinking Water | E150.1/SM4500H+B | pH | 3 | 3 | N/A | Daily |
| AEL Tallahassee | Drinking Water | E300.0 | Anions (CL,F,NO2,NO3,NO2+NO3,SO4)by IC | 5 | 3 | 0.995 | 6 months or QC fails |
| AEL Tallahassee | Drinking Water | SM9215B | HPC | N/A | N/A | N/A | N/A |
| AEL Tallahassee | Drinking Water | SM9223B | Escherichia coli by MMO-MUG | N/A | N/A | N/A | N/A |
| AEL Tallahassee | Drinking Water | SM9222B | Total Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Tallahassee | Drinking Water | SM9223B | Total Coliform by MMO-MUG | N/A | N/A | N/A | N/A |
| AEL Tallahassee | Water | E150.1/E9040 | pH | 3 | 3 | N/A | Daily |
| AEL Tallahassee | Water | SM4500H+B | pH | 3 | 3 | N/A | Daily |
| AEL Tallahassee | Water | E300.0 | Anions (CL,F,NO2,NO3,NO2+NO3,SO4)by IC | 5 | 3 | 0.995 | 6 months or QC fails |
| AEL Tallahassee | Water | E405.1 | BOD | 1 | N/A | N/A | Daily |
| AEL Tallahassee | Water | SM2540D | TSS | N/A | N/A | N/A | N/A |
| AEL Tallahassee | Water | SM5210B | BOD | 1 | N/A | N/A | Daily |
| AEL Tallahassee | Water | SM5210B | CBOD | 1 | N/A | N/A | Daily |
| AEL Tallahassee | Water | SM9222B | Total Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Tallahassee | Water | SM9222D | Fecal Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Drinking Water | SM2120B | Color | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Drinking Water | SM2150B | Odor | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Drinking Water | E150.1/SM4500H+B | pH | 3 | 2 | N/A | Daily |
| AEL Fort Myers | Drinking Water | E300.0 | (CL,F,NO2,NO3,NO2+NO3,OP,SO4) Anions by IC | 5 | 3 | 0.995 | 6 months or QC fails |
| AEL Fort Myers | Drinking Water | SM9223B | Total Coliform-E. coli i by MMO-MUG | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Drinking Water | SM9222B-SM9221F | Total Coliform-E. coli by MF | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Drinking Water | SM2540C | TDS | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Drinking Water | SM9215B | HPC | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Drinking Water | 180.1 | Turbidity | 3 | 3 | 0.995 | 6 months or QC fails |
| AEL Fort Myers | Water | E150.1/E9040 | pH | 3 | 3 | N/A | Daily |
| AEL Fort Myers | Water | 150.1/SM4500H+B 9040C | pH | 3 | 3 | N/A | Daily |
| AEL Fort Myers | Water | E300.0 | (CL,F,NO2,NO3,NO2+NO3,OP,SO4) Anions by IC | 5 | 3 | 0.995 | 6 months or QC fails |
| AEL Fort Myers | Water | SM2540D | TSS | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Water | SM5210B | BOD | 1 | N/A | N/A | Daily |
| AEL Fort Myers | Water | SM5210B | CBOD | 1 | N/A | N/A | Daily |
| AEL Fort Myers | Water | SM9222B | Total Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Water | SM9222D | Fecal Coliform by MF | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Water | SM9223B Quanti-tray | Total Coliform and E. Coli | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Water | Enterolert | Enterococci by Quanti-tray | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Water | Colilert 18-Quantitray | Fecal | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Water | SM9221E | Fecal Coliform by MPN | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Water | SM9215B | HPC | N/A | N/A | N/A | N/A |

Table 14 Methods, Tests, Calibration Points and Frequency

| Lab | Matrix | Method | Test Name | # of AEL Calibration | Method Calibration | Type of Regression | Calibration Frequency |
|-------------------|--------|---------|---|----------------------|--------------------|--------------------|-----------------------|
| AEL Fort Myers | Water | SM2540C | TDS | N/A | N/A | N/A | N/A |
| AEL Fort Myers | Water | 180.1 | Turbidity | 3 | 3 | 0.995 | 6 months or QC fails |
| AEL Fort Myers | Soil | E9056A | (CL,F,NO2,NO3,NO2+NO3,OP,SO4) Anions by IC | 5 | 3 | 0.995 | 6 months or QC fails |
| AEL Fort Myers | Soil | SM9221E | Fecal Coliform MPN | N/A | N/A | N/A | N/A |

15.0 Data Integrity

See also ADMIN SOP-035 Ethics and Data Integrity, Proactive Fraud Prevention and Detection. See also **Section 4.0 for cyber-security and annual setting of the password for the Jacksonville facility.**

15.1 The goal of the AEL Quality System is to provide data with the greatest integrity possible to the clients. The entire Quality Manual works to ensure data integrity, but some specific examples not yet covered are explained in detail in this section.

15.2 Data integrity training is provided as part of the initial training for all employees, as discussed in the AEL QM Sections 1.0 and 3.0.

15.2.1 An annual refresher course on data integrity/ethics is provided by the QA Department and attendance is documented and placed in the employee binder or saved electronically on the designated Quality Assurance (Q) drive of the AEL networked servers

15.2.1.1 This refresher course will have a signed attendance sheet to provide documentation of the training.

15.3 Manual Calculations and Manual Integrations

15.3.1 All manual calculations will be detailed in the analytical SOPs.

15.3.1.1 All manual calculation spreadsheets will be validated according to the procedures outlined below.

15.3.1.2 Manual calculations will be performed in accordance, and meet the requirement, with the Ethics policy outlined in Section 1.0

15.3.2 All manual integrations will be performed in accordance with the policy outlined in Section 12.3 and ADMIN SOP-038, and will also be performed in accordance with the Ethics policy stated in Section 1.0

15.3.2.1 The analytical SOPs will reference this QM as well as ADMIN SOP-038 to ensure that all aspects of manual integrations are fully explained.

15.3.2.2 Each analyst responsible for interpreting chromatograms will have documentation of file that they have read,

understood, and agree to follow the rules for manual integration as stated in ADMIN SOP-038

15.4 Hardware Validation Procedure

15.4.1 AEL has a program set up to ensure that the computer hardware functions properly. This will ensure that the software performs properly, and valid data is generated.

15.4.2 This process is accomplished by the methods listed below:

15.4.2.1 All computer equipment is purchased directly by the IT department where it is configured for its assigned task. Once setup, it is then either put in place by IT or sent to the lab and put in place under IT's direct guidance or by IT's instructions. IT has remote access through the network for all computers and servers.

15.4.2.2 On an annual basis, or sooner if necessary, the computer will have the routine maintenance performed on it, i.e. scandisk and disk defragmenter. This is again handled by the IT department.

15.4.2.3 If there are problems encountered during this process, then the software or operating system may need to be reinstalled to correct the problem. This is only done under advisement of the IT Manager.

15.4.2.4 An inventory of all the computers under AEL control is kept with the IT department listing the lab, location, and their OS through network listing.

15.4.3 The data integrity is further guaranteed by of backups of the network servers on a nightly basis and the use of a backup servers at a secure offsite collocation center.

15.4.3.1 There is a nightly backup of the directories that contains the LIMS database, users folders, system folders, and other directories that contain the admin folders for the lab.

15.4.3.2 Daily, weekly, and monthly backups schedules for all computers are performed according to SOP ADMIN-029.

15.4.4 The data generated by the software that runs each instrument is backed up and stored on CD-ROMs, backed up to the company servers, or kept as hard copy data. This is the responsibility of the analyst in conjunction with IT personnel and the backup is stored in the individual departments in accordance with ADMIN SOP-029.

15.5 Software validation and integrity

15.5.1 AEL's system for ensuring validation and integrity in its software use is accomplished through:

15.5.1.1 Purchasing software such as our LIMS system and Microsoft Office from approved vendors

15.5.1.2 Assigning every employee with a login name and password to the networked computer.

15.5.1.3 Purchase newer software as it becomes available or update the software already in use to ensure most efficient and secure software is in use

15.5.2 LIMS integrity is accomplished through:

15.5.2.1 In addition to every employee having a password authentication to access the system, each employee is also assigned certain permissions (See also section 15.8). These permissions are set up by the administrator (I.T. Manager) and can only be changed by someone with administrator privileges.

15.5.2.2 These permissions consist of which departments an analyst is allowed to view, enter, validate, or approve data. These vary upon position in AEL, as well as department.

15.5.2.3 In addition to these types of permissions, there are also designated which features of the LIMS system each employee is allowed to use. Such as only employees with administrator privileges are able to set up tests or change parameters, while most employees are allowed to create worklist. These permissions are also set up by the administrator and can only be changed by an administrator.

15.6 Electronic Calculation Verification

- 15.6.1 Any calculations that are created or modified by AEL are verified before use.
- 15.6.2 All commercially available software that is purchased from an outside source is assumed to be correct and no further verification is performed for that software.
- 15.6.3 Any customization or modifications of software performed by AEL are verified for accuracy before implementation.
 - 15.6.3.1 The IT Director creates most modifications. He will create the modification and then pass it along to someone else for verification that it is functioning as expected before placing the modification in widespread use.
 - 15.6.3.2 The person verifying the modification will typically be one of the following: QA Officer, Lab Manager, or Department Supervisor.
 - 15.6.3.3 Under no circumstances will modification be performed without verification before implementation.
 - 15.6.3.4 An example of the modification process is the significant figure calculations created by the IT Director. Every possible combination of numbers was verified to function properly before this was implemented.
 - 15.6.3.4.1 The review of any analytical report will prove the rounding and significant figure modification is functioning properly.
 - 15.6.3.4.2 This modification follows the significant figure rules as outlined in SOP ADMIN-011.
 - 15.6.3.5 If any errors are determined for any modification after being placed in service, the modification will be corrected immediately, and its use stopped until it is proven to be corrected.
 - 15.6.3.5.1 If it is determined that any errors in a customized calculation or modification has generated erroneous data and affects results that

have already been given to the client, then the following will happen:

15.6.3.5.1.1 The client will be notified, in writing, of the error and how it affects the data, and

15.6.3.5.1.2 An amended report will be generated with the corrected results.

15.7 Amended Reports

15.7.1 Any erroneous result that is determined after the report has been distributed to the client will involve an amended report.

15.7.2 The amended report will have the error corrected and the rest of the report will remain unchanged.

15.7.3 The amended report will be identified with the words 'AMENDED REPORT' on every page of the report.

15.7.4 There will also be a date amended on the cover page of the report to identify when the report was amended and also provide reference in circumstances where a report may need to be identified more than once.

15.7.5 All amended reports will have a reason as to why they are being amended. This reason will be written into the case narrative and attached to the end of the report.

15.7.6 No report will be amended without a valid reason documented. This documentation is to be in the form of a case narrative and electronically attached and saved with the report.

15.8 LIMS and Network Access

15.8.1 Another way of ensuring data integrity is by limiting access to certain portions of the computer system. The following processes accomplish this.

15.8.2 LIMS Access

15.8.2.1 The software itself controls this. Each employee has their own user profile assigned in the LIMS, which gives them

access to certain features and can perform various stages of data entry, validation, and approval.

15.8.2.2 Each employee has their own username and password to access the LIMS software to ensure each employee is accessing what they are allowed to access. Note: Some permissions are assigned by department in non- critical areas such as extractions where log traceability is clearly evident. For DoD work, user login shall also be through the EISC reporting software, always with individual password.

15.8.2.2.1 This username is assigned to all functions performed while inside the LIMS so any functions completed by the employee will be stamped with the username and time, so all changes are traceable.

15.8.2.3 The IT Director manages the permissions and profiles.

15.8.2.4 Data Review Permissions

15.8.2.4.1 The following hierarchy is used for setting permissions for data review and entry.

15.8.2.4.1.1 All employees have access to View Results.

15.8.2.4.1.2 Analysts have permission to enter results for their respective department only.

15.8.2.4.1.3 Department supervisors have permission to validate results for their respective departments. This implies they also have permission to enter results as well.

15.8.2.4.1.4 Project Managers, Lab Managers, and QA Personnel have permission to approve (and reject) data for all departments.

15.8.2.4.1.4.1 Project managers also have permission and are

responsible for entering results from subcontracted laboratories if the entire subcontracted laboratory's report is not going to be included as part of the report.

15.8.3 Network Access

15.8.3.1 Each employee also has his or her own username and password for access to the AEL network.

15.8.3.2 Each employee is setup with different network rights, such as User or Administrator.

15.8.3.3 The rights are determined by the employee's role in the company and what the employee needs access to. The IT Director and QA Officer determine these rights.

15.8.3.4 AEL controls the access to many folders on individual PCs and the network server to administrator rights, or even specific employees, to ensure the integrity of the documents contained in those files are not impacted.

15.8.3.4.1 This would include employee files or human resource documents that need not be accessible by all employees.

15.8.3.5 Some files are inaccessible or read-only to ensure their integrity as well. This would include protected SOP files.

15.8.3.6 See SOP ADMIN-025 for the procedure on writing SOPs and how access is protected to ensure the original document is not changed without permission.

15.9 Analytical Software Access

15.9.1 AEL makes no special provisions for access to analytical software. While some of the software does allow individual passwords to be set up for the individual analysts, it is not utilized across the board by AEL. Since most instruments are running 24/7, controlling access to the software is not practical.

15.9.2 Access is controlled via the username and password for network/computer access only. The supervisors ensure there is no

unauthorized access to the analytical software through normal supervision processes.

15.9.3 This aspect is covered in data integrity training and fully explained to the analysts that they are responsible for processing their own data and they are not to access any software they have not demonstrated the capability of performing.

15.10 Electronic Record Revisions

15.10.1 All record changes are to be maintained and documented through the use of the LIMS. See LIMS instruction manuals and training documents provide by the IT department for use of audit trail features.

15.10.2 In the event of the audit trail being unavailable for use, the following alternate tracking shall be used.

15.10.2.1 All changes made to electronic records during the approval process, or after the data has been approved, will be retained in the project folder.

15.10.2.2 The PM reviewing the project and making the change will document the change on the benchsheet or report and provide a reason for the change along with the initials and date of the PM. The document being changed will then be retained in the project folder as a record as to when the change was made, by whom, and why.

15.10.2.2.1 If the report is where the change occurred, then the cover page of the report will be indicated as being 1st review, 2nd review, etc., to indicate there was a change made. All reviews will be retained in the project folder according to the storage procedures encompassing the project folder.

15.10.2.2.2 If the benchsheets are where the change is made, then new benchsheets shall be printed and both sets will be retained in the project folder.

15.10.2.2.2.1 Both sets will be paper clipped together to improve organization of the project folder.

15.10.2.2.3 If the change is made on the Login report during the initial project review, then a new Login report will be printed and attached to the folder. Both copies will be retained in the project folder.

15.10.2.2.3.1 Any emails relating to this change being processed and completed will be printed out and retained in the project folder as well.

15.10.2.2.3.2 The emails will be stapled to the erroneous Login report for organizational purposes and to retain the audit trail effectively.

16. References

- 16.1. TNI 2016 Standards, Volume 1 rev 2.1: Management and Technical Requirements for Laboratories Performing Environmental Analysis.
- 16.2. 2017 DEP SOPs issued January 2017, from FDEP as found at:
<http://www.floridadep.org/labs/qa/sops.htm>
- 16.3. Process for Assessing Data Usability, DEP-EA 001/07, effective 3/31/2008 from FDEP Bureau of Standards and Special Projects, Environmental Assessment Section.
- 16.4. FDOH onsite assessment checklists, effective July 2009 as found at:
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- 16.5. Department of Defense Quality Systems Manual for Environmental Laboratories, version 5.3 effective June 2019.
- 16.6. Department of Defense Quality Systems Manual for Environmental Laboratories, version 5.4 effective October 2021.
- 16.7. Standard Methods for the Examination of Water and Wastewater, Online editions 18th through 23rd editions (On-line editions).
- 16.8. ISO/IEC 17025:2017 General requirements for the competence of testing and calibration laboratories.
- 16.9. AEL Administrative, Technical, Metals, Microbiology, Semi-volatiles, Volatiles, and Wet Chemistry Standard Operating Procedures, most current revisions.
- 16.10. AEL Policies and Procedures Manual, rev 8, effective March 1, 2022
- 16.11. AEL Chemical Hygiene Plan and Safety Manual, rev 3, effective 9/03/2019
- 16.12. 40 CFR Part 136, Appendix B, Definition and Procedure for the Determination of the Method Detection Limit, August 28, 2017.
- 16.13. US EPA Methods approved for Drinking Water and Ground Water Analysis, most recent revisions.
- 16.14. Methods for Chemical Analysis of Water and Wastes, USEPA Office of Research and Development
- 16.15. US EPA SW-846 Test Methods for Evaluating Solid Waste and Physical/Chemical Methods, most recent revisions. Office of Solid Waste and Emergency Response, Washington, DC

- 16.16. The Nelac Institute (TNI) website for Fields of Proficiency Testing as found at: <http://www.nelac-institute.org/>
- 16.17. Code of Federal Regulations, Title 40, Part 136; U.S. Government Printing Office, Washington, D.C., electronic version as of February 24th, 2020. and referenced thereafter at <http://www.ecfr.gov>
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STANDARD OPERATING PROCEDURE

For

Method 3550B & 3550C

Ultrasonic Extraction





SOP Revision Log for AEL SOP SVOC-002

| Revision Number | Revision Date | Reason for Revision | Section(s) and Page(s) Revised |
|-----------------|---------------|---|--------------------------------|
| Revision 00 | 11/28/01 | Initial Creation in this format | All sections affected |
| Revision 01 | 1/13/03 | Initial start of revision log tracking | All sections affected |
| Revision 02 | 2/08/07 | Revisions and updates throughout | All sections affected |
| Revision 03 | 7/09/10 | Revisions and re-writes throughout. Added revision log, maintenance section. | All sections affected |
| Revision 03 | 4/20/12 | Revisions to language and spellings throughout. Addition of warnings on solvent usage. No procedural changes | All sections affected |
| Revision 04 | 8/14/15 | Review with revisions to language and spellings throughout. Update to references and tables. No procedural changes | All sections affected |
| Revision 05 | 4/24/19 | Update references, update validating analysts. Update Table 1 to add DRO | Section 23, 24 |
| Revision 06 | 4/15/20 | Update references, update validating analysts. Minor changes to language throughout. Added detail to procedures for spiking and summary of cleanup methods. | All sections affected |
| Revision 07 | 9/17/21 | Updated analysts, equipment and supplies, references and routine maintenance | Sections 9, 12 and 21-24 |
| Revision 08 | 02/07/2023 | Updated validating statement, equipment and supplies, and references. | Sections 9, 23, and 24 |
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1.0 Identification of Test Method

1.1 Method 3550B & 3550C, Ultrasonic Extraction

1.2 This SOP deviates from EPA method 3550B and 3550C in the following areas. Samples are extracted then concentrated using Zymark tubes and TurboVap concentrators and not the method stated K-D apparatus, Snyder columns and concentrator tubes (see section 9 and 14 for details). Extractions are done ultrasonically in sonication baths and not with a sonication horn. These deviations from the referenced test method represent equivalent or improved performance over the referenced test method conditions.

2.0 Applicable Matrix or matrices

2.1 Method 3550 is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soil, sludge, and waste. The ultrasonic process ensures intimate contact of the sample matrix with the extraction solvent.

3.0 Detection Limit

3.1 Not Applicable for an extraction procedure. See the analytical method that analyzes the extract for details of the MDLs.

4.0 Scope and Application, including components to be analyzed

4.1 The method is based on the expected concentration of organics in the sample. If a sample is expected to have a high concentration of target (or interfering non-target) analytes, a lesser volume for extraction may be used.

4.2 For some analytical methods, it is highly recommended that the extracts be cleaned up prior to analysis. See individual analysis test methods for cleanup requirements.

4.3 Ultrasonic extraction may not be as rigorous as some of other extraction methods for soils/solids. Therefore, it is critical that the method be followed explicitly in order to achieve the maximum extraction efficiency.

4.4 **Samples must be extracted dried of water.** Water will make a barrier difficult for some solvents to penetrate.

4.5 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

4.6 This method is not appropriate for applications where high extraction efficiencies of analytes at very low concentrations are necessary (e.g., demonstration of effectiveness of corrective action).

4.7 This method will be used to extract sample for semi-volatile organics including EPA Methods 8270, 8270-SIMS, DRO, 8081, 8082, and 8141.

5.0 Summary of Method

- 5.1 A 30-g (or lesser volume dependent upon requirements and conditions) sample is mixed with anhydrous sodium sulfate to form a **dry, free-flowing powder**. This is solvent extracted three times using ultrasonic extraction. The extract is separated from the sample by gravity filtration or centrifugation. The extract is ready for cleanup and/or analysis following concentration.
- 5.2 For samples with high concentrations of target (or interfering non-target) analytes, a lower gram weight sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent extracted once using ultrasonic extraction. A portion of the extract is removed for cleanup and/or analysis. High density samples may also require extraction at lower gram weight.
- 5.3 Any excess water is removed by pouring the extract through a sodium sulfate column and then concentrating, and as necessary, exchanged into a solvent compatible with the cleanup or determinative method to be used (see Table 1 for appropriate exchange solvents).

6.0 Definitions

See also AEL ADMIN SOP-039 Laboratory Definitions

- 6.1 CCV: Continuing Calibration Verification – injected at the beginning of a 12-hour analytical shift. For methods without internal standards, also injected at the end of an analytical batch of 20 to confirm calibration throughout the run. In those instances, CCVs bracket an analytical run of 20 samples or less.
- 6.2 ICV: Initial Calibration Verification – injected after the initial calibrated of an instrument in order to verify the initial calibration. If the response (or calculate concentration) for any analyte is within method stated control criteria of the initial calibration response per analyte, then the ICV and the curve is considered valid. ICV standards are made from a source different than that used to make the curve standards.
- 6.3 Second source standard: (Separate source standard) A standard obtained or prepared from a source independent of the source of standards used for the initial calibration. Its concentration should be at or near the middle of the calibration range unless otherwise defined by the method. It is used to confirm the accuracy of the initial calibration.
- 6.4 Internal Standard: Pure analytes added to a sample, extract, or standard solution in a known amount and is used to measure the relative responses of other method analytes that are components of the same sample of solution. The internal standard must be an analyte that is not a sample component.
- 6.5 Laboratory Control Sample, (LCS), also referred to as Laboratory fortified blank (LFB): a quantity of reagent water to which known quantities of analytes are added in the laboratory. The LCS is analyzed exactly like a sample with its purpose being to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.



- 6.6 Laboratory Control Sample Duplicate: (LCSD) a second quantity of reagent water to which known quantities of analytes are added in the laboratory. The LCSD is analyzed exactly like the LCS with its purpose being to determine whether the laboratory is capable of obtaining accurate and reproducible measurements.
- 6.7 Method Blank, (MB), also referred to as Laboratory reagent blank (LRB): a quantity of reagent water that is treated exactly as a sample including exposure to all equipment, acids, and internal standards and surrogate standards where applicable. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, reagents or other materials.
- 6.8 Matrix Spike, (MS), also referred to as Laboratory fortified matrix, (LFM): An aliquot of a client supplied sample, chosen at random, to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like the sample with its purpose being to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the values in the MS corrected for the background concentration. Matrix Spikes are analyzed with every batch or per 20 samples.
- 6.9 Matrix Spike Duplicate: (MSD) A second aliquot of a client supplied sample to which known quantities of the method analytes are added in the laboratory. The MSD is analyzed exactly like the MS with its purpose being to confirm Matrix bias and determine whether the laboratory is capable of obtaining reproducible and accurate results. Matrix Spikes are analyzed with every batch or per 20 samples.
- 6.10 Case Narrative (CN) -- A case narrative is simply a means of describing exactly what transpired with the samples during the analytical process. Case narratives are required for variances that occur within a project.
- 6.11 Non-Conformity Form (NCF) -- Form which will be completed and processed for each QC failure or deviation from normal protocol that occurs outside the scope of normal operation as defined by the AEL QM Section 10, AEL SOP Admin-016 and Method SOP.
- 6.12 Standard -- A solution prepared by diluting stock standard solutions used to calibrate the instrument response with respect to analyte concentrations. Also referred to as calibration standards (CALs) as in section 10.10.
- 6.13 Stock Standard -- A concentrated solution containing method analytes that is purchased from a commercial source having Certificates of Analysis.
- 6.14 Surrogate Analyte (SA) -- A pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(s) before extraction and is measured with the same procedures used to measure other sample components. The purpose of a surrogate analyte is to monitor method performance with each sample.
- 6.15 Stock Standard Solution (SSS) -- A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the

laboratory with an assayed reference compound. Stock standard solutions are used to prepare primary dilution standards.

- 6.16 Primary Dilution Standard Solution (PDS) -- A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.
- 6.17 Calibration Standard (CAL) -- A solution prepared from the primary dilution standard solution and stock standard solutions of the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 6.18 Quality Control Sample (QCS) -- A sample matrix containing method analytes or a solution of method analytes in a water miscible solvent which is used to fortify reagent water or environmental samples. The QCS is obtained from a source external to the laboratory and is used to check laboratory performance with externally prepared test materials.
- 6.19 Safety Data Sheets (SDS): Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire and reactivity data, including storage, spill and handling precautions.
- 6.20 Analytical Batch: The set of samples started through the analytical process to a maximum of 20 field samples. A Method Blank, a Laboratory Control Sample, and a Sample Duplicate of at least one of the field samples, if available, must accompany each analytical batch of 20 or fewer samples.
- 6.21 LOD - Limit of Detection (LOD): LOD is **not** synonymous with MDL. LOD is an estimate of the minimum amount of a substance that an analytical process can reliably detect with a high level of confidence (99% Confidence, or a false negative rate of 1%) An LOD is analyte, prep method, cleanup method, analysis method, and matrix specific and is laboratory dependent. The LOD is at the level of the MDL verifications. The LOD when used as the MDL verification must go through all the same processes that a sample will go through and be detected above instrument noise level.
- 6.22 Limits of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g. target analyte) that can be reported with a specific degree of confidence. It is also the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DOD work the LOQ will be set at or above the concentration of the lowest initial calibration standard. For DOD work the LOQ will be the concentration at which the PQL is verified. The LOQ can equal the PQL but is not synonymous with the PQL.
- 6.23 Limits of Quantitation (LOQ) Verification: LOQ verifications are a spiked clean matrix sample that must go through all the same processes that regular samples go through and be within the precision and bias acceptance criteria of the method. The LOQ verifications are spiked at the concentrations of the LOQ.
- 6.24 Method Detection Limit: (MDL) – an estimate of the minimum amount of a substance that an analytical process can readily detect. The minimum concentration of an analyte that can be

identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. Is determined most often as 3.14 times the standard deviation of a low level seven replicate study, however, can be determined for some methods as the lowest increment measurable with confidence. MDLs are determined for each analyte, matrix, prep method, cleanup method, analysis method, and instrument. (See each method's requirements). The MDL is one way to establish a Detection Limit, not a Limit of Detection.

- 6.25 **Method Detection Limit: (MDL) Verification:** MDL verifications are a spiked clean matrix sample that must go through all the same processes that a regular sample will go through and be detected above instrument noise level. For labs accredited under DOD ELAP, the MDL verifications must be performed immediately after the initial MDL study and on a quarterly basis thereafter. For labs accredited under NELAC (TNI) Standards only, MDL verifications are to be performed immediately after the initial MDL study and on a yearly basis thereafter.
- 6.26 **Practical Quantitation Level (PQL):** the lowest calibration standard or lowest quantitation level for the method and matrix. The concentration below which data is to be qualified as having less certainty. PQLs are at a concentration greater than that of the MDL.
- 6.27 **Liquid Sample:** A sample classified as a groundwater, surface water, wastewater or other water-soluble liquid.
- 6.28 **Representative Sample:** A well-mixed aliquot of sample that constitutes an accurate representation of contents within container. Methods used to achieve representative sub-sample are described below:
- 6.28.1 **Soil Samples.**
- 6.28.1.1 If container size is sufficient, sample is mixed within until homogeneous. If container size is insufficient, the entire sample is transferred to an appropriate container and mixed.
- 6.28.1.2 **Miscellaneous Solid Samples.** Sample is crushed, pulverized, shaken and stirred as appropriate to ensure the aliquot used for analysis represents the entire contents of the original sample container as accurately as possible.
- 6.28 **Sample Duplicate (DUP):** A second aliquot of a client supplied sample. The sample duplicate is analyzed exactly as the client supplied sample and is used to determine whether the laboratory is capable of obtaining reproducible results.

7.0 Interferences

- 7.1 Some matrices are absorbent and produce low recoveries for spikes and surrogates such as concretes and certain types of ashes.

8.0 Safety

8.1 Refer to the AEL Health and Safety Manual for safety precautions and for the Hygiene Plan and Emergency Response Plan.

8.2 See Standard Methods, 22nd Edition, Section 1090 Laboratory Occupational Health and Safety.

9.0 Equipment and Supplies

Note: SOPs are updated on a set schedule and as a result may not reflect new additions or updates to laboratory equipment inventory at each site as the year progresses. For real-time equipment tracking, please reference the ADMIN-049a AEL QM Section 7.0 - Contemporary Equipment List (most recent revision) link on the intranet SOP system for the current listing by room location and letter designation for each piece of major equipment (by make, model, and serial number) and a full inventory of all major pieces of equipment in each lab.

9.1 Apparatus for grinding dry waste samples.

9.2 Ultrasonic preparation - A horn-type device equipped with a titanium tip, or a device that will give equivalent performance, shall be used.

9.2.1 Ultrasonic Disrupter - The disrupter must have minimum power wattage of 300 watts, with pulsing capability. A device designed to reduce the cavitation sound is recommended. Follow the manufacturer's instructions for preparing the disrupter for extraction of samples with low and medium/high concentration.

9.2.2 Sonication Bath – Branson Model 8510/x2 or equivalent.

9.2.2.1 The sonication bath can be used in place of the sonication horns as data indicates similar recoveries with those listed in the published methods. This is in compliance with the equipment section 6.0 of both 3550B and 3550C in which the use of a horn type device or a device of appropriate performance is listed. However once one type of sonication device has been validated by an initial demonstration of capability, that device must remain in use for all future extractions, unless a new initial demonstration of capability is performed with passing results for that new device type.

9.3 Apparatus for determining percent dry weight.

9.3.1 Drying oven - capable of maintaining 105°C.

9.3.2 Desiccator.

9.3.3 Crucibles - Porcelain or disposable aluminum.

9.4 Borosilicate glass pipets - 1-mL, disposable.

9.5 Beakers - 400-mL.



9.6 Turbopap tubes: 300 mL concentration vessels with a 1.0 mL collection tip.

9.7 80mm glass powder-funnels.

9.8 Large Volume (200-500mL) concentration system Turbo-Vap consists of a warm water bath. Each unit concentrates six samples simultaneously with a flow of nitrogen gas.

9.9 Boiling chips - Solvent-extracted, approximately 10/40 mesh (silicon carbide or equivalent).

9.10 Balance - Top-loading, capable of accurately weighing to the nearest 0.01 g.

9.11 Vials - 2-mL, for GC autosampler, with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

9.12 Glass scintillation vials - 40-mL, with PTFE-lined screw caps.

9.13 Spatula - Stainless steel or PTFE.

9.14 Syringe – 10.0µL -1000µL

9.15 Glass Wool

9.16 Hood that meets safety rating

9.17 Solvent dispensers

10.0 Reagents and Standards

10.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise specified, it is intended that all inorganic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.2 Reagents are ordered and tracked in accordance with SOP's ADMIN-013 and ADMIN-031.

10.3 The mixing of reagents shall be tracked in a reagent logbook kept in the extractions room and will contain the following information: for parent materials the lot number, manufacturer name, chemical name, expiration date, AEL receiving lot#, amount used, how it was mixed, and then the newly created reagent will have a lot #, the date it expires, and its concentration. Examples of this would be the cutting of acid 1:1 and the mixing of sodium hydroxide.

10.4 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, which will be from the di water tap. Siemens Company in accordance with Admin-032 maintains our present di water system.



- 10.5 Sodium sulfate (granular, anhydrous), Na_2SO_4 . Purify by heating at 400°C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate.
- 10.6 Extraction solvents - Samples should be extracted using a solvent system that gives optimum, reproducible recovery of the analytes of interest from the sample matrix. Table 1 provides recovery data for selected semivolatile organic compounds extracted from an NIST SRM. The following sections provide guidance on the choice of solvents for various classes of analytes. All solvents must be pesticide quality or equivalent.
- 10.6.1 Semivolatile organics may be extracted with acetone/methylene chloride (1:1, v/v), $\text{CH}_3\text{COCH}_3/\text{CH}_2\text{Cl}_2$ or acetone/hexane (1:1, v/v), $\text{CH}_3\text{COCH}_3/\text{C}_6\text{H}_{14}$.
- 10.6.1.1 Reducing the ratio of acetone in the mix reduces the amount of water pulled during the extraction process. If sample is well dried so as to prevent any water barrier to the hydrophobic methylene chloride, then the acetone can be left out of the mix and the use of methylene chloride alone can be used. To repeat, the sample MUST be completely dry and free flowing.
- 10.6.2 Organochlorine pesticides may be extracted with acetone/hexane (1:1, v/v), $\text{CH}_3\text{COCH}_3/\text{C}_6\text{H}_{14}$ or acetone/methylene chloride (1:1, v/v), $\text{CH}_3\text{COCH}_3/\text{CH}_2\text{Cl}_2$.
- 10.6.2.1 Reducing the ratio of acetone in the mix reduces the amount of water pulled during the extraction process. If sample is well dried so as to prevent any water barrier to the hydrophobic methylene chloride, then the acetone can be left out of the mix and the use of methylene chloride alone can be used. To repeat, the sample MUST be completely dry and free flowing.
- 10.6.3 PCBs may be extracted with acetone/hexane (1:1, v/v), $\text{CH}_3\text{COCH}_3/\text{C}_6\text{H}_{14}$, acetone/methylene chloride (1:1, v/v), $\text{CH}_3\text{COCH}_3/\text{CH}_2\text{Cl}_2$ or hexane, C_6H_{14} .
- 10.6.3.1 Reducing the ratio of acetone in the mix can reduce the amount of water pulled during the extraction process. If sample is well dried so as to prevent any water barrier to methylene chloride (which is hydrophobic), then the acetone can be left out of the mix and the use of methylene chloride alone can be used. To repeat, the sample MUST be completely dry and free flowing.
- 10.6.4 Other solvent systems may be employed, provided that the analyst can demonstrate adequate performance for the analytes of interest in the sample matrix (see Method 3100, Sec. 8.0)
- 10.7 Exchange solvents - All solvents must be pesticide quality or equivalent.
- 10.7.1 Hexane, C_6H_{14} .
- 10.7.2 2-Propanol, $(\text{CH}_3)_2\text{CHOH}$.



10.7.3 Cyclohexane, C₆H₁₂.

10.7.4 Acetonitrile, CH₃CN.

10.7.5 Methanol, CH₃OH.

11.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

11.1 See AEL Quality Manual section 6.0 for sample acceptance policy.

11.2 See AEL Admin-005 and Admin-023

11.3 See FDEP SOP FS1000 for preservation requirements, shipping conditions, and holding time requirements.

12.0 Quality Control

12.1 Any reagent blanks, matrix spikes, or replicate samples should be subjected to exactly the same analytical procedures as those used on actual samples.

12.2 See each analytical method used for the amount and type of QC needed. At a minimum a method blank, laboratory control spike, matrix spike and matrix spike duplicate will need to be extracted with any extraction batch up to 20 samples.

12.3 An initial demonstration of capability for extraction for the analyte list for the analytical method is required for any analyst wishing to perform extractions on client samples. At a minimum 4 replicate samples spiked from a separate source standard will be extracted and analyzed with passing results under the analytical method limits. See each analytical method used for those limits.

12.4 The Demonstration of Capability is not considered complete until the analyst has signed a statement saying that they have read, understood, and agreed to follow the AEL-SOP for this method and the associated EPA and/or Standard Methods on which the AEL-SOP was based.

12.5 Initial DOCs must be successfully performed by each analyst in accordance with QM and ADMIN-030.

13.0 Calibration and Standardization

13.1 Not applicable to extraction procedures, however; all support equipment must be maintained and calibrated as to manufacturer's instructions and under the guidance of AEL's Quality Manual. Examples of this are the calibration yearly of the turbovaps digital temperature readout and daily calibration of the balance in the extractions room.

14.0 Procedure

WARNING: Do not breathe solvent vapors. Avoid skin contact with solvents. **Methylene Chloride is a known carcinogen.** Methylene chloride and all other solvents used during extractions have known health risk associated with short and long term exposure. All analysts are required to know these health risks and to take precautions. All analysts are to wear the appropriate personal protective equipment (PPE) to reduce exposure. If as an analyst you are not aware of the health risks, seek out your supervisor who will direct you to the Health and Safety Officer. Work Clean. Work Safe.

14.1 Sample handling

- 14.1.1 Sediment/soil samples - Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks. Note removed objects in comments. (Unless requested by the client, upon which a comment should be written on the extraction benchsheet describing materials left in the extraction sample.)
- 14.1.2 Waste samples - Samples consisting of multiple phases must be prepared by the phase separation method in Chapter Two before extraction. This extraction procedure is for solids only.
- 14.1.3 Dry waste samples amenable to grinding - Grind or otherwise subdivide the waste so that it either passes through a 1-mm sieve or can be extruded through a 1-mm hole. Introduce sufficient sample into the grinding apparatus to yield at least 10 g after grinding.
- 14.1.4 Gummy, fibrous, or oily materials not amenable to grinding should be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. The addition of anhydrous sodium sulfate to the sample (1:1) may make the mixture amenable to grinding.

14.2 Determination of percent dry weight - When sample results are to be calculated on a dry weight basis, (standard practice at AEL is to report all analysis under dry weight, unless otherwise requested) a second portion of sample should be weighed out at the same time as the portion used for analytical determination.

- 14.2.1 **WARNING:** The drying oven should be contained in a hood or vented. Significant laboratory contamination may result from drying a heavily contaminated sample.
- 14.2.2 Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tarred crucible. Dry this aliquot overnight at 105°C. Allow to cool in a desiccator before weighing.
- 14.2.3 A moisture analyzer can be used instead of a drying oven for soil samples, but not for waste or other solid matrices.



14.3 Extraction method for samples expected to contain concentrations of organics and pesticides:

14.3.1 The following steps should be performed rapidly to avoid loss of the more volatile extractables.

14.3.2 Weigh approximately 30 g (or lower gram weight if highly concentrated) of sample into a 400-mL beaker. Record the weight to the nearest 0.1 g.

14.3.3 Samples are mixed with sodium sulfate until a free-flowing consistency is achieved. After addition of sodium sulfate, the sample should be free flowing as would sand through an hourglass. If clumpy, break up until that free-flowing consistency is reached. **Samples must be extracted dried of water.** Water will make a barrier difficult for some solvents to penetrate.

14.3.4 Nonporous, wet, gummy or clay type samples that do not have a free-flowing sandy texture must be mixed with twice the amount (60 g) of anhydrous sodium sulfate, using a spatula. If required, more sodium sulfate may be added until the sample is free flowing.

14.3.5 Add 1.0 mL of the surrogate standard solution to all samples, spiked samples, QC samples, and blanks. **Spike into the sample and not on the surface.**

14.3.5.1 Spike directly into the matrix before adding any extraction solvent. Do not “air spike”. The syringe must be below the surface in both aqueous and soil samples. For soils, add the solvent to the sample as soon as practical after the spike is made. Adding solvent right after each spike can ensure the best spike recoveries. The longer a spike sits on a soil and can evaporate out of the sample, the poorer the recoveries will be.

14.3.5.2 Have a rinse beaker handy and twirl the tip of the syringe into the rinse after each sample is spiked. For soils it may be necessary to wipe the tip of the syringe to remove debris. Wipe using a fresh clean Kimwipe and then rinse. Do not use a paper towel, lab coat, or your fingers as you can cross contaminate or add new contaminant. Paper towels can contain phthalates, etc... Your lab coat, who knows? When it was last washed? Reusing the same Kimwipe can cross contaminate samples as well.

14.3.5.3 Use mnemonic devices so that if spiking is interrupted, it can be seen where you left off by visual cue. If you get pulled away, you will know where you left off. One example is to move the cap of the spike solution vial to the next beaker to be spiked.

14.3.5.4 In the event where you are still not sure if a sample was spiked or not, it is better to double spike than to not spike at all. Just note in the extraction log in the comment column that the sample may be double spiked. It is also worth



noting that on some projects that are high profile, or if the lab is having difficulty with spiking, it may be warranted to have a spike witness for all spiking. The spike witness should be noted on the extraction log when employed.

- 14.3.6 For the sample in each batch selected for spiking, add 1.0 mL of the matrix spiking solution.
- 14.3.7 Immediately after spiking, add 60 mL of the appropriate/recommended extraction solvent or solvent mixture (see Table 1).
 - 14.3.7.1 The method calls for using 60mls with the soil samples. The sample must be covered with solvent. Use more than 60mls if it is necessary.
- 14.3.8 Place the beaker containing the sample into the sonication bath.
- 14.3.9 Extract ultrasonically for 5 minutes in the Branson 8510 sonication bath. (Or other equivalent type baths as listed in section 9)
- 14.3.10 Decant the extract and filter it through a glass powder-funnel that is attached to a clean 200-mL Zymark tube. The funnel should be filled with anhydrous sodium sulfate to dry the extract of any water. Be sure there is enough glass wool or Whatman 1 filter paper in the funnel to prevent any solid portions from entering the Zymark tube.
- 14.3.11 Repeat the extraction two or more times with two additional 60 mL portions of solvent. Decant off the solvent after each ultrasonic extraction. If it appears that there will be more than the turbovap will hold after the second decanting, start blowing down while the third sonication is ongoing, as this will free up some room for the last solvent capture.
- 14.3.12 After the third sonication and after the final decanting, dump the soil on top of the sodium sulfate filled funnel and do a final rinse to get the last of the extract out of the soil, so basically it will be three sonications, followed by a chaser rinse.
- 14.3.13 Do not break the rhythm once extraction begins. Stopping in the middle of an extraction sequence, such as taking lunch, can cause a dip in recoveries. A smooth consistent routine will give good consistent recoveries. Know the best stopping points, such as when first set up but before spiking, or after all three sonications have been collected into the turbovap tubes.
- 14.4 Perform the concentration (if necessary) using the Turbo-Vap Sample Concentrator
 - 14.4.1 Before beginning Turbo-Vap concentration, turn on the Turbo-Vap unit and set the bath temperature to the desired temperature and document the actual temperature in the extraction log. Check the water level in all of the units. The water should be halfway up to the lower set of perforations in the back of the chamber. Make sure that the hood is on to collect all solvent vapor given off by the Turbo-Vap



14.4.2 There are four different options that can be programmed into the Turbo-Vap to stop the concentration process. The Turbo-Vap can be programmed to concentrate until it is manually shut off, to concentrate only for a certain period of time, to concentrate until a sensor indicates the endpoint is reached, or to concentrate for a certain period of time after the sensor endpoint is reached. Ensure that the Turbo-Vap is programmed to stop when a sensor endpoint is reached. If this option is selected, the Turbo-Vap should stop concentration when the sample volume is 1.0 ml. If any of the other options are selected, the sample may go dry.

NOTE: It is the practice here at this lab to not depend on the sensors to end the concentration. All samples are to be watched and manually completed. A dark sample can leave a residue on the tubes that will negate the sensors.

14.4.3 Place the Zymark tube containing the sample extract in the Turbo-Vap. The Turbo-Vap can concentrate up to six samples at one time. Press the start button the appropriate tube to be concentrated. This allows the Nitrogen gas to flow across the sample. Blow down the sample to about 10 ml. Rinse the walls of the thimbles several times to bring back up to about 15 ml. Repeat this at least once more as a residue will form on the thimbles that needs to be "pushed down" the sides.

14.4.4 If a solvent exchange is needed, add 10 ml of the final solvent (normally hexane) once the sample extract has concentrated down to about 10 ml. Do this at least once more also rinsing down the walls of the thimble.

14.4.5 The outside of the condenser may be covered in water droplets. Be sure that they do not drip into the sample extract.

NOTE: If the sample evaporates to dryness, re-extract the entire sample. The loss of analytes can be assumed. In most cases, there should be enough sample to allow for re-extraction.

14.4.6 Concentrate the sample extract to 1ml. If the sample extract will not concentrate to 1.0 ml or less, is very viscous, or is very dark in color, make comments in the extraction logbook, and this sample may need to be diluted up to a higher volume

CAUTION: When the volume of solvent is reduced much below 1 ml, semi volatile analytes may be lost. It is recommended not to go below 0.75 ml and mandated that no samples are to go below 0.5mls during concentration in the turbovaps.

14.4.7 The extract may now be analyzed for the target analytes using the appropriate determinative technique(s) (see Sec. 4.3 of this Chapter). Transfer to a 1 ml vial with a PTFE-lined screw cap or crimp top and labeled appropriately.

14.4.8 Use a reference vial filled with one ml of reference solvent (filled using a 1ml syringe) to comparatively measure the 1ml of extract of each sample. All analysts should have



a study to show that they can accurately measure out one ml of sample extract prior to doing this measurement technique, or they must use calibrated 1ml vials for transferring exactly 1ml to the vials.

14.4.9 As a rule, the extractionist should develop a routine with good habits built in. If you change a step in the routine, talk with the analyst. With good communications, any trends seen by the analyst should be passed on to extraction personnel. The instruments are a clear indicator of the quality of the extractions. It can be determined by sample recoveries if samples are not extracted thoroughly, or are spiked incorrectly, blown down too quickly, take too long to blow down, if blown down to dry, have recoveries that are too good, or have other issues. Telling the analyst of any abnormalities will help immensely in their evaluation of the results. You can also gauge how well you are doing.

14.5 Extract Cleanup Methods: Cleanups in most cases will be dictated by the instrument analyst and sometimes only performed by the instrument analyst. In some cases, such as for PCB (8082) and FL-PRO, cleanup is a mandatory step of processing samples prior to analysis. Whenever using a cleanup in the extraction, make sure to list that on the extraction log, except with 8082 as it is understood that it is part of the extraction process as listed in our SOP. Before doing any cleanups that are not already routine, speak with the analyst first before starting.

14.5.1 EPA 3620C --Florasil Cleanup: The Florisil column allows for a select fractionation of the compounds and will eliminate polar interferences. See SVOC-013 Florisil Cleanup 3620C.

14.5.2 EPA 3630C: Silica Gel Cleanup-strips away polar fatty acids, such as animal fats and grease for FL-PRO analysis. Also, may be used to separate single component organochlorine pesticides from some interferences. See SVOC-033 Silica Gel Cleanup 3630C

14.5.3 EPA 3660B: Copper cleanup removes elemental Sulfur, which interferes with the electron capture gas chromatography of certain pesticides. See SVOC-034 Sulfur Cleanup 3660B.

14.5.4 EPA 3665A: Sulfuric Acid Cleanup (Required for 8082) The use of Sulfuric acid will remove organic interferences which cause problems with the electron capture gas chromatography of certain PCB/Aroclors. Acid cleanup will remove pesticides from the sample extract; therefore, this procedure must only be implemented if PCB/Aroclors are the only analytes of interest. If any pesticides are required for analytical determination, acid clean up must not be performed. See SVOC-014 Acid Clean 3665A.

15.0 Calculations

15.1 Not applicable for extraction methods



16.0 Method Performance

- 16.1 Refer to the determinative methods for performance data. See section 12 concerning the performance of a Demonstration of Capability.

17.0 Pollution Prevention

- 17.1 See Standard Methods section 1100, 22nd Edition – Waste Minimization and Disposal.
- 17.2 See SOP Admin-018 and the AEL Safety Manual.

18.0 Data Assessment and acceptance criteria for quality control measures

- 18.1 The assessment of the data will take place during the analysis methods
- 18.2 If a sample is allowed to evaporate to dryness, the sample will be re-extracted if there is sufficient sample, or the client will be notified to resample.
- 18.2.1 If resampling is not an option, the extract will have solvent added to it to dissolve the residue back into solution and the results will be qualified with a J5 - the data is questionable because of improper laboratory or field protocols.

19.0 Corrective actions for out of control data

- 19.1 Out of control data is not applicable to the extraction procedures.
- 19.2 If there are consistent out of control data problems for a particular method, then the extraction procedure will be reviewed by the supervisor to determine and resolve the problem.
- 19.2.1 Examples would be consistent low (or high) recoveries for surrogates or spikes.

20.0 Contingencies for handling out of control or unacceptable data

- 20.1 All out of control data will be qualified according to SOP Admin-008.
- 20.2 Any nonconformity will be documented on the extraction log so the analysts can transfer all appropriate qualifiers to the bench sheets before reporting the data.

21.0 Waste Management

- 21.1 Refer to SOP for Waste Management (ADMIN-018) for any other questions.
- 21.2 See Standards Method section 1100, 22nd Edition – Waste Minimization and Disposal.



22.0 Maintenance

- 22.1 Change Turbo-Vap bath water routinely.
- 22.2 Check the Nitrogen gas weekly.
- 22.3 Routinely inspect all glassware for scratches, cracks, and possible contamination.
- 22.4 Clean out sonicator baths routinely.

23.0 References

- 23.1 EPA Method 3550B revision 2, Dec 1996
- 23.2 EPA Method 3550C revision 3, Feb 2007
- 23.3 Standards Method section 1090 and 1100, 23rd Edition (On-line edition)
- 23.4 AEL Safety Manual
- 23.5 AEL Quality Manual
- 23.6 ADMIN-049a AEL QM Section 7.0 - Contemporary Equipment List (most recent revision)
- 23.7 ADMIN SOPs
- 23.8 TNI Standards 2016
- 23.9 ISO 17025: 2005 & 2017 Standards
- 23.10 DoD ELAP QSM (Latest revision)

24.0 Tables, Diagrams, flowcharts, and validation data

- 24.1 Validation Data: See the employee files (stored electronically on the network or hardcopy in the laboratory's QA office) for the individuals for an acceptable initial demonstration of capability, which serves as validation data for this method in AEL.



24.2 Table 1

Table 1

**SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS
(For methods below, initial extraction solvent is methylene chloride or methylene chloride/acetone mix)**

| Determinative method (mL) ^a | Extraction pH | Exchange solvent required for analysis | Exchange solvent required for cleanup | Volume of extract required for cleanup (mL) | Final extract volume for analysis |
|--|---------------|--|---------------------------------------|---|-----------------------------------|
| 8081 | as received | hexane | hexane | 1.0 | 1.0 |
| 8082 | as received | hexane | hexane | 1.0 | 1.0 |
| 8270 ^c | as received | none | - | - | 1.0 |
| 8141 | as received | hexane | hexane | - | 1.0 |
| DRO | <2 | none | - | - | 1.0 |

^a For methods where the suggested final extract volume is 10.0 mL, the volume may be reduced to as low as 1.0 mL to achieve lower detection limits.

^c The specificity of GC/MS may make cleanup of the extracts unnecessary. Refer to Method 3600 for guidance on the cleanup procedures available if required



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STANDARD OPERATING PROCEDURE

For

Method 8270C/D/E SIM

SEMIVOLATILE ORGANIC COMPOUNDS

BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) IN SELECTIVE ION MODE
(SIM)





SOP Revision Log for AEL SOP SVOC-028

| Revision Number | Revision Date | Reason for Revision | Section(s) and Page(s) Revised |
|-----------------|---------------|--|--|
| Revision 00 | 3/10/05 | Initial Creation | All sections affected |
| Revision 01 | 2/17/07 | Revisions throughout, update language and definitions. | All sections affected |
| Revision 02 | 7/9/10 | Revisions and re-writes throughout. Added revision log, section 22 maintenance, renumbered sections 22, and 23. | All sections affected |
| Revision 03 | 2/7/11 | Added section 1.3 and 11.4 to add allowance for acid as well as base-neutral extraction so that PAHs can be extracted in conjunction with FL-PRO samples, revised 6.24 for PQL definition, Added to section 24.1 new personnel for method validation. | Sections 1.3, 6.24, 11.4, 24.1 |
| Revision 04 | 9/30/14 | Revisions throughout, update language and definitions. Update equipment list. Allows for reduced volume extractions. Update control limits. Update validating analysts section 24. | All sections affected |
| Revision 05 | 2/25/15 | Instrument J7H added to Jacksonville instrumentation list. Sec 9.3.6. Stock standards are purchased from an approved vendor. Reference to Crescent and CPI standards removed. Section 10. Add GC/MS instrument operating conditions for QP2010Plus. Section 13.1.2, Update validating analyst Sec 24 | Section 9.3.6, 10, 13.1.2, 24 |
| Revision 06 | 4/30/15 | SIMS operating condition added as table 5. | Section 24 |
| Revision 07 | 4/14/17 | Update method to include requirements of tuning with DFTPP, specific surrogates, and checking DDT breakdown, when performing for DoD clients under DoD QSM 5.1 criteria. Update setting of RRT. Update method validating analyst. Update references and tables to latest editions (DoD QSM 5.1) | Sections 1.2, 4.1, 10.16, 10.8.2, 12.3, 12.7.1 13.2, 13.3, 13.5.6, 23, 24.1, 24.2 tables 2 - 5 |
| Revision 08 | 4/09/18 | Revision, re-writes throughout and addition of 1,4-Dioxane. | All sections affected. |
| Revision 09 | 2/28/19 | Updated target reference ions. Update to include criteria for 8270E. Update to DoD QSM 5.2 criteria. | 24.3 Table 1 |
| Revision 10 | 4/20/2020 | Re-writes throughout, update to DoD QSM 5.3 criteria, and removal of 1,4-Dioxane soils. | All sections affected. |
| Revision 11 | 11/06/2020 | Revise to edit surrogate level 1 concentration from 0.1 ug/ml to the correct value of 1.0ug/ml | 10.7.2.1 Initial Cal. Standards Table |
| Revision 12 | 12/22/2020 | Include rules for relative error and its calculation. Add procedure for processing Appendix 9 compounds (1,4-Dioxane) | Section 13.5, 14.2.6.3 |
| Revision 12 | 3/21/2022 | Reviewed with updated references to QSM most recent version no other changes | All Sections |



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| Revision Number | Revision Date | Reason for Revision | Section(s) and Page(s) Revised |
|-----------------|---------------|---|--------------------------------|
| Revision 12 | 3/27/2023 | Reviewed with updated references to equipment list, and data validation. Updated QSM tables to most recent version. | Sections 9, 12, 13, and 24. |



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1.0 Identification of Test Method

1.1 METHODS 8270C, D, & E; SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) IN SELECTIVE ION ACQUISITION MODE (SIM) (as opposed to full scan acquisition mode).

1.2 This SOP deviates from the EPA 8270 referenced method in the following areas:

NOTE: These deviations from the referenced test method are the best conditions for our instrumentation and represent improved performance over the referenced test method conditions.

1.2.1 The mass spectrometer uses a scan range of 40 to 450 amu at a rate of 0.38 seconds per scan versus the referenced test method range of 35 to 500 amu and a scan rate of 1 second per scan. The scan range and scan rate of this SOP give better fits for the compounds normally analyzed under both 8270 test methods.

1.2.2 Standards are all purchased from certified vendors and are not made from stock reference materials.

1.2.3 Instrument chromatographic conditions have been optimized for the column and analytes of interest and do not match exactly those recommended in the method. These deviations from the referenced test method are the best conditions for our instrumentation and represent improved performance over the referenced test method conditions.

1.2.4 Tuning when in SIMs mode only is not required (except when performing under DoD criteria for DoD client work).

1.3 In house studies have shown that Polyaromatic Hydrocarbons do not require pH adjustment for extraction; therefore, PAH samples can be extracted under acid as well as base or neutral conditions. This allows for the extracts, when silica gel treated, to be used for both PAH analysis and FL-PRO analysis as long as QC is extracted that meet both method's acceptance criteria and all QC passes through all the processes that samples pass through. Also allowed per method is a volume less than 1 liter. New MDLs at this lesser volume are comparable to those of 1-liter volumes and will be in use for these lower volume extractions.

2.0 Applicable Matrix or matrices

2.1 This method is applicable to nearly all types of samples, regardless of water content, including ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, solid materials, and sediments.

3.0 Detection Limit

3.1 The method detection limit is determined in accordance with AEL SOP ADMIN-012, referencing 40CFR136, appendix B.



3.2 For the current MDLs, see the electronic version specific for the method, instrument, and matrix in the “AEL-QA” folder on the lab server.

4.0 Scope and Application, including components to be analyzed:

4.1 Analytes:

| PARAMETER | CAS NUMBER |
|------------------------|------------|
| Acenaphthene | 83-32-9 |
| Acenaphthylene | 208-96-8 |
| Anthracene | 120-12-7 |
| Benz(a)anthracene | 56-55-3 |
| Benzo(b)fluoranthene | 205-99-2 |
| Benzo(k)fluoranthene | 207-08-9 |
| Benzo(g,h,i)perylene | 191-24-2 |
| Benzo(a)pyrene | 50-32-8 |
| Carbazole | 86-74-8 |
| Chrysene | 218-01-9 |
| Dibenz(a,h)anthracene | 53-70-3 |
| Dibenzofuran | 132-64-9 |
| Fluoranthene | 206-44-0 |
| Fluorene | 86-73-7 |
| Indeno(1,2,3-cd)pyrene | 193-39-5 |
| 2-Methylnaphthalene | 91-57-6 |
| 1-Methylnaphthalene | 90-12-0 |
| Naphthalene | 91-20-3 |
| Phenanthrene | 85-01-8 |
| Pyrene | 129-00-0 |
| 1,4-Dioxane | 123-91-1 |

- 4.2 Method 8270 C, D, & E can be used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.
- 4.3 See Table 1 in section 24.3 for a list of compounds and their characteristic ions that have been evaluated.
- 4.4 This is a gas chromatographic/mass spectrometry (GC/MS) method applicable to the determination of the compounds listed in Section 4.0 in municipal and industrial discharges as provided under 40 CFR Part 136.1.



- 4.5 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

5.0 Summary of Method

- 5.1 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation (SVOC-001; 3510C, SVOC-002; 3550B, and SVOC-003; 3580A).
- 5.2 The semi volatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.
- 5.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

6.0 Definitions

See also AEL ADMIN SOP-039 Laboratory Definitions

- 6.1 Extraction Batch – A group of field samples with similar matrices, which are prepared at the same time in the same location using the same procedure and processed as a unit. A Method Blank, a Laboratory Control Sample, a Matrix Spike, and a Duplicate Matrix Spike must accompany each extraction batch of 20 or fewer field samples.
- 6.2 Continuing Calibration Verification (CCV) – A known interference free matrix spiked with a known concentration (near the mid-point of the Initial Calibration) of the target analytes. The CCV is analyzed at the beginning of a 12-hour analytical run and is used to verify that the instrument calibration is in control before and after sample analysis. A CCV, with a criterion of 50-150% is required at the end of the 12-hour analytical run ONLY for DOD samples.
- 6.3 DoD (DOD): Acronym for Department of Defense.
- 6.4 Initial Calibration (ICAL) – Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method.
- 6.5 Calibration Curve – A calibration or standard curve is a curve that plots known standard concentrations of an analyte versus the instrument response for that analyte.
- 6.6 Initial Calibration Verification (ICV) – A known interference free matrix spiked with a



known concentration (near the mid-point of the Initial Calibration) of the target analytes. ICV standards are made from a stock solution that is different from the stock used to prepare calibration standards. This standard is analyzed immediately after the calibration to confirm the usability of the calibration.

- 6.7 Instrument Blank (IB) – An instrument blank is an aliquot of the method solvent, containing no analytes of interest. The purpose of an IB is to ensure that the analytical system is free from contamination associated with the instrument analysis. An IB also provides one way of determining the level of noise and baseline rise attributable solely to the analytical system, in the absence of any other analytes or non-analytical related contaminants. The blank should contain the internal standard.
- 6.8 Internal Standard (IS) – An internal standard is pure analytes added to a sample, extract, or standard solution in a known amount. The IS is used to measure the relative responses of other method analytes that are components of the same sample of solution. The internal standard must be an analyte that is not a sample component.
- 6.9 Method Blank (MB); also referred to as Laboratory Reagent Blank (LRB) – An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.
- 6.10 Laboratory Control Sample (LCS); also referred to as Laboratory Fortified Blank (LFB) – A known interference free matrix spiked prior to sample extraction with a known concentration of standard. The LCS is analyzed exactly like a sample; the purpose of the LCS is to monitor analytical control for the batch. Percent recoveries are calculated for each of the analytes.
- 6.11 Laboratory Control Sample Duplicate (LCSD) – A second known interference free matrix spiked prior to sample extraction with a known concentration of standard. Analyses of duplicates indicate precision associated specifically with the laboratory procedures, removing any associated variables that might occur during sample collection, preservation, or storage procedures.
- 6.12 Matrix Spike/Matrix Spike Duplicate (MS/MSD); also referred to as Laboratory Fortified Matrix (LFM) – An aliquot of a client supplied sample that is chosen at random, to which known quantities of the method analytes are added in the laboratory. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the values in the MS corrected for the background concentration. Matrix Spikes are analyzed with every batch or per 20 samples.
- 6.13 Matrix – The substrate (e.g. water, soil, etc.), which may contain the analyte of interest.
- 6.14 Liquid Sample - A sample classified as a groundwater, surface water, wastewater or other water-soluble liquid.



6.15 Representative Sample: A well-mixed aliquot of sample that constitutes an accurate representation of contents within the container. Methods used to achieve a representative sub-sample are described below:

6.15.1 Aqueous/Liquid samples.

6.15.1.1 Sample is shaken until homogeneous and then poured or pipetted into appropriate container.

6.15.2 Soil Samples.

6.15.2.1 If container size is sufficient, sample is mixed within until homogeneous. If container size is insufficient, the entire sample is transferred to an appropriate container and mixed.

6.15.2.2 Miscellaneous Solid Samples. Sample is crushed, pulverized, shaken and stirred as appropriate to ensure the aliquot used for analysis represents the entire contents of the original sample container as accurately as possible.

6.16 Sample Duplicate (DUP) - A second aliquot of a client supplied sample. The sample duplicate is analyzed exactly as the client supplied sample and is used to determine whether the laboratory is capable of obtaining reproducible results.

6.17 Stock Standards Solution – A concentrated solution of one or more target analytes at a known concentration, purchased from a reputable commercial vendor, and having Certificates of Analysis. Stock standard solutions are used to prepare working calibration standards. Stock standards once opened must be replaced after 1 year or sooner if routine QC indicates a problem.

6.18 Working Calibration Standard (WS) – A solution of all the target analytes at a known concentration prepared either from one or more intermediate calibration standards and/or from one or more stock standard solutions. Working standards once made must be replaced after 6 months or sooner if routine QC indicates a problem.

6.19 Analysis Window – Samples are analyzed in a time frame referred to as a “window.” The window is initiated with the analysis of the continuing calibration verification (CCV) standard. If the CCV passes the specific criteria, then samples are analyzed until the 12-hour time limit expires. Before more samples can be analyzed, a new window must be opened.

6.20 DOD Analysis Window – Samples are analyzed in a time frame referred to as a “window.” The window is initiated with the analysis of the DFTPP Tune standard. If the Tune passes specific criteria, a 12-hour analysis window is opened at the time of the injection of the DFTPP. Next, a continuing calibration verification (CCV) standard is analyzed. If both of these analyses pass their specific criteria, then samples are analyzed until the 12-hour time limit expires. Before more samples can be analyzed, a new window must be opened.

6.21 Decafluorotriphenylphosphine (DFTPP) Tune Standard – Used to verify mass spectral



instrument performance (mass and ion abundance criteria) for semi volatile analysis.

- 6.22 Surrogate Analyte (Surr) – A surrogate analyte is an organic compound that is similar to the analytes of interest in chemical composition, extraction characteristics, and chromatography, but is not normally found in environmental samples. The purpose of the surrogate analyte is to evaluate the preparation and analysis of the samples. These compounds are spiked into blanks, standards, samples, and matrix spiked samples prior to analysis. Percent recoveries are calculated for each surrogate and are used to evaluate the method performance.
- 6.23 GC/MS - used as an abbreviation for gas chromatograph/mass spectrometer. When MS abbreviated alone, always defined as matrix spike, when MS used in conjunction with GC as in GC/MS, always defined as mass spectrometer.
- 6.24 Selective Ion Monitoring (SIM) – Mass spectrometry technique where ions resulting from fragmentation are selectively monitored, therefore excluding other ions. The technique enhances sensitivity as compared to full scan analysis. Because the analysis results in significantly less mass spectral information, this gain in sensitivity is made at the expense of analyte selectivity. Therefore, the use of SIM results in significantly lower instrument detection limits, but increases the uncertainty associated with the analysis.
- 6.25 Case Narrative (CN) – A case narrative is simply a means of describing exactly what transpired with the samples during the analytical process. Case narratives are required for variances that occur within a project.
- 6.26 Non-Conformity Form (NCF) – A form which will be completed and processed for each QC failure or deviation from normal protocol that occurs outside the scope of normal operation as defined by the AEL QM Section 10, AEL SOP Admin-016, and Method SOP.
- 6.27 Semi-volatile department abbreviations used in sample preparation logbooks, data printouts and other Semi-volatile areas:
- 6.27.1 RR/RA – Rerun/Reanalyze
 - 6.27.2 CF – Confirmation
 - 6.27.3 NR – Not a Real Hit
 - 6.27.4 NAP – Not a Peak
 - 6.27.5 DNC – Does Not Confirm
 - 6.27.6 STR – Straight/No Dilution
 - 6.27.7 NT – Not Target
 - 6.27.8 WRT – Wrong Retention Time
 - 6.27.9 DNR – Do Not Report



6.27.10 BDL –Below Detection Limit

6.27.11 FH – Fails High

6.27.12 FL – Fails Low

6.27.13 DF – Dilution Factor

6.27.14 DIL – Dilution

6.27.15 STD – Standard

6.27.16 IS – Internal Standard

6.27.17 Surr – Surrogate

6.27.18 OOT – Out of Tune/CCV Window

6.27.19 WS – Working Standard

6.28 Safety Data Sheets (SDS) Safety Data Sheets (SDS) – Written information provided by vendors concerning a chemical’s toxicity, health hazards, physical properties, fire, and reactivity data, including storage, spill, and handling precautions.

6.29 Limit of Detection (LOD) – The LOD is **not** synonymous with the MDL. The LOD is an estimate of the minimum amount of a substance that an analytical process can reliably detect with a high level of confidence (99% Confidence; that is a false negative rate of 1%). The LOD is at the level of the MDL. The LOD must go through all the same processes that a sample will go through and be detected above instrument noise level.

6.30 Limits of Quantitation (LOQ) – The minimum levels, concentrations, or quantities of a target variable (e.g. target analyte) that can be reported with a specific degree of confidence. It is also the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DOD work the LOQ will be set at or above the concentration of the lowest initial calibration standard. For DOD work the LOQ will be the concentration at which the PQL is verified. The LOQ can equal the PQL, but is not synonymous with the PQL.

6.31 Limits of Quantitation (LOQ) Verification – LOQ verifications are a spiked clean matrix sample that must go through all the same processes which regular samples will go through, and be within the precision and bias acceptance criteria of the method. The LOQ verifications are spiked at the concentrations of the LOQ.

6.32 Method Detection Limit (MDL) – The MDL is an estimate of the minimum amount of a substance that an analytical process can readily detect. The MDL is the minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined most



often as 3.14 times the standard deviation of a low level, seven replicate study; however, it can be determined for some methods as the lowest increment measurable with confidence. MDLs are determined for each analyte, matrix, prep method, cleanup method, analysis method, and instrument. (See each method's requirements). The MDL is one way to establish a Detection Limit, not a Limit of Detection.

6.33 Method Detection Limit (MDL) Verification – MDL verifications are spiked clean matrix samples that must go through all the same processes that a regular sample will go through and be detected above instrument noise level. For labs accredited under DOD ELAP, the MDL verifications must be performed immediately after the initial MDL study and on a quarterly basis thereafter. For labs accredited under NELAC (TNI) Standards only, MDL verifications are to be performed immediately after the initial MDL study and on a yearly basis thereafter..

6.34 Practical Quantitation Level (PQL); also know as the Method Reporting Limit (MRL or RL) – the lowest calibration standard or lowest quantitation level for the method and matrix. The concentration below which data is to be qualified as having less certainty. PQLs are at a concentration greater than that of the MDL.

6.35 Qualifier Codes (For Florida and FDEP work)

6.35.1 A - Value reported is the mean (average) of two or more determinations. This code shall be used if the results of two or more discrete and separate samples are averaged. These samples shall have been processed and analyzed (e.g. laboratory replicate samples, field duplicates, etc.) independently. Do not use this code if the data is the result of replicate analyses on the same sample aliquot, extract or digestate. Under most conditions, replicate values shall be reported as individual analyses.

6.35.2 I - The reported Value is between the laboratory method detection limit (MDL) and the laboratory practical quantitation limit (PQL).

6.35.3 K- Off scale low.

6.35.4 L- Off scale high. Use if reporting above the acceptable level of quantitation.

6.35.5 U- Indicates that a compound was analyzed for but not detected. The value associated with the qualifier will be the MDL.

6.35.6 V- Indicates that the analyte was detected in both the sample and the associated method blank. NOTE: The method blank value **cannot** be subtracted from the associated sample to give a result. The sample result will be reported as is with the "V" qualifier.

6.35.7 H - Value based on field kit determination; results may not be accurate. This code shall be used if a field-screening test (i.e. field gas chromatograph data, immunoassay, vendor-supplied field kit, etc.) was used to generate the value and



the field kit or method has not been recognized by the Department as equivalent to laboratory methods.

- 6.35.8 O - Sampled, but analysis lost or not performed. NOTE: if reporting data to STORET, a numerical value must be entered. Such values are not meaningful and shall not be used.
- 6.35.9 Q - Sample held beyond the accepted holding time. This code shall be used if the value is derived from a sample that was prepared and/or analyzed AFTER the approved holding time restrictions for sample preparation and analysis.
- 6.35.10 Y - The laboratory analysis was from an unpreserved or improperly preserved sample. The data may not be accurate.
- 6.35.11 REJ - Data is rejected and should not be used. Some or all of the quality control data for the analyte were outside criteria, and the presence or absence of the analyte cannot be determined from the data.
- 6.35.12 NAI - Not analyzed due to interference.
- 6.35.13 J - Estimated value; value not accurate.
- 6.35.13.1 This code shall be used in the following instances:
- 6.35.13.1.1 "1" Surrogate recovery limits have been exceeded;
 - 6.35.13.1.2 "2" No known quality control criteria exists for the component;
 - 6.35.13.1.3 "3" The reported value failed to meet the established quality control criteria for either precision or accuracy;
 - 6.35.13.1.4 "4" The sample matrix interfered with the ability to make any accurate determination; or
 - 6.35.13.1.5 "5" The data is questionable because of improper laboratory or field protocols (e.g. composite sample was collected instead of a grab sample).
- 6.35.13.2 A "J" value shall be accompanied by justification for its use (ex. J(4)).
- 6.35.13.3 A "J" value shall not be used if another code applies (ex. K, L, M, T, V, Y, PQL).
- 6.35.14 If more than one code applies, and the data is to be entered into STORET, only one code shall be reported. The code shall be selected based on the following hierarchy: REJ, NAI, O, Y, V, H, J, B, K, L, M, PQL, T, Z, A.



7.0 Interferences

Note: All references to other methods are in reference to EPA SW-846 methods

7.1 Refer to Methods 3500 (Sec. 3.0, in particular) and 8000, for a discussion of interferences.

7.2 Sources of interference in this method can be grouped into three broad categories.

7.2.1 Contaminated solvents, reagents, or sample processing hardware.

7.2.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

7.2.3 Compounds extracted from the sample matrix to which the detector will respond.

7.3 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running method blanks. Reagents of sufficient quality are used to reduce this possibility and purchased in accordance with ADMIN-013.

7.4 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinsing with tap water, followed by organic-free reagent water. Drain the glassware and dry it in an oven at 130°C for several hours, or rinse with methanol or acetone and drain. Store dry glassware in a clean environment.

7.5 The chromatographic conditions described allow for a unique resolution of the specific PAH compounds covered by this method. Other PAH compounds, in addition to matrix artifacts, may interfere.

7.6 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled.

7.7 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is rinsed automatically with solvent between sample injections. Whenever an unusually concentrated sample (10 times the upper limit of the curve) is encountered, it should be followed by the analysis of solvent to check for cross-contamination.

8.0 Safety

8.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

8.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations



regarding the safe handling of the chemicals specified in this method. A reference file of Safety Data Sheets (SDS) should be made available to all personnel involved in the chemical analysis. These are stored in the common areas of the labs.

8.3 Refer to the AEL Chemical Hygiene Plan and Safety Manual for safety precautions and for the Hygiene Plan and Emergency Response Plan.

8.4 See Standard Methods, 22nd / Online Edition, Section 1090 Laboratory Occupational Health and Safety.

9.0 Equipment and Supplies

9.1 See SOP SVOC-001; 3510C, SVOC-002; 3550B, and SVOC-003; 3580A.

9.2 Gas Chromatograph (GC)/Mass Spectrometer (MS) system:

Note: SOPs are updated on a set schedule and as a result may not reflect new additions or updates to laboratory equipment inventory at each site as the year progresses. For real-time equipment tracking, please reference the ADMIN-049a AEL QM Section 7.0 - Contemporary Equipment List (most recent revision) link on the intranet SOP system for the current listing by room location and letter designation for each piece of major equipment (by make, model, and serial number) and a full inventory of all major pieces of equipment in each lab.

9.2.1 Gas Chromatograph (Shimadzu model: GC-2010 or GC-2010 Plus and Agilent model 6890N) – An analytical system complete with gas chromatograph; suitable for sample introduction and all required accessories, including detectors, column supplies, recorder, gases, and syringes. Each Shimadzu GC shall be mounted with the Shimadzu Auto sampler; Model # AOC-20i and the Agilent GC shall be mounted with the Agilent 7683 for direct injection into the injector and onto the column.

9.2.1.1 Jacksonville: Shimadzu: GC model 2010 (serial number 6091951425), GC model 2010-Plus (serial number 10681550 and C7062400216). Agilent GC model 6890N (serial number US10623036).

9.2.2 Auto sampler Syringe – 10uL.

9.2.3 Chromatographic Column – Phenomenex Zebron ZB-Semi Volatiles 30m x 0.25mm x 0.25um, or equivalent.

9.2.4 Detector - Mass Spectrometer (MS) (Shimadzu QP2010SE, or QP2010 and Agilent 5973).

9.2.5 Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Decafluorotriphenylphosphine (DFTPP), which meets the criteria in Table 2 Section 24 when 2uL of the GC/MS tuning standard is injected through the GC



(50ng of DFTPP).

9.2.6 GC/MS interface (Direct) – Any GC-to-MS interface that gives acceptable calibration points at 50ng per injection for each compound of interest and achieves acceptable tuning performance criteria may be used. For a narrow-bore capillary column, the interface is usually capillary-direct into the mass spectrometer source.

9.3 Data System (Shimadzu Lab Solutions – version GCMS solution 4.2 and 4.3. Agilent – Environmental Chemstation/MSD Chemstation version E.02.02.1431) – A data system used for measuring and storing peak heights and peak areas. The computer system should be interfaced to the mass spectrometer. The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software searches any GC/MS data file for ions of a specific mass and can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software allows integrating the abundances in any EICP between specified time and scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library is available.

9.4 Volumetric flasks, Class A - Appropriate sizes with ground-glass stoppers, for preparation of standards.

9.5 Auto sampler Vials – 2.0 mL glass with polytetrafluoroethylene (PTFE) - lined screw caps.

9.6 Standard Solution Storage Containers – 10-20mL amber glass vials with Teflon lined screw caps.

9.7 Pasteur Pipettes – Glass, disposable.

9.8 Micro-Syringes –10 μ L, 25 μ L, 50 μ L, 100 μ L, 250 μ L, 500 μ L, and 1000 μ L.

10.0 Reagents and Standards

Note: Although sources of the reagents and standards noted in this SOP may be provided, they may also change based on availability, quality, and cost. The use of a different source or concentration is acceptable without modification of the procedures, provided the products are equivalent. Reagent grade or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Note: All reagents and standards must be labeled with their unique ID, the name of the material, the concentration, the date prepared, and the expiration date.

Note: Stock standard solutions are ordered from NELAC (TNI) approved vendors. Stock standards are received from the vendor in sealed amber ampoules. Once opened, store the stock standard solutions in amber vials with Teflon screw tops. All stock standards are stored at -10°C to -20°C or



0°C to 6°C and protected from light (unless otherwise instructed by the manufacturer). Check stock standards frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards. Opened stock standards should be replaced after 6 months or sooner, if comparison with check standards indicates a problem.

Note: Prior to making any laboratory prepared standards, allow the stock standard to warm to room temperature. Store intermediate and working standards in screw-top vials at -10°C to -20°C or 0°C to 6°C and protect from light. Working standards should have an expiration date of six months (unless the manufacturer's expiration date is sooner). Allow working standards to warm to room temperature prior to use.

- 10.1 Reagents and Standards are ordered and tracked internally in accordance with SOPs ADMIN-013 and ADMIN-031.
- 10.2 The mixing of reagents shall be tracked in a reagent logbook kept in the Extractions room. The mixing of intermediate and/or working standards shall be tracked in a standard logbook kept in the Semi-Volatiles room. Both logbooks will contain the following information:
 - 10.2.1 For parent material:
 - 10.2.1.1 The manufacturer lot number.
 - 10.2.1.2 The manufacturer name.
 - 10.2.1.3 The chemical name and/or chemical description.
 - 10.2.1.4 The expiration date.
 - 10.2.1.5 The AEL receiving lot number.
 - 10.2.2 For any laboratory prepared reagents and/or standards:
 - 10.2.2.1 The recipe (the amount of parent material used and how the standard was mixed) is included in the logbook.
 - 10.2.2.2 The creation date.
 - 10.2.2.3 The expiration date.
 - 10.2.2.4 The standard concentration.
- 10.3 Reagents:
 - 10.3.1 Reagent grade or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is



first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.3.2 Methylene chloride, hexane, acetone, purge and trap methanol – pesticide grade or better in accordance with ADMIN-013.

10.3.3 Pentafluorotributylamine (PFTBA) – Calibration gas supplied with the MS, using in tuning the MS.

10.4 Stock standards - Stock solutions purchased as certified solutions. Opened stock standards must be replaced after 1 year or sooner; if comparison with check standards indicates a problem (common problems occur from degradation or evaporation of the standard). Certificates of analysis are stored in the SVOC lab in accordance with Quality Manual 5.0. Store purchased standards according to manufacturer specifications. All purchased stock standards solutions must be replaced after reaching the manufacturer's expiration date assigned to the standard.

Note: Alternative standards from the ones listed below may be used, provided they are from a certified vendor.

10.4.1 Primary Stock Standards

10.4.1.1 2000ug/mL 8270 Calibration Mix 5 (Restek C/N 31995)

10.4.1.2 2000ug/mL Carbazole Solutions (O₂SI C/N 010239-03)

10.4.1.3 1000ug/mL Dibenzofuran Solution (O₂SI C/N 010067-01)

10.4.2 1,4-Dioxane Primary Stock Standards

10.4.2.1 40,000ug/mL 1,4-Dioxane (Phenova C/N ALO-130054)

10.4.3 Secondary Stock Standards

10.4.3.1 2000ug/mL Custom PAH Plus Mix (Phenova C/N ALO-130250)

10.4.3.2 1000ug/mL Custom SV Mix (Phenova C/N ALO-130250)

10.4.4 1,4-Dioxane Secondary Stock Standards

10.4.4.1 2000ug/mL 1,4-Dioxane Standard (Restek C/N 30287)

10.4.5 Surrogate Stock Standards

10.4.5.1 5000ug/mL B/N Surrogate Mix (Restek C/N 31086)

10.4.5.2 2000ug/mL SoM01.1 Deuterated Monitoring Compound Mix Sim Compounds (DMCs) (Restek C/N 33913) – DoD samples only



10.4.6 Internal Stock Standard

10.4.6.1 4000ug/mL SV Internal Standard Mix (Restek C/N 31006)

10.4.7 1,4-Dioxane Internal Stock Standard

10.4.7.1 1,4-Dioxane-d8 Standard (Restek C/N 30614)

10.4.8 DFTTP Stock Standards (DOD requirement only)

10.4.8.1 1000ug/mL GC/MS Tuning Mixture (Restek C/N 31615)

10.5 Laboratory Prepared Standards

10.5.1 All laboratory prepared standard solutions must be replaced after 1 year or sooner if routine QC indicates a problem or the method requires a shorter expiration date. An assigned expiration date of a lab prepared standard cannot exceed the manufacturer's expiration date for any component used in the standard formulation.

10.5.2 Extraction Spiking Standards

10.5.2.1 Surrogate Spike – Prepared by diluting 200uL of the 5000ug/mL B/N Surrogate Mix to a final volume of 100mL in Acetone. The surrogate spike concentration is 10.0ug/mL. 1.0mL of the surrogate spike will be added to each sample and QC sample before the extraction.

10.5.2.2 DoD Surrogate Spike - Prepared by diluting 100uL of the 5000ug/mL B/N Surrogate Mix and 250uL of the 2000ug/mL Deuterated Monitoring Compound Mix Sim Compounds to a final volume of 50mL in Acetone. The surrogate spike concentration is 10.0ug/mL. 1.0mL of the surrogate spike will be added to each sample and QC sample before the extraction.

10.5.2.3 LCS/MS/MSD Spike – Prepared by diluting 125uL of 2000ug/mL PAH Plus Methyl naphthalenes and 250uL 1000mg/L Custom SV Mix Solution up to 50mL with Acetone. The LCS/MS/MSD Spike concentration is 5.0ug/mL. 1.0mL of the spike will be added to each QC sample before the extraction.

10.5.2.4 1,4-DX LCS/MS/MSD Spike – Prepared by diluting 125uL of 2000ug/mL 1,4-Dioxane Standard up to 50mL with Methanol. The 1,4-DX spike concentration is 5.0ug/mL. 1.0mL of the 1,4-DX spike will be added to each QC samples before the extraction.

10.5.2.5 1,4-DX Isotope IS – Prepared by diluting 125uL of 2000ug/mL 1,4-dioxane-d8 standard up to 50mL with Methanol. The 1,4-DX Isotope IS concentration is 5ug/mL. 1.0mL of the 1,4-DX Isotope IS will be added



to each sample and QC sample before extraction.

- 10.5.3 Tune Standard (DOD requirement only) – Prepared by diluting 50uL of the 1000ug/mL GC/MS Tuning Mixture and 50uL of the 2mg/mL 4,4—DDT & Endrin Standard to a final volume of 1.0mL DCM. Final concentration of the tune standard is 50ug/mL.
- 10.5.4 PAH Internal Standard (PAH IS) – Prepared by diluting 200uL of 4000ug/mL SV Internal Standard Mix up to 2.0mL with DCM. The IS concentration is 400ug/mL. 10uL of the IS will be added to each sample and QC sample before analysis.
- 10.5.5 1,4-Dioxane Isotope Internal Standard (1,4-DX IS) - Prepared by diluting 250uL of 2000ug/mL 1,4-dioxane-d8 standard up to 1.0mL with DCM. The 1,4-DX Isotope IS concentration is 500ug/mL. 10uL of the 1,4-DX IS will be added to each sample and QC sample before analysis.
- 10.6 Working Standard – Using stock standard, prepare a working standard, as needed, which contains the compounds of interest, either singly or mixed together. The working standard should be replaced at least every 6 months.
- 10.6.1 PAH Primary Working Standard (1° WS) – Prepared by diluting the following to a final volume of 5.0mL in DCM. The final concentration of the PAH 1° WS is 100ug/mL:
- 10.6.1.1 250uL of the 2000ug/mL 8270 Calibration Mix #5,
 - 10.6.1.2 250uL of the 2000ug/mL Carbazole Solution,
 - 10.6.1.3 250uL of the 2000ug/mL SOM01.1 Deuterated Monitoring Compound Mix Sim Compounds,
 - 10.6.1.4 500uL of the 1000ug/mL Dibenzofuran Solution,
 - 10.6.1.5 100uL of the 5000ug/mL B/N Surrogate Mix.
- 10.6.2 PAH Secondary Working Standard (2° WS) – Prepared by diluting the following to a final volume of 5.0mL in DCM. The final concentration of the PAH 2° WS is 100ug/mL:
- 10.6.2.1 250uL of the 2000ug/mL Custom PAH Plus Mix,
 - 10.6.2.2 250uL of the 2000ug/mL SOM01.1 Deuterated Monitoring Compound Mix Sim Compounds,
 - 10.6.2.3 100uL of the 5000ug/mL B/N Surrogate Mix,
 - 10.6.2.4 500uL of the 1000ug/mL Custom SV Mix.



10.6.3 1,4-DX Primary Working Standard (1° WS) – Prepared by diluting the following to a final volume of 10.0mL in DCM. The final concentration of the 1,4-DX 1° WS is 20ug/mL 1,4-DX;100ug/mL B/N Surrogate:

10.6.3.1 5uL of the 40,000ug/mL 1,4-Dioxane,

10.6.3.2 200uL of the 5000ug/mL B/N Surrogate.

10.6.4 1,4-DX Secondary Working Standard (2° WS) – Prepared by diluting the following to a final volume of 10.0mL in DCM. The final concentration of the 1,4-DX 1° WS is 20ug/mL 1,4-DX;100ug/mL B/N Surrogate:

10.6.4.1 100uL of the 2000ug/mL 1,4-Dioxane Standard,

10.6.4.2 200uL of the 5000ug/mL B/N Surrogate.

10.7 Initial Calibration (ICAL) Standards

10.7.1 8270 PAH Calibration Standards – Prepared as described below in DCM.

10.7.1.1 Typical 8270 PAH Initial Calibration Standards

| ICAL Level µg/mL | Volume of 8270 PAH 1° WS used | Final Volume (mL) | Amount of PAH IS to Use* | Concentration of the IS |
|-------------------------|-------------------------------|-------------------|--------------------------|-------------------------|
| ICAL 1 0.025µg/mL*** | 50 µL of ICAL 4** | 1.0mL DCM | 10 µL | 4µg/mL |
| ICAL 2 0.05µg/mL | 100 µL of ICAL 4** | 1.0mL DCM | 10 µL | 4µg/mL |
| ICAL 3 0.2µg/mL | 2 µL | 1.0mL DCM | 10 µL | 4µg/mL |
| ICAL 4 0.5µg/mL | 5 µL | 1.0mL DCM | 10 µL | 4µg/mL |
| ICAL 5 1.0µg/mL | 10 µL | 1.0mL DCM | 10 µL | 4µg/mL |
| ICAL 6 2.0µg/mL | 20 µL | 1.0mL DCM | 10 µL | 4µg/mL |
| ICAL 7 5.0µg/mL | 50 µL | 1.0mL DCM | 10 µL | 4µg/mL |
| ICAL 8 50µg/mL | 500 µL | 1.0mL DCM | 10 µL | 4µg/mL |
| ICAL 8 100µg/mL | 1000 µL | 1.0mL DCM | 10 µL | 4µg/mL |

*IS is added to the 1.0mL final volume

**A second ICAL 4 is prepared for the ICAL levels 1 & 2; do not add internal standard to this ICAL 4 vial.

***ICAL level 1 reports only Benzo(b)Fluoranthene

****ICAL levels 1 and 2 are the PQLs for water samples. ICAL level 3 is the PQL for soil samples.

10.7.2 8270 1,4-Dioxane Calibration Standards – Prepared as described below in DCM.



10.7.2.1 Typical 8270 1,4-Dioxane Initial Calibration Standards

| ICAL Level µg/mL | Surrogate Final Conc. µg/mL | Volume of 8270 1,4- DX 1° WS used | Final Volume (mL) | Amount of PAH IS and 1,4-DX Isotope IS to Use* | Concentration of the IS | Concentration of the 1,4-DX Isotope IS |
|---------------------|-----------------------------------|--|-------------------------|--|----------------------------|--|
| ICAL 1 0.2µg/mL | 1.0 µg/mL | 10 µL | 1.0mL DCM | 10 µL | 4µg/mL | 5µg/mL |
| ICAL 2 0.5µg/mL | 2.5 µg/mL | 25 µL | 1.0mL DCM | 10 µL | 4µg/mL | 5µg/mL |
| ICAL 3 1.0µg/mL | 5.0 µg/mL | 50 µL | 1.0mL DCM | 10 µL | 4µg/mL | 5µg/mL |
| ICAL 4 2.0µg/mL | 10 µg/mL | 100 µL | 1.0mL DCM | 10 µL | 4µg/mL | 5µg/mL |
| ICAL 5 5.0µg/mL | 25 µg/mL | 250 µL | 1.0mL DCM | 10 µL | 4µg/mL | 5µg/mL |
| ICAL 6 10µg/mL | 50 µg/mL | 500 µL | 1.0mL DCM | 10 µL | 4µg/mL | 5µg/mL |
| ICAL 7 20µg/mL | 100 µg/mL | 1000 µL | 1.0mL DCM | 10 µL | 4µg/mL | 5µg/mL |

***IS is added to the 1.0mL final volume**

- 10.8 8270C/D/E PAH Initial Calibration Verification (ICV) Standard – Prepared by diluting 200uL of the PAH Secondary Working Standard (Section 10.6.2) to a final volume of 1.0mL DCM. Add 10uL of the 400ug/mL PAH Internal Standard (Section 10.5.4) to the 1.0mL final volume. The final concentration of the target analytes is 20ug/mL.
- 10.9 8270C/D/E 1,4-DX Initial Calibration Verification (ICV) Standard – Prepared by diluting 100uL of the 1,4-DX Secondary Working Standard (Section 10.6.4) to a final volume of 1.0mL DCM. Add 10uL of the 4000ug/mL Internal Standard (Section 10.5.4) and 10uL of the 5000ug/mL 1,4-DX Isotope Internal Standard (Section 10.5.5) to the 1.0mL final volume. The final concentration of the target analytes is 2.0ug/mL. The final concentration of the surrogate is 10ug/mL.
- 10.10 8270C/D/E Continuing Calibration Verification (CCV) Standards – Prepared at the same concentration as Initial PAH Calibration Standard 7 or 8 (Table 10.7.1.1) or the same concentration as the ICV (Section 10.8).
- 10.11 8270C/D/E 1,4-DX Continuing Calibration Verification (CCV) Standards – Prepared at the same concentration as Initial 1,4-DX Calibration Standard 4 (Table 10.7.2.1).



11.0 Sample collection, preservation, shipment and storage

11.1 See AEL Admin-005 and Admin-023.

11.2 See AEL Quality Manual section 6.0 for sample acceptance policy.

11.3 See FDEP SOP FS1000 for preservation requirements, shipping conditions, and holding time requirements.

11.3.1 PAH/PRO combo samples - Samples for PAH analysis do not require; other than thermal preservation to follow FDEP SOP preservation tables. (If chlorinated samples expected, FDEP requires dechlorinating). FL-Pro extracts do require pH adjustment to <2 with either H₂SO₄ or HCL. In house studies have shown that PAH extraction recoveries are not affected by variations in pH; therefore PAH sample can be extracted under these acid conditions. This allows for the extracts, when silica gel treated, to be used for both PAH analysis and FL-Pro analysis as long as QC is extracted that meet both method's acceptance criteria and all QC passes through all the processes that samples pass through.

11.4 Aqueous samples must be extracted within 7 days of collection and analyzed within 40 days of extraction.

11.5 Soil/sediment samples must be extracted within 14 days of collection and analyzed within 40 days of extraction.

12.0 Quality Control

12.1 The GC/MS system must be tuned to meet the DFTPP criteria – see Table 2 Section 24.4.

12.2 There must be an initial calibration of the GC/MS system – see Section 13.

12.3 The GC/MS system must meet the calibration verification acceptance criteria – see Section 13.

12.4 The Relative Retention Time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

12.5 Initial Demonstration of Capability – each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff is trained or significant changes in instrumentation are made. This will consist at a minimum of a method blank and 4 replicates spiked with a standard from a different source than that used to create the analytical curve.

12.5.1 Recovery levels for IDOC analytes shall fall within the lab generated QC limits for laboratory control spikes (see Table 12 of Section 24.14 and Table 13 of Section 24.15). These limits are taken directly from the tables of limits in the



DOD Quality Systems Manual rev 5.4. These limits are based on an extensive collection of limits from DOD labs across the country and are consistent with those charted from AEL data. The analytes that are not listed in the DOD tables will use the laboratory generated control limits. If laboratory generated limits are not available, the following range is used for QC acceptance criteria: 33-132%.

12.5.2 The Demonstration of Capability is not considered complete until the analyst has documentation saying that they have read, understood, and agreed to follow the AEL-SOP for this method and the associated EPA and/or Standard Methods on which the AEL-SOP was based.

12.5.3 Initial DOC's must be successfully performed by each analyst in accordance with the Quality Manual and ADMIN-030.

12.6 Method Detection Limit (MDL) - MDLs must be established for all analytes, following the procedures outlined in ADMIN SOP-012, which conforms to EPA CFR 40 part 136.6 appendix B, updated October 2017. Below is a summary of the steps for performing MDLs. Please refer to the ADMIN SOP-012 for the full procedures.

12.6.1 New MDL Study: Most MDL determinations are for already established analyte, matrix, and instrument combinations. In those cases where a new analyte is to be introduced, an initial MDL study will have to be implemented. Select a spiking level, typically 2 to 10 times the estimated MDL. Process a minimum of seven spiked samples and seven method blank samples through all steps of the method. The samples used for the MDL must be prepared in at least three batches on three separate calendar dates and analyzed on three separate calendar dates.

12.6.2 New Instrument: To bring on a new instrument for an already established method a full set of seven low-level replicates is not needed. Only two spiked samples and two method blank samples prepared and analyzed on different calendar dates are required for the new instrument. The resulting values shall be compared against existing MDLs for validity. If both method blank results are below the existing MDL, then the existing MDL_b is validated. Combine the new spiked sample results to the existing spiked sample results and recalculate the MDLs. If the recalculated MDL_s is within 0.5 to 2.0 times the existing MDL and fewer than 3% of the MB have results above the existing MDL, the existing MDL can be left unchanged and the new instrument is validated.

12.6.3 Existing Instrument, Major Maintenance: Follow the procedures for bringing on a new instrument.

12.6.4 Ongoing Data Collection: During any quarter in which samples are being analyzed, prepare and analyze a minimum of two spiked samples on each instrument, in separate batches (separate prep batches and separate analytical batches), using the same spiking concentration used with established MDLs. If any analytes are repeatedly not detected in the quarterly spiked sample analyses, or do not meet the qualitative identification criteria of the method then this is an



indication that the spiking level is not high enough and should be adjusted upward. Note that it is not necessary to analyze additional method blanks together with the spiked samples; the method blank population should include all of the routine method blanks analyzed with each batch during the course of sample analysis.

12.6.4.1 At least once per year, re-evaluate the spiking level. If more than 5% of the spiked samples do not return positive numerical results that meet all method qualitative identification criteria, then the spiking level must be increased and the initial MDL re-determined following the procedure for establishing a new MDL.

12.6.5 Ongoing Annual Verification: At least once every thirteen months, re-calculate MDLs and MDL_b from the collected spiked samples and method blank results using the equations in the ADMIN SOP-012. These calculations shall be performed by the QA department along with updating and maintaining all chart and LIMs entry of any MDL changes.

12.7 Sample Quality Control for Preparation and Analysis

Note from QMS 5.4: The lab may use the same extract for full scan and SIM analysis if the SIM-specific Deuterated Monitoring Compounds (DMCs) are added prior to extraction.

12.7.1 The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank (MB), a matrix spike (MS), a duplicate (MSD, DUP, or LCSD), a laboratory control sample (LCS), and the addition of surrogates to each field sample and QC sample.

12.7.1.1 Each sample, MB, LCS, LCSD, MS, and MSD must be spiked with the surrogate spike.

12.7.2 Every twelve-hour analytical shift, prior to sample analysis, the laboratory must analyze an Instrument Blank (IB).

12.7.2.1 The IB contains only the surrogates and internal standards in DCM.

12.7.2.2 The IB must not contain any analyte of interest above the Method Detection Limit (MDL), or else the instrument system is contaminated.

12.7.3 Every twelve-hour analytical shift, prior to sample analysis, the laboratory must analyze a DFTPP standard (DoD criteria only). See Section 10.5.3 for the recipe for the Tune Standard. If the DFTPP fails criteria (see Section 24 Table 2) take corrective action before proceeding with calibration and/or analysis.

12.7.4 Every twelve-hour analytical shift, prior to sample analysis, the laboratory must analyze a CCV (see Section 13.3 for acceptance criteria).



12.7.5 With every batch of 20 samples (or less) the laboratory must analyze the following:

12.7.5.1MB -1 per batch of 20 or less.

12.7.5.1.1 Any analyte of interest must not be detected above the MDL.

12.7.5.2LCS – 1 per batch of 20 or less.

12.7.5.2.1 A Laboratory Control Sample (LCS) should be included with each analytical batch and once every twenty samples. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike.

12.7.5.2.2 The spike recoveries must be within the upper and lower control limits. See Table 7 of Section 24.7 and Table 8 of Section 24.10 for the control limits.

12.7.5.3MS/MSD – May be analyzed with every batch of 20 samples or less per matrix.

12.7.5.3.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate un-spiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an un-spiked field sample. If samples are not expected to contain target analytes, the laboratories should use a matrix spike and matrix spike duplicate pair. A matrix spike is required for every twenty samples and every extraction batch.

12.7.5.3.2 The spike recoveries must be within the upper and lower control limits. See Table 7 of Section 24.7 and Table 8 of Section 24.10 for the control limits.

12.7.5.3.3 The RPD for all analytes shall be within the limits specified in Table 7 of Section 24.7 and Table 8 of Section 24.10.

12.7.6 The laboratory must maintain performance records to document the quality of data that is generated.



- 12.7.7 Surrogate recoveries: The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory.
- 12.7.8 Deuterated surrogate recoveries (DoD samples only): The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. (Note: QSM 5.4 Appendix C limits do not exist for these surrogates, so in-house limits do not need to be reviewed against QSM 5.4 Appendix C table limits.)
- 12.7.9 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. If the peaks look normal, if the response obtained is comparable to the response from previous calibrations, etc., the instrument is considered still in calibration.
- 12.7.10 Assessing the Internal Standard – The peak areas or heights of the internal standards in the calibration verification standard must be within 50% to 200% (1/2 to 2x) of their respective peak areas or heights in the mid-point calibration standard.
- 12.7.10.1 If the IS response is above the QC acceptance criteria, there is an implied negative bias associated with the sample data.
- 12.7.10.2 If the IS response is below the QC acceptance criteria, there is an implied positive bias associated with the sample data.
- 12.7.10.3 See Section 20.0 for the contingencies for handling out-of-control IS responses.

13.0 Calibration and Standardization

Note: See also Section 10 for initial calibration curve standard preparation and curve concentration levels.

Note: See also AEL SOP ADMIN-038 for Calibration, Manual Integrations, and Rules for Chromatography, which outlines the procedures for choosing curve type, calculations performed, integrations allowed, and associated statistics.

13.1 Initial Calibration

13.1.1 The GC/MS operating conditions –

13.1.1.1 The GC operating conditions listed in Table 3 section 24.5 serve as guidelines only. Changes to the chromatographic conditions can be made by the analyst in order to improve the speed of analysis, lower the cost of analysis, and/or improve the separation or lower the detection



limit as long as the changes meet the initial and continuing calibration criteria and quality assurance criteria listed in this SOP.

13.1.1.2 Once these operating conditions are established they will be used to calibrate the instrument. All samples, blanks and quality assurance samples must be analyzed with the same operating conditions.

13.1.2 Mass spectrometer tuning and GC performance requirements

13.1.2.1 Before injecting calibration standards, the IB should be analyzed at the beginning of a run to confirm that the analytical system is free from contamination. The IB is used to determine the level of noise and baseline rise attributable solely to the analytical system, in the absence of any other analytes or non-analytical related contaminants. The IB is considered to be passing if the results for all target compounds are below the method detection limit (MDL).

13.1.2.2 Use of the DFTPP mass intensity criteria as tuning acceptance criteria has been dropped for sample analysis when for local and state regulatory reporting. On work for DoD clients and for any work performed under DoD QSM 5.4 Standards, tuning shall still be required.

Note: As of the 9/30/14 SOP revision, it will no longer be required to analyze a tune with DFTPP when performing SIM only analysis for sample analysis (when for local and state regulatory reporting), as we are citing the language in CLP requirements (see reference in section 23.7). Auto-tuning of the MS is still required after major instrument maintenance and before a new calibration (ICAL). A tune with DFTPP **MUST** be performed for all SIM DoD samples (this includes the ICAL associated to the samples and iDOCs).

13.1.2.3 For local and state regulatory work - Before injecting calibration standards and after the IB, the CCV or ICV should be analyzed at the beginning of a run. The CCV or ICV must pass all criteria before any standards and samples can be run. The CCV or ICV standard is the start of the 12-hour analysis window.

13.1.2.4 For DOD work - Before injecting calibration standards and after the IB, the tune should be analyzed at the beginning of a run. The tune must pass all criteria before any standards and samples can be run. The tune standard is the start of the 12-hour analysis window.

13.1.2.4.1 The GC/MS system must be hardware-tuned using a 50 ng injection of DFTPP. Analyses must not begin until the tuning criteria are met. The tuning acceptance criteria are listed in Table 2 section 24.4.

13.1.2.4.2 Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be



accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak.

- 13.1.2.4.3 Use the DFTPP mass intensity criteria in Table 2 section 24.4 as tuning acceptance criteria.

Note: All subsequent standards, samples, MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

13.1.2.4.3.1 For DoD work - A DDT degradation standard should be analyzed with DDT breakdown to be less than or equal to 20%. See Section 15.0 for the DDT breakdown calculation.

13.1.2.4.3.2 If degradation is excessive and/or poor chromatography is noted, the injection liner shall be changed. If, after changing the injection liner, poor chromatography is still noted, the injection port may require cleaning. It may also be necessary to cut off the first 6-12 in. of the capillary column

13.1.3 Initial Calibration Curve (ICAL) – The internal calibration technique is used for this method. The internal standard approach assumes that variations in instrument sensitivity, amount injected, etc. can be corrected by determining the ratio of the response of the analyte to the response of an internal standard that has been added to the extract. Nine standards are used to calibrate the instrument for the PAH analysis; a minimum of five standards must be used. Seven standards are used to calibrate the instrument for the 1,4-Dioxane analysis; a minimum of five standards must be used. The lowest concentration standard (Practical Quantitation Limit (PQL)) is at the MRL; the highest concentration is at the end of the linear range (Upper Quantitation Limit (UQL)). See Section 10 for appropriate dilutions to prepare the calibration curve.

13.1.3.1 Analyze the 8270C/D/E PAH and 1,4-Dioxane Calibration Standards following the same GC operating procedures as the client samples (recommended procedures are listed in Table 3 Section 24.5).

13.1.3.2 When using an average response factor (RF) calibration (using a calibration factor (CF) fit), for the curve evaluation, a %RSD of $\leq 15\%$ (8270C) and a %RSD of $\leq 20\%$ (8270D/E), for the CF's over the working range verifies acceptance of the calibration curve. A minimum of five calibration points are required for an average RF calibration model. AEL evaluates the calibration curve using the tightest criteria in order to analyze method 8270C samples with samples for 8270D & E.



13.1.3.3 When using a linear regression model, a minimum of 5 calibration points are required, and the coefficient of determination (r^2) must be equal to or greater than 0.990 (or the correlation coefficient (r) must be equal to or greater than 0.995).

13.1.3.4 When using a quadratic regression model, a minimum of 6 calibration points are required, and the coefficient of determination (r^2) must be equal to or greater than 0.990 (or the correlation coefficient (r) must be equal to or greater than 0.995).

13.1.3.5 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

13.1.4 System Performance Check Compounds (DoD only)

13.1.4.1 The minimum acceptable RRF for Fluoranthene-d10 and 2-methylnaphthalene-d10 criteria is 0.4. Therefore, they must meet the minimum requirement when the system is calibrated.

13.1.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

13.2 Initial Calibration Verification (ICV) – Before sample analysis can begin, the integrity of the standard used to prepare the calibration curve must be verified by the analysis of a second source (see section 10.13, 10.14 and 10.15).

13.2.1 For the ICV to be valid, the recovery for all analytes must be between 70-130%.

13.2.2 For DOD samples the ICV recovery for all analytes must be between 80-120%.

13.3 Continuing Calibration Verification (CCV) – Analyzed prior to sample analysis, after the IB, Tune (DoD criteria only), and ICAL/ICV (if a calibration was performed). A CCV must be run at the beginning of every 12-hour analysis window, after the tune standard. For DOD samples, a CCV must be run at the start of the 12-hour analysis window as well as at the end of the 12-hour analysis window and be of the same source as that which made the curve.

13.3.1 8270C/D/E CCV criteria – All target analytes must be between 80-120%.

13.3.2 8270C/D/E Ending CCV criteria - All target analytes must be between 50-150% (DoD criteria only).



- 13.3.3 CCV resolution criteria - The height of the valley between benzo(b)fluoranthene and benzo(k)fluoranthene must be less than 50% of the average of the two peak heights.

Note: DOD QSM 5.2 requires that ICV's meet 80-120% drift or % difference.

13.4 Retention Times

- 13.4.1 CCV retention times - The retention times of the CCV components are checked so that the relative retention time (RRT) of the CCV is within ± 0.06 RRT units of the RRT of the initial calibration. For example, for a 34 minute run, the methods allow the earlier eluting compounds a ± 9 second window and late eluting compounds a ± 122 second window. RRTs may be updated based on the daily CCV.

NOTE: Experience with the instruments may dictate a tighter window, especially for later eluting compounds. If a small shift is seen, adjust the retention times at this time. A small shift is one that can be seen as a result of trimming the column and should be relative in size to that trimming. Any shift that is adjusted for shall have an associated reasonable explanation for that shift. An unknown shift shall require an investigation into the cause of the shift; if warranted, a new calibration curve should be performed.

- 13.4.2 Internal retention times - The retention times of the internal standards in the CCV must be evaluated immediately after or during the data acquisition. If the retention time for any internal standard changes by more than ± 10 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, re-analysis of the QC and samples analyzed while the system was malfunctioning is required. On days when the ICAL is not performed, the initial CCV is used.

- 13.5 From the 2016 TNI Standards, the laboratory is required to use and document a measure of relative error in the calibration. By employing relative error acceptance criteria, concentrations calculated from the low end of the curve shall have the same confidence in their accuracy as those taken from any other point on the curve. Acceptance criteria must be met.

- 13.5.1 For calibrations evaluated using an average response factor, the determination of the relative standard deviation (RSD) is the measure of the relative error. If the average response factor acceptance criteria have been met, then acceptance is also met for the relative error.

- 13.5.2 For calibrations evaluated using correlation coefficient or coefficient of determination, the laboratory shall evaluate relative error by either the measurement of the Relative Error (%RE) or the measurement of the Relative Standard Error (%RSE)



Measurement of the Relative Error (%RE)

Relative error is calculated using the following equation:

$$\% \text{ Relative Error} = \frac{x'_i - x_i}{x_i} \times 100$$

x_i = True value for the calibration standard

x'_i = Measured concentration of the calibration standard

13.5.2.1 This calculation shall be performed for two (2) calibration levels: the standard at or near the mid-point of the initial calibration and the standard at the lowest level. The mid level of the calibration curve shall pass the method specified or CCV criteria. The low level of the calibration curve shall pass the method specified criteria (otherwise the default assigned limits will be +/-50% difference).

14.0 Procedure

14.1 Sample Preparation:

14.1.1 Water Extraction: see SOP SVOC-001 for waters and aqueous sample.

14.1.2 Soil Extraction: see SOP SVOC-002 for soils and sludges.

14.1.3 Waste Dilutions: see SOP SVOC-003 for wastes requiring a waste dilution.

14.2 GC/MS analysis of samples:

14.2.1 Allow the sample extract to warm to room temperature. Just prior to analysis, add 10 μL of the internal standard solution (section 10.5.4 or 10.5.5) to the 1-mL concentrated sample extract obtained from sample preparation.

14.2.2 Inject a 2 μL aliquot of the sample extract into the GC/MS system, using the same operating conditions that were used for the calibration (Section 24.5 Table 3). The injection volume must be the same volume used for the calibration standards.

14.2.3 If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and re-analyzed. Additional internal standard must be added to the diluted extract to maintain the same concentration as in the calibration standards (4 mg/mL).

14.2.4 The use of selected ion monitoring (SIM) is acceptable for applications requiring detection limits below the normal range of electron impact mass spectrometry.

14.2.5 Qualitative analysis:



See also ADMIN SOP-038: Calibration, Manual Integration, and Rules for Chromatography.

See also TECH SOP-009 Multi-peak Compound Identification for Organics.

See also TECH SOP-010 Establishing and Maintaining Retention Time Windows.

(These three SOPs are required reading for any analyst performing this method).

14.2.5.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the GC/MS operating conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met.

14.2.5.1.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

14.2.5.1.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

14.2.5.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

14.2.5.2 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. Diastereomeric pairs (e.g., Benzo(b)fluoranthene and Benzo(k)fluoranthene) that may be separable by the GC should be identified, quantified and reported as the sum of both compounds by the GC.

14.2.5.3 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima),



appropriate selection of analyte spectra and background spectra is important.

14.2.5.4 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

14.2.6 Quantitative analysis

See also ADMIN SOP-038 Calibration, Manual Integration, and Rules for Chromatography

14.2.6.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP.

14.2.6.2 Quantitation is automatically calculated with GCMS Solutions software.

14.2.6.3 The analytical method selected to process (often referred to as “crunch”) the raw data will be the one with the most recent calibration for the analytes to be quantitated. The normal PAH SIMs list will be processed with the PAH curve and will generate a single quant report. If the Appendix 9 (namely 1,4-Dioxane) analytes are to also be processed, a separate analytical method for Appendix 9 (1,4-Dioxane) compounds will be loaded and the raw data run will be quantitated under that processing method as well. This will generate a second quant report. The curve and QC must also be evaluated as passing for those analytes under that extra processing step as well in order to report. The original PAH report and the Appendix 9 report will both then constitute the full processed report.

15.0 Calculations

15.1 Multiply all concentrations by any external dilutions as well as any dilutions incurred during extraction to obtain the final result.

15.2 Relative Retention Time (expressed as a unitless quantity):

$$RRT = \frac{\text{Retention Time of the analyte}}{\text{Retention Time of the IS}}$$

15.3 DDT Breakdown

$$\% \text{ Breakdown } 4,4\text{-DDT} = \frac{\text{area } 4,4'\text{-DDE} + \text{area } 4,4'\text{-DDD}}{\text{area } 4,4'\text{-DDE} + \text{area } 4,4'\text{-DDD} + \text{area } 4,4'\text{-DDT}} * 100$$



15.4 Average (% RSD) Calibration Calculations:

15.4.1 Calculate the Response factors (RF) or Relative response factor (RRF); for each compound:

$$RF = \frac{A \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s = Response for the analyte to be measured

A_{is} = Response for the internal standard

C_{is} = Concentration of internal standard

C_s = Concentration of the analyte to be determined in the standard

15.4.2 Calculate the mean response factor (RF) for each compound:

$$\overline{RF} = \frac{\sum_{i=1}^n RF_i}{n}$$

Where: n = The number of standards analyzed.

15.4.3 The processing software will calculate the standard deviation (SD) and the RSD of the calibration factors for each analyte as:

$$SD = \sqrt{\frac{\sum_{i=1}^n [RF_i - \overline{RF}]^2}{(n - 1)}} \quad RSD = \frac{SD}{\overline{RF}} * 100$$

Where: n = The number of standards analyzed.

15.4.4 Calculation of the percent difference (%D) for all analytes in the ICV and/or CCV:

$$\%D = \frac{C_{expected} - C_{found}}{C_{expected}} * 100 = \frac{RF_{ccv} - \overline{RF}}{\overline{RF}} * 100$$

Where:

$C_{expected}$ = the true value of the analyte or surrogate.

C_{found} = the on-column analyte or surrogate result

15.4.5 Determine the concentration of individual compounds in the sample using the following equation:



$$\text{Concentration (ug/L)} = \frac{(A_s) (I_s)}{(A_{is}) (\overline{RF})}$$

Where:

A_s = response of the analyte in the sample

I_s = concentration of internal standard present (in ug/L).

A_{is} = response of the internal standard

\overline{RF} = Average Response Factor (unitless)

15.5 Linear Calibration Calculations (using a least squares regression (first-order) calibration fit):

15.5.1 Option 1: X_s is the concentration of the analyte in the calibration standard aliquot introduced into the instrument and Y_s is the ratio of response of the analyte to the response of internal standard times the mass of the internal standard in the calibration standard aliquot introduced into the instrument.

$$X_s = C_s \quad \text{and} \quad Y_s = A_s \times \frac{C_{is}}{A_{is}}$$

Where:

C_s = concentration of analyte in the volume of calibration standard introduced into the instrument.

C_{is} = concentration of internal standard in the volume of calibration standard injected into the instrument.

A_s = Peak response of analyte.

A_{is} = Peak response of internal standard.

15.5.2 Option 2: x is the ratio of the analyte concentration in the calibration standard aliquot introduced into the instrument to the internal standard concentration in the calibration standard aliquot introduced into the instrument and y is the ratio of response of the analyte to the response of internal standard.

$$x = \frac{C_s}{C_{is}} \quad \text{and} \quad y = \frac{A_s}{A_{is}}$$

Where:

C_s = concentration of analyte in the volume of calibration standard introduced into the instrument.

C_{is} = concentration of internal standard in the volume of calibration standard injected into the instrument.

A_s = Peak response of analyte.

A_{is} = Peak response of internal standard.



15.5.3 The linear least squares regression equation is:

$$y = ax + b$$

Where:

a = The slope of the linear regression.

b = The intercept of the linear regression.

15.5.4 The processing software will calculate the coefficient of determination (r^2) for each analyte by squaring the correlation coefficient as:

$$r^2 = \left[r = \frac{n \sum_{i=1}^n x_i y_i - \sum_{i=1}^n x_i \sum_{i=1}^n y_i}{\sqrt{(n \sum_{i=1}^n x_i^2 - (\sum_{i=1}^n x_i)^2)(n \sum_{i=1}^n y_i^2 - (\sum_{i=1}^n y_i)^2)}} \right]^2$$

15.5.5 Calculation of the Percent Drift for all analytes in the ICV and/or CCV:

$$\% \text{ Drift} = (\text{Analyte Result} / \text{True Value}) * 100$$

15.5.6 Calculations of sample amounts when using a linear regression calibration fit:

$$\text{Option 1} \quad X_s = \frac{\left(\frac{A_s \times C_{is}}{A_{is}} \right) - b}{a}$$

$$\text{Option 2} \quad X_s = \frac{\left(\frac{A_s}{A_{is}} \right) - b}{a} \times C_{is}$$

Where:

X_s = calculated concentration of the analyte or surrogate in the sample aliquot introduced into the instrument

A_s = peak response of the analyte or surrogate in the sample

A_{is} = peak response of the internal standard in the sample

C_{is} = concentration of the internal standard in the sample aliquot introduced into the instrument.

a = The slope of the linear regression.

b = The intercept of the linear regression.

15.6 Percent Recovery for standards and LCS/LCSD

$$\% \text{ Recovery} = (\text{LCS Result} / \text{True Value}) * 100$$

15.7 Percent Recovery for MS and MSD samples



$$\% \text{ Recovery} = [(\text{MS/D Result} - \text{Parent Sample Result}) / \text{Spike True Value}] * 100$$

15.8 Dry weight determination:

$$\text{mg/dry kg PH} = \frac{C_s}{1 - (\% \text{moisture}/100)}$$

Where:

C_s = Concentration of Pesticides (in mg/L or mg/kg)

15.9 Relative Percent Difference (RPD)

$$\% \text{ RPD} = \frac{|\text{Difference b/w Dups}|}{\text{Average of Dups}} * 100$$

15.10 Method Detection Limit: The MDL is typically calculated as 3.143 times the standard deviation (SD).

$$\bar{X} = \frac{\sum_{i=1}^n X_i}{n} \quad SD = \sqrt{\frac{\sum_{i=1}^n [X_i - \bar{X}]^2}{(n - 1)}} \quad MDL = SD \times 3.143$$

Where: n = The number of standards analyzed.

X = One of the seven replicate MDL values.

16.0 Method Performance (See Sections 12.0 Quality Control and 18.0 Data Assessment and acceptance criteria for quality control measures).

17.0 Pollution Prevention

17.1 See Standard Methods section 1100, 22nd Edition / On-Line edition – Waste Minimization and Disposal.

17.2 See SOP Admin-018 and the AEL Safety Manual.

18.0 Data Assessment and acceptance criteria for quality control measures

18.1 It is the responsibility of the analyst to assess all data for quality control acceptance criteria.

18.2 It is the responsibility of the analyst to review all data/data entry for adherence to quality control criteria and any transcription or typographical errors prior to peer or supervisor review.



- 18.3 It is the responsibility of the analyst and/or supervisor to initiate and/or recommend correction action for out of control data.
- 18.4 All out of control data will be qualified according to SOP Admin-008.
- 18.5 See AEL QM Section 10.0.
- 18.6 All calibration will have a minimum of a five-point curve (Note: a minimum six-point curve is required for a quadratic calibration fit). Points at the ends of the curve may be dropped if they are non-linear. If a data point is not usable or unacceptable within the curve, it will be re-analyzed. A data point for the ICAL can be dropped only if it can be documented as a non-repeatable error.
- 18.7 Each analytical batch at a minimum will follow the “12 hour rule,” where, at the beginning of each 12-hour period, an IB, TUNE (DoD requirement only), and CCV must be analyzed.
- 18.7.1 The instrument must be free of analytes as exhibited by an instrument blank at the beginning of each analysis window.
- 18.7.2 The Tune (DoD requirement), where applicable must pass the method criteria for any following data to be reported.
- 18.7.3 The CCV must meet method acceptance criteria to report unqualified data.
- 18.7.3.1 If any analyte fails with a low recovery, no data results can be reported for that analyte.
- 18.7.3.2 If any analyte fails with a high recovery, non-detects can be reported if qualified or properly noted in the case narrative. Corrective action (ex: remixing standard, cleaning injection liner, etc.) will be taken before the next analysis window (or next analytical sequence if the instrument is running multiple analysis windows in one night).
- 18.8 Method Blank (MB) – The MB must pass the surrogate recovery acceptance limits and be clean of analytes of interest. If there are hits in the method blank, these hits must be below the method detection limit.
- 18.8.1 If the hits are above the method reporting level, and the samples themselves are clean of those hits (below the MDL), the sample results are fine to report.
- 18.8.2 If the MB results are above the MDL, and samples have the same hits, then sample results will not be accurate and the MB and samples must be re-analyzed and/or re-extracted to prove that the hits were not contamination.
- 18.9 Laboratory Control Spikes (LCS) – The LCS must fall within control chart limits for percent recoveries of both analytes for valid data reporting. If the LCS fails, first check the instrument for possible problems. Document any issues, and then re-analyze the



LCS. If the LCS still fails, then all samples in the extraction batch must be re-extracted or re-analyzed.

- 18.10 Matrix Spike/Matrix Spike Duplicate (MS/MSD) – Failure to meet control limits for analyte and surrogate recoveries does not in itself require data to be rejected. Data can be flagged (J(4)) for matrix interference.
 - 18.11 At any point in the analytical batch, an analyst may use his/her discretion to fail or reject data he/she feels to be suspicious or in error. At this point, the analyst should seek the help of a supervisor or the QC officer.
 - 18.12 Data should be rejected for individual sample runs if the chromatogram looks odd, retention times shift outside retention time windows, or surrogates fail due to reasons other than matrix interferences.
 - 18.13 Any and all QC failures must be reported to a supervisor and the QC officer.
 - 18.14 Extraction personnel should be informed of any failures immediately. They should also be informed of any trends that develop in sample recoveries.
- 19.0 Corrective action for out of control data
- 19.1 See Section 20.0 for out of control or unacceptable data.
 - 19.2 See SOP ADMIN-016 and ADMIN-028.
- 20.0 Contingencies for handling out of control or unacceptable data
- 20.1 If a blank failure occurs, any sample containing that analyte will get a ‘V’ qualifier and an NCF is required.
 - 20.1.1 If the MB exhibits contamination, but the samples are BDL for the analytes in question, there is no need for any qualifier nor does an NCF need to be written.
 - 20.2 If the surrogate(s) happen to fall outside of the limits mentioned in Table 5 and Table 6 of section 24, they will be qualified with a “J(1)” or “J(4)” qualifier, depending on whether or not there is matrix interference involved.
 - 20.3 LCS failure
 - 20.3.1 If the LCS fails the lower criteria and there is insufficient sample to re-extract, the sample will be reported as is; however, every analyte that failed will get a “J(3)” qualifier and an NCF is required.
 - 20.3.2 If the LCS fails the upper criteria and the sample is BDL for the analyte in question, the failure will be case narrated. The sample will not be qualified nor does an NCF need to be written.



- 20.3.3 If the LCS fails the upper criteria and the sample contains a hit for the analyte in question and there is insufficient sample to re-extract, the sample will be reported as is; however, the analyte in question will get a “J(3)” qualifier and an NCF is required.
- 20.4 If the MS/MSD requires a dilution greater than 1:5, the recovery is suspect and will not be calculated. The MS/MSD failure will be case narrated and the sample will not be qualified.
- 20.5 If the native sample that is used for the MS/MSD has a target analyte detected at a concentration greater than 4 times the spike value, the spike recovery in the MS and MSD will not be calculated. The MS/MSD failure will be case narrated and the sample will not be qualified.
- 20.6 See ADMIN-016 for the NCF writing process.
- 20.7 See ADMIN-028 for the Case Narrative writing process.
- 20.8 Internal Standard (IS) – The peak areas or heights of the internal standards in the calibration verification standard must be within 50% to 200% (1/2 to 2x) of their respective peak areas or heights in the mid-point calibration standard.
- 20.8.1 If the IS response is above the QC acceptance criteria, then there is an implied negative bias associated with the sample data:
- 20.8.1.1 If a deviation of greater than 200% occurs with an individual extract, but the surrogate yield is within the acceptance criteria, then the data can be reported. The acceptable surrogate yield proves that the elevated IS response did not affect the quantitation of extraction efficiency. The elevated IS should be noted on the coversheet for the data pack and in the case narration for the batch.
- 20.8.1.2 If a deviation of greater than 200% occurs with an individual extract, and the surrogate yield is NOT within the acceptance criteria, optimize instrument performance and inject a second aliquot. The elevated IS response may be impacting the data quantitation.
- 20.8.1.2.1 If the re-injected aliquot produces an acceptable internal standard response, report the results for the re-analysis.
- 20.8.1.2.2 If a deviation of greater than 200% is obtained for the re-injected extract, then sample matrix interference may be causing issues with the Internal Standard recoveries. Re-analysis of the sample should be repeated at a dilution beginning with Section 14.0, provided the sample is still available. Otherwise, report the results obtained from the re-injected extract, but annotate as suspect.



20.8.1.2.2.1 If the dilution re-analysis produces an acceptable internal standard response, report results and note in the case narration that a dilution was required due to sample matrix interferences.

20.8.1.2.2.2 If the dilution re-analysis does not produce an acceptable internal standard response, report results and note in the case narration that the internal standard recoveries were not within QC criteria due to sample matrix interferences.

20.8.2 If the IS response is below the QC acceptance criteria, then there is an implied positive bias associated with the sample data:

20.8.2.1 If a deviation of less than 50% occurs with an individual extract, and the sample does NOT contain target analytes above the Method Detection Limit (MDL), then the data can be reported.

20.8.2.1.1 The positive bias implied by the elevated IS recovery has no effect on a result below the detection limit. The data will be "U" qualified automatically in the report as non-detected. The low IS should be noted on the coversheet for the data pack and in the case narration for the batch.

20.8.2.2 If a deviation of less than 50% occurs with an individual extract, and the sample does contain target analytes above the Method Detection Limit (MDL), but the surrogate yield is within the acceptance criteria, then the data can be reported.

20.8.2.2.1 The acceptable surrogate yield proves that the elevated IS response did not affect the quantitation of extraction efficiency. The low IS should be noted on the coversheet for the data pack and in the case narration for the batch.

20.8.2.3 If a deviation of less than 50% occurs with an individual extract, and the sample does contain target analytes above the Method Detection Limit (MDL), and the surrogate yield is NOT within the acceptance criteria, optimize instrument performance and inject a second aliquot. The elevated IS response may be impacting the data quantitation.

20.8.2.3.1 If the re-injected aliquot produces an acceptable internal standard response, report results for the re-analysis.

20.8.2.3.2 If a deviation of less than 50% is obtained for the re-injected extract, then sample matrix interference may be causing issues with the Internal Standard recoveries. Re-analysis of the sample should be repeated at a dilution beginning with



Section 14.0, provided the sample is still available.
Otherwise, report the results obtained from the re-injected
extract, but annotate as suspect.

20.8.2.3.2.1 If the dilution re-analysis produces an acceptable
internal standard response, report results and note in
the case narration that a dilution was required due to
sample matrix interferences.

20.8.2.3.2.2 If the dilution re-analysis does not produce an
acceptable internal standard response, report results
and note in the case narration that the internal
standard recoveries were not within QC criteria due
to sample matrix interferences.

21.0 Waste Management

21.1 Refer to SOP for Waste Management (ADMIN-018) for any other questions.

21.2 See Standards Method section 1090, 23rd Edition / On-Line edition– Waste Minimization
and Disposal.

22.0 Cautions/Preventative Maintenance

22.1 Routine, preventative instrument maintenance must be performed and documented in a
maintenance logbook to assure optimum instrument performance. All maintenance is
documented in the maintenance log in accordance with Quality Manual 8.0.

22.2 System Carryover – Highly concentrated calibration standards and client samples
containing high concentrations of target analytes can be retained in the GC systems and
bleed out (carryover) into subsequent QC and client samples. Blanks must be analyzed
after the initial calibration and “hot” client samples to prove the system is free of such
contamination before more batches QC (MB, etc.) are analyzed. It is not acceptable to
delete carryover from batch QC or client samples. Client samples must be rerun to
confirm suspected hits from carryover.

22.3 Gas Chromatograph

22.3.1 A regular schedule of maintenance is dictated more by the instrument checks and
a visual check of the chromatography more than by any set schedule. Most
maintenance is done in response to a failure of one of the QC checks done during
the course of normal operation or poor chromatographic performance. These
checks ensure that the instrument is working at top performance and is proof that
the instrument is in good working order.

22.3.2 Injector liners, inlet seals, and other injection port consumables must be cleaned
or changed if any related problems occur (i.e. increase baseline, noise,
integration, or signal deviations).



- 22.3.3 Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and “clean” (uniform, without fragmentation) by using the proper column-cutting tool.
- 22.3.4 Over time, the column will exhibit poorer overall performance. This is due to repeated temperature cycling and the number of heavily contaminated samples being analyzed. The length of time for this to occur varies. The most obvious evidence is calibration difficulties (e.g., more compounds requiring linear or non-linear calibration models) poor resolution, excessive column bleed, peak tailing and/or the peaks are seen to broaden and flatten. When a noticeable decrease in column performance is evident, and other maintenance options do not result in improvement, the analytical column should be replaced. On average, a column will require replacement once every 6 months to 12 months, but that time can be much sooner dependent on sample matrices and sample load.
- 22.3.5 “Hot” or “dirty” samples or the cumulative effect of many samples can cause the chromatography to degrade as well. Performing routine maintenance can bring performance back to normal operation. However, in some cases when the chromatography is not improved with this maintenance, the column will require replacement.
- 22.3.6 A new analytical column must be conditioned according to manufacturer specifications. This typically involves a slow temperature ramp over a period of hours with moderate carrier gas flow through the column. Heating the column without carrier gas flow will irreparably damage the column. The columns should not be heated past the manufacturer's maximum temperature recommendation.
- 22.3.7 A dirty detector can result with use over time. If the baseline is seen as becoming erratic or the signal response is seen to degrade, this may indicate that the detector needs cleaning. Cleaning the source will most times restore full signal response.
- 22.4 When any maintenance is performed, the system should be carefully inspected for leaks prior to beginning analysis. Any parts of the instrument that have been recently taken apart and re-assembled are the most likely places for a leak to develop. It is also important to periodically check for leaks at the detectors where the columns are inserted.
- 22.5 Typical Instrument Preventative Maintenance Schedule – GC/MS
- 22.5.1 Daily:
- 22.5.1.1 Keep wash/rinse vials filled with appropriate solvent.
- 22.5.1.2 Check CCV results to see if a liner or septa change is needed. If the CCV indicates instrument issues, perform inlet maintenance.



22.5.1.3 Check the Helium tank pressure, replace tank as needed.

22.5.2 Monthly:

22.5.2.1 Clean auto-samplers and check that needles are clean and in working condition (clean or replace the syringes if needed).

22.5.2.2 Clean any accumulated dust and dirt off of the instrumentation.

22.5.2.3 Check the fore line pump oil level. Add pump fluid as needed until the oil level in the window is near, but not above, the upper fill line.

22.5.3 Semi-Annual:

22.5.3.1 Run a new Calibration Curve.

22.5.3.2 Clean MS (includes filament replacement and cleaning of ion source, pre-rods, and lenses) when needed.

22.5.4 Yearly:

22.5.4.1 Replace oil in rotary pump.

22.5.4.2 Replace column (or earlier if needed).

23.0 References

- 23.1 *Semi-Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Capillary Column Technique*, Method 8270C, Revision 3, December 1996 in Test Method for Evaluating Solid Waste, Physical/Chemical Methods, U.S. EPA, SW-846.
- 23.2 *Semi-Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Capillary Column Technique*, Method 8270D, Revision 5, July 2014 in Test Method for Evaluating Solid Waste, Physical/Chemical Methods, U.S. EPA, SW-846.
- 23.3 *Semi-Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Capillary Column Technique*, Method 8270E, Revision 6, June 2018 in Test Method for Evaluating Solid Waste, Physical/Chemical Methods, U.S. EPA, SW-846.
- 23.4 *Determinative Chromatographic Separations*, Method 8000C, Revision 3 in Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, U.S. EPA, SW-846.
- 23.5 *Determinative Chromatographic Separations*, Method 8000D, Revision 4 May-2018 in Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, U.S. EPA, SW-846.
- 23.6 Standard Methods 23rd Edition.



- 23.7 EPA CFR 40 Part 136.6, Appendix B.
 - 23.8 TNI Standards 2016
 - 23.9 ISO 17025: 2005 & 2017 Standards
 - 23.10 DOD Quality Systems Manual most recent version
 - 23.11 AEL Health and Safety Manual.
 - 23.12 AEL Policy and Procedures Manual.
 - 23.13 AEL Quality Manual, latest revision.
 - 23.14 AEL Admin SOPS.
- 24.0 Tables, Diagrams, flowcharts, and validation data
- 24.1 Validation Data: See the employee files (stored electronically on the network or hardcopy in the laboratory's QA office) for the individuals for an acceptable initial demonstration of capability, which serves as validation data for this method in AEL.
- (see following pages for tables)



24.2 Table 1 – Characteristic Ions of Target Analytes, Retention Time, Internal Standard Group, Sim Window

Characteristic Ions of Target Analytes PAH

| Compound | Retention Time (min) | Primary Ion | Secondary Ion | Tertiary Ion | Quaternary Ion | Internal Standard Group | Sim Window |
|-------------------------|----------------------|-------------|---------------|--------------|----------------|-------------------------|------------|
| Nitrobenzene-d5 | 2.543 | 82 | 54 | 128 | | 1 | 1 |
| Naphthalene-d8 | 3.666 | 136 | 54 | 108 | | 1 | 1 |
| Naphthalene | 3.71 | 128 | 129 | 127 | | 1 | 1 |
| 2-Methylnaphthalene-d10 | 5.243 | 152 | 150 | 122 | | 1 | 2 |
| 2-Methylnaphthalene | 5.335 | 142 | 141 | 115 | | 1 | 2 |
| 1-Methylnaphthalene | 5.56 | 142 | 141 | 115 | | 1 | 2 |
| 2-Fluorobiphenyl | 6.208 | 172 | 170 | 85 | | 2 | 2 |
| Acenaphthylene | 7.274 | 152 | 151 | 153 | | 2 | 3 |
| Acenaphthene-d10 | 7.572 | 164 | 162 | 80 | | 2 | 3 |
| Acenaphthene | 7.635 | 154 | 152 | 153 | | 2 | 3 |
| Dibenzofuran | 7.987 | 168 | 139 | 84 | | 2 | 3 |
| Fluorene | 8.639 | 166 | 165 | 167 | | 2 | 4 |
| Phenanthrene-d10 | 10.303 | 188 | 80 | 187 | 184 | 3 | 5 |
| Phenanthrene | 10.341 | 178 | 179 | 176 | | 3 | 5 |
| Anthracene | 10.429 | 178 | 179 | 176 | | 3 | 5 |
| Carbazole | 10.74 | 167 | 166 | 139 | | 3 | 5 |
| Fluoranthene-d10 | 12.292 | 212 | 106 | 104 | | 3 | 6 |
| Fluoranthene | 12.328 | 202 | 101 | 203 | | 3 | 6 |
| Pyrene | 12.687 | 202 | 203 | 200 | | 4 | 6 |
| p-Terphenyl-d14 | 13.016 | 244 | 245 | 122 | | 4 | 6 |
| Benzo[a]anthracene | 14.642 | 228 | 229 | 226 | | 4 | 7 |
| Chrysene-d12 | 14.657 | 240 | 241 | 120 | | 4 | 7 |
| Chrysene | 14.698 | 228 | 229 | 226 | | 4 | 7 |
| Benzo[b]fluoranthene | 16.364 | 252 | 253 | 125 | | 5 | 8 |
| Benzo[k]fluoranthene | 16.407 | 252 | 253 | 125 | | 5 | 8 |
| Benzo[a]pyrene | 16.874 | 252 | 253 | 125 | | 5 | 8 |
| Perylene-d12 | 16.972 | 264 | 132 | 130 | 260 | 5 | 8 |
| Indeno(1,2,3-cd)pyrene | 19.059 | 276 | 138 | 277 | | 5 | 9 |
| Dibenzo(a,h)anthracene | 19.134 | 278 | 139 | 279 | | 5 | 9 |
| Benzo(g,h,i)perylene | 19.672 | 276 | 138 | 277 | | 5 | 9 |

Characteristic Ions of Target Analytes 1-4 Dioxane Curve

| Compound | Retention Time (min) | Primary Ion | Secondary Ion | Internal Standard Group | Sim Window |
|------------------------|----------------------|-------------|---------------|-------------------------|------------|
| 1,4-Dioxane-d8 | 2.12 | 98 | 64 | 1 | 1 |
| 1,4-Dioxane | 2.16 | 88 | 58 | 1 | 1 |
| 1,4-Dichlorobenzene-d4 | 5.33 | 150 | 115 | 2 | 2 |
| Nitrobenzene-d5 | 5.89 | 82 | 54 | 2 | 2 |



24.3 Table 2 – DFTPP Criteria (DoD only)

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

| Mass | Ion Abundance Criteria |
|------|------------------------------------|
| 51 | 30-60% of mass 198 |
| 68 | < 2% of mass 69 |
| 70 | < 2% of mass 69 |
| 127 | 40-60% of mass 198 |
| 197 | < 1% of mass 198 |
| 198 | Base peak, 100% relative abundance |
| 199 | 5-9% of mass 198 |
| 275 | 10-30% of mass 198 |
| 365 | > 1% of mass 198 |
| 441 | Present but less than mass 443 |
| 442 | > 40% of mass 198 |
| 443 | 17-23% of mass 442 |

24.4 Table 3 - Recommended 8270 and DFTPP GC/MS Operating Conditions

Typical 8270 PAH GC Operating Conditions

| System | Condition |
|---------------------|---|
| Injection Volume | 2uL |
| Column Oven Temp | 120°C |
| Injection Temp | 240°C |
| Injection Mode | Split |
| Flow Control Mode | Pressure |
| Pressure | 97.3 kPa |
| Total Flow | 19.0mL/min |
| Column Flow | 1.19mL/min |
| Linear Velocity | 40.9cm/sec |
| Purge Flow | 0.0mL/min |
| Split Ratio | 15.0 |
| Temperature Program | 120°C hold 4min 15mL/min to 270°C hold 0min 12mL/min to 300°C hold 7min |
| GC Run program time | 23.50min |



Typical 8270 PAH MS Operating Conditions

| System | Condition |
|-------------------|-----------|
| Ion Source Temp | 260°C |
| Interface Temp | 300°C |
| Solvent Cut Time* | 1.8min |
| Event time | 0.2 sec |

*The solvent cut time is a recommended start time. It can be adjusted after the column has been clipped and/or a new column has been installed.

Typical 8270 1,4-Dioxane GC Operating Conditions

| System | Condition |
|---------------------|--|
| Injection Volume | 2uL |
| Column Oven Temp | 40°C |
| Injection Temp | 240°C |
| Injection Mode | Split |
| Flow Control Mode | Pressure |
| Pressure | 112.0 kPa |
| Total Flow | 64.6 mL/min |
| Column Flow | 2.0 mL/min |
| Linear Velocity | 51.0 cm/sec |
| Purge Flow | 0.0mL/min |
| Split Ratio | 31.3 |
| Temperature Program | 40°C hold 2.0min 25mL/min to 250°C hold 0min 40mL/min to 300°C hold 0min |
| GC Run program time | 11.65min |

Typical 8270 1,4-Dioxane MS Operating Conditions

| System | Condition |
|-------------------|-----------|
| Ion Source Temp | 260°C |
| Interface Temp | 300°C |
| Solvent Cut Time* | 1.5min |
| Event time | 0.3 sec |

Typical DFTPP GC Operating Conditions

| System | Condition |
|-------------------|------------|
| Injection Volume | 2uL |
| Column Oven Temp | 125°C |
| Injection Temp | 240°C |
| Injection Mode | Split |
| Flow Control Mode | Pressure |
| Pressure | 123.1kPa |
| Total Flow | 11.9mL/min |
| Column Flow | 1.49mL/min |
| Linear Velocity | 45.9cm/sec |
| Purge Flow | 0mL/min |



| | |
|---------------------|--|
| Split Ratio | 7.0 |
| Temperature Program | 125°C hold 2min 25mL/min to 300°C hold 4min |
| GC Run program time | 14.0 min |

Typical DFTPP MS Operating Conditions

| System | Condition |
|-------------------|-----------------|
| Ion Source Temp | 260°C |
| Interface Temp | 300°C |
| Solvent Cut Time* | 2.9min |
| Mass Range | 40-450m/z (amu) |
| Scan Time | 0.22scan/sec |
| Scan speed | 2000 |

24.5 Table 4 - Water Surrogate Acceptable Recoveries (from Laboratory Control Charts)

Surrogate Acceptance Range for Waters for Method SIM 8270

| Surrogate | Acceptable % Recovery |
|-------------------------------------|-----------------------|
| Nitrobenzene d ₅ | 55-111 |
| 2-Methylnaphthalene-d ₁₀ | 50-150 |
| 2-Fluorobiphenyl | 53-106 |
| Fluoranthene-d ₁₀ | 50-150 |
| p-Terphenyl-d ₁₄ | 58-132 |

24.6 Table 5 - Soil Surrogate Acceptable Recoveries (from Laboratory Control Charts)

Surrogate Acceptance Range for Soils for Method SIM 8270

| Surrogate | Acceptable % Recovery |
|-------------------------------------|-----------------------|
| Nitrobenzene d ₅ | 37-122 |
| 2-Methylnaphthalene-d ₁₀ | 50-150 |
| 2-Fluorobiphenyl | 46-115 |
| Fluoranthene-d ₁₀ | 50-150 |
| p-Terphenyl-d ₁₄ | 54-127 |



24.7 Table 6- Typical PAH Analytical Sequence

| Calibration Curve Analytical Sequence | Daily Analytical Sequence |
|--|--|
| DCM Rinse | DCM Rinse |
| IB | IB |
| Tune (DOD only) | Tune (DOD only) |
| ICAL (1 through 9) | CCV |
| ICV | QC and client samples (12-hour window = 24 injections after tune or CCV) |
| QC and client samples (12-hour window = 24 injections after tune or CCV) | |

**DOD samples require a closing CCV with the criteria of 50-150%. The opening CCV of the second window can be used as the closing CCV for the first analysis window.

24.8 Table 7 –Typical 1,4-Dioxane Analytical Sequence

| Calibration Curve Analytical Sequence | Daily Analytical Sequence |
|--|--|
| DCM Rinse | DCM Rinse |
| IB | IB |
| Tune (DOD only) | Tune (DOD only) |
| ICAL (1 through 7) | CCV |
| ICV | QC and client samples (12-hour window = 30 injections after tune or CCV) |
| QC and client samples (12-hour window = 30 injections after tune or CCV) | |

**DOD samples require a closing CCV with the criteria of 50-150%. The opening CCV of the second window can be used as the closing CCV for the first analysis window.



24.9 Table 8 – Method Required Quality Control for 8270C/D/E

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action |
|--|---|--|---|
| Initial Blank (IB) | At the beginning of each analytical sequence | No analytes detected above the MDL | Correct problem then re-analyze |
| Continuing Calibration Blank (CCB) | Analyzed after “hot” client samples | No analytes detected above the MDL | Correct problem then re-analyze |
| DFTPP Tune (DOD only) | Analyzed after the IB prior to any QC and project samples and every 12 hours | DFTPP criteria – Table 2 DDT breakdown <20% | Correct problem and re-analyze all QC and project |
| Resolution Check | Performed benzo(b) and benzo(k)fluoranthene on the mid-point calibration standard and on the CCV of every analytical run. | Resolution Criteria - The height of the valley between the 2 isomers must be less 50% of the average height of the 2 peak heights. Correct problem and re-analyze all QC and project | |
| Minimum five-point initial calibration for %RSD and linear regression (minimum six-point if quadratic regression used) | Prior to sample analysis | 8270C: %RSD ≤ 15%; linear regression or quadratic regression coefficient of determination $r^2 \geq 0.995$ or correlation coefficient $(r) \geq 0.990$. 8270D/E: %RSD ≤ 20%; linear regression or quadratic regression coefficient of determination $r^2 \geq 0.995$ or correlation coefficient $(r) \geq 0.990$. | Correct problem then repeat initial calibration |
| Initial Calibration Verification (ICV) | Analyzed after the ICAL; prior to sample analysis | 70% - 130% DOD samples 80% - 120% | Correct problem then repeat initial calibration |
| Continuing Calibration Verification (CCV) | At the beginning of each analytical sequence and every 12-hours. Run after the DFTPP tune standard. | 8270C/D/E: CCC: 80-120% & DMC surrogates RF > 0.4 (DOD only) Closing bracketing CCV required for DOD samples 50-150% | Correct the problem. If the CCV fails high re-analyze all samples that contained hits above the MDL. If the CCV fails low re-analyze all samples bracketed by the CCV failure |
| Initial Demonstration of Capability (IDOC) and Continuing Demonstration of Capability (CDOC) | MB, and 4 replicate second source analyses, prior to analysis of samples; once per year to maintain CDOC's | See Table 13 & Table 14 in Section 24 for control limits, RPD less than 30% | Correct problem and re-analyze or re-extract for analytes that did not meet criteria |



| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action |
|--|--|---|---|
| Initial Method Detection Limit (MDL) Study | 7 replicate LCS (MDL _s) at a concentration at the RL and 7 Method Blanks (MDL _b) | Calculate the MDL _s and the MDL _b . Choose the greater of the two as the initial MDL. | None |
| Ongoing MDL Data Collection | Every quarter, prepare and analyze 2 spiked samples at the RL on each instrument, in separate batches | Evaluate the spiked samples against the control limits in Table 9 & Table 10 in Section 24. | None |
| Method Blank (MB) | One per analytical batch of 20 samples or less | No analytes detected above the MDL | Correct problem and re-analyze all samples that contain target analytes above the MDL |
| LCS and LCSD | One LCS/LCSD per analytical batch of 20 samples or less | See Table 9 and Table 10 in section 24 for control limits, RPD less than 30% | Correct problem and re-analyze all samples affected by failure |
| MS and MSD | One MS/MSD per analytical batch of 20 samples or less | See Table 9 and Table 10 in section 24 for control limits, RPD less than 30% | Flag the data if matrix interference is evident |
| Surrogate Recoveries | Spiked into every sample including all QC samples | See Table 5 and Table 6 in section 24 for control limits. | Reanalyze, or re-extract and re-analyze, or flag the data |



24.10 Table 9 - DOD QSM 5.4 QC Requirements Table B-22

| Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode | | | | | |
|---|--|--|--|---|--|
| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
| Tune Check | Prior to ICAL and prior to each 12-hour period of sample analysis. | Specific ion abundance criteria of DFTPP from method 8270. Tune check can be acquired as a full scan. | Retune instrument and verify. | Flagging is not appropriate. | No samples shall be analyzed without a valid tune. In addition to the full scan tune check, optimization for the analytes of interest is recommended. |
| Deuterated Monitoring Compounds (DMCs) (surrogates) | All field and QC samples. | <u>PAH analysis:</u> DMCs required for polyaromatic hydrocarbon (PAH) target analytes: fluoranthene-d ₁₀ and 2-methylnaphthalene-d ₁₀ . Minimum RRF for PAH DMCs: 0.40. <u>All DMCs:</u> Requires 50-150% recovery until in-house limits can be established. | Correct problem, and then reprep and reanalyze all samples with failing DMCs if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the Case Narrative. | Apply Q-flag to all associated samples and analytes if acceptance criteria are not met and explain in the Case Narrative. | For non-PAH target analytes, other DMCs with similar chemistry must be assigned. Laboratories may use the same extract for full scan and SIM analysis if the SIM-specific DMCs are added prior to extraction. |

| Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode | | | | | |
|---|--|----------------------------|--|------------------------------|--|
| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
| Performance Checks | At the beginning of each 12-hour period, prior to analysis of samples. | Degradation ≤ 20% for DDT. | Correct problem, then repeat performance checks. | Flagging is not appropriate. | No samples shall be analyzed until the performance checks are within criteria. DDT breakdown and tailing factors are considered overall measures of port inertness and column performance and are required checks for SIM operation. DDT breakdown and tailing factor checks can be acquired as a full scan. |



Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|--|---|-----------------------------------|------------------------------|--|
| Initial Calibration (ICAL) for all analytes | At instrument set-up, prior to sample analysis. | Each analyte must meet one of the following options: RSD for each analyte \leq 20% [If pentachlorophenol is a target analyte, an RSD of \leq 40% allowed] or Linear least squares regression for each analyte: $r^2 \geq 0.99$. | Correct problem then repeat ICAL. | Flagging is not appropriate. | Minimum 5 levels required for ICAL with one calibration point at the same concentration as the daily CCV. No samples shall be analyzed until ICAL has passed. |
| Retention Time window position establishment | Once per ICAL and at the beginning of the analytical sequence. | Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used. | NA. | NA. | Calculated for each analyte. |

Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|--|---|---|------------------------------|---|
| Evaluation of Relative Retention Times (RRT) | With each sample. | RRT of each reported analyte within ± 0.06 RRT units of the mean RRT of the calibration standards. RRTs may be updated based on the daily CCV. | Correct problem, then rerun ICAL. | NA. | RRTs shall be compared with the most recently updated RRTs. Characteristic ions must maximize in the same scan or within one scan of each other. After any maintenance is performed which could affect retention times, RRTs may be updated based on the daily CCV. |
| Initial Calibration Verification (ICV) | Once after each ICAL, analysis of a second source standard prior to sample analysis. | All reported analytes within $\pm 20\%$ of true value. If pentachlorophenol is a target analyte, a %D from true value of $\pm 50\%$ is allowed. | Correct problem. Rerun ICV. If that fails, repeat ICAL. | Flagging is not appropriate. | No samples shall be analyzed until calibration has been verified with a second source. |



Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|--|--|--|--|---|--|
| Continuing Calibration Verification (CCV) | Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run. | <p>Concentration the same as the mid-point calibration standard (or lower).</p> <p>All reported analytes within $\pm 20\%$ of true value.</p> <p>If pentachlorophenol is a target analyte, a %D from true value of $\pm 50\%$ is allowed.</p> <p>All reported analytes within $\pm 50\%$ for end of analytical batch CCV.</p> | <p>Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis.</p> <p>If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) until a passing CCV is attained, and then reanalyze all associated samples since last acceptable CCV.</p> <p>Alternatively, perform an ICAL (including appropriate instrument QC) if necessary; then reanalyze all associated samples since the last acceptable CCV.</p> | <p>If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative.</p> <p>Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable calibration verification.</p> | <p>Results may not be reported without valid CCVs.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p> <p>If the specific version of a method requires additional evaluation (e.g., average RFs), these additional requirements must also be met.</p> |

Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|--------------------------------|---|--|---|---|--|
| Internal Standards (IS) | Every field sample, Standards, blanks, and QC sample. | <p>Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.</p> <p>On days when ICAL is not performed, the initial CCV is used.</p> | <p>Inspect mass spectrometer and GC for malfunctions and correct problem.</p> <p>Reanalysis of samples analyzed while system was malfunctioning is mandatory.</p> | <p>If corrective action fails in field samples, data must be qualified and explained in the Case Narrative. Apply Q-flag to analytes associated with the non-compliant IS.</p> <p>Flagging is not appropriate for failed standards.</p> | <p>Internal Standard is spiked no greater than 0.40 ng/μL concentration. According to the EPA Contract Laboratory Program Statement of Work (CLP SOW), this is the concentration of internal standard specified for SIM analysis. The SOW indicates calibration standards range from 0.10 to 1.0 ng/μL, so 0.40 ng/μL is mid-range. 1, 4-dichlorobenzene-d4 is ignored for SIM.</p> |



Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|--|--|--|--|--|--|
| Method Blank (MB) | One per preparation batch, prior to analysis of any field samples. | No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater. | Conduct investigation to determine the source of the contamination and take appropriate corrective actions. Correct problem. If required, reprep and reanalyze MB and all QC samples and field samples processed with the contaminated blank. | If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated analytical batch. | Laboratories may use the same extract for full scan and SIM analysis provided the applicable DMCS and IS are spiked at the appropriate concentrations. Results may not be reported without a valid Method Blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. |
| Laboratory Control Sample (LCS) | One per preparation batch. | A laboratory must use the QSM Appendix C Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. | Correct problem, and then reanalyze the LCS and all samples in the associated analytical batch for failed analytes if sufficient sample material is available. | If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated analytical batch. | Must contain all analytes to be reported. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed. |

Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|--|----------------------------|---|--|--|---|
| Matrix Spike (MS) | One per preparation batch. | A laboratory must use the QSM Appendix C Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. | Examine the project-specific requirements. Contact the client as to additional measures to be taken. | For the specific analyte(s) in the parent sample, apply the J-flag if acceptance criteria are not met and explain in the Case Narrative. | Must contain all analytes to be reported spiked at concentrations appropriate for SIM analysis. For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error). |
| Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD) | One per preparation batch. | A laboratory must use the QSM Appendix C Limits (8270 SIM) for batch control if project specific limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 40% (between MS and MSD or sample and MD). | Examine the project-specific requirements. Contact the client as to additional measures to be taken. | For the specific analyte(s) in the parent sample, apply the J-flag if acceptance criteria are not met and explain in the Case Narrative. | The MSD must contain all analytes to be reported spiked at concentrations appropriate for SIM analysis. All data must be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ. |



Table B-22. Organic Semi-Volatile Analysis by GC/MS in SIM Mode

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|-------------------|---|--|-------------------|--|
| Characteristic ions for MS confirmation | Minimum 3 ions. | The relative intensities of the characteristic ions of target analytes agree within 30% of the relative intensities in the reference spectrum and the relative intensities must be > 0. Confirmation requires S/N ratio of ≥ 3 for each quant and confirmation ion. | No data can be reported without MS confirmation. | NA. | Need 3 structurally significant ions that are logical fragments – not isotopic clusters. Internal standard and DMC can use fewer than 3 ions. |



24.11 Table 10 -DoD/DOE QSM 5.4 method 8270 LCS Acceptance Criteria Appendix C Table 27 for all SIM mode analysis.

| CAS ID | Analyte | N Records | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|----------|-----------------------------|-----------|-------|--------------------|---------------------|---------------------|
| 90-12-0 | 1-Methylnaphthalene | 2267 | 76.6 | 11.3 | 43 | 111 |
| 95-95-4 | 2,4,5-Trichlorophenol | 169 | 79.9 | 14.9 | 35 | 125 |
| 91-58-7 | 2-Chloronaphthalene | 615 | 76.7 | 10.5 | 45 | 108 |
| 321-60-8 | 2-Fluorobiphenyl | 1961 | 80.6 | 11.6 | 46 | 115 |
| 91-57-6 | 2-Methylnaphthalene | 2535 | 76.8 | 12.5 | 39 | 114 |
| 83-32-9 | Acenaphthene | 2813 | 77.7 | 11.2 | 44 | 111 |
| 208-96-8 | Acenaphthylene | 2761 | 77.1 | 12.8 | 39 | 116 |
| 120-12-7 | Anthracene | 2812 | 82.1 | 10.7 | 50 | 114 |
| 56-55-3 | Benz(a)anthracene | 2827 | 88 | 11.4 | 54 | 122 |
| 50-32-8 | Benzo(a)pyrene | 2789 | 87.3 | 12.5 | 50 | 125 |
| 205-99-2 | Benzo(b)fluoranthene | 2790 | 90.3 | 12.6 | 53 | 128 |
| 191-24-2 | Benzo(g,h,i)perylene | 2739 | 87.8 | 13 | 49 | 127 |
| 207-08-9 | Benzo(k)fluoranthene | 2761 | 89.3 | 11.2 | 56 | 123 |
| 111-44-4 | Bis(2-chloroethyl) ether | 192 | 65.4 | 15.8 | 18 | 113 |
| 117-81-7 | Bis(2-ethylhexyl) phthalate | 181 | 108.9 | 13.9 | 67 | 150 |
| 85-68-7 | Butyl benzyl phthalate | 144 | 103.5 | 10.6 | 72 | 135 |
| 86-74-8 | Carbazole | 183 | 79.3 | 14.6 | 36 | 123 |



Table C-27. Method 8270 SIM Solid Matrix

| CAS ID | Analyte | N Records | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|----------|------------------------|-----------|-------|--------------------|---------------------|---------------------|
| 218-01-9 | Chrysene | 2812 | 87.5 | 10.2 | 57 | 118 |
| 84-74-2 | Di-n-butyl phthalate | 150 | 106.5 | 12.9 | 68 | 145 |
| 117-84-0 | Di-n-octyl phthalate | 144 | 105.5 | 16.8 | 55 | 156 |
| 53-70-3 | Dibenzo(a,h)anthracene | 2778 | 89.2 | 13.2 | 50 | 129 |
| 132-64-9 | Dibenzofuran | 282 | 71.9 | 12.2 | 35 | 108 |
| 84-66-2 | Diethyl phthalate | 147 | 99.3 | 10.9 | 67 | 132 |
| 131-11-3 | Dimethyl phthalate | 149 | 99.3 | 9.3 | 71 | 127 |
| 206-44-0 | Fluoranthene | 2782 | 87.3 | 10.7 | 55 | 119 |
| 86-73-7 | Fluorene | 2795 | 80.6 | 11.2 | 47 | 114 |
| 118-74-1 | Hexachlorobenzene | 201 | 81.9 | 14.2 | 39 | 125 |
| 193-39-5 | Indeno(1,2,3-cd)pyrene | 2812 | 89.6 | 13.5 | 49 | 130 |
| 62-75-9 | n-Nitrosodimethylamine | 117 | 90.7 | 10.9 | 58 | 124 |
| 91-20-3 | Naphthalene | 2823 | 74.7 | 12.2 | 38 | 111 |
| 87-86-5 | Pentachlorophenol | 259 | 82.4 | 15.5 | 36 | 129 |
| 85-01-8 | Phenanthrene | 2792 | 80.8 | 10.6 | 49 | 113 |
| 129-00-0 | Pyrene | 2792 | 85.8 | 10.2 | 55 | 117 |



24.12 Table 11 -DoD/DOE QSM 5.4 method 8270 LCS Acceptance Criteria Appendix C Table 28 for all SIM mode analysis.

Table C-28. Method 8270 SIM Water Matrix

| CAS ID | Analyte | N Records | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|----------|-----------------------|-----------|------|--------------------|---------------------|---------------------|
| 92-52-4 | 1,1-Biphenyl | 106 | 77.3 | 7.3 | 56 | 99 |
| 90-12-0 | 1-Methylnaphthalene | 2566 | 77.9 | 12.5 | 41 | 115 |
| 95-95-4 | 2,4,5-Trichlorophenol | 488 | 84.1 | 13.4 | 44 | 124 |
| 118-79-6 | 2,4,6-Tribromophenol | 164 | 83.7 | 12.7 | 46 | 122 |
| 606-20-2 | 2,6-Dinitrotoluene | 118 | 67.2 | 15.8 | 20 | 115 |
| 91-58-7 | 2-Chloronaphthalene | 717 | 72.4 | 12.7 | 34 | 111 |
| 321-60-8 | 2-Fluorobiphenyl | 747 | 79.2 | 8.8 | 53 | 106 |
| 91-57-6 | 2-Methylnaphthalene | 2984 | 76.5 | 12.6 | 39 | 114 |
| 83-32-9 | Acenaphthene | 3241 | 80.9 | 11.1 | 48 | 114 |
| 208-96-8 | Acenaphthylene | 3234 | 77.8 | 14.4 | 35 | 121 |
| 120-12-7 | Anthracene | 3224 | 85.8 | 11 | 53 | 119 |



Table C-28. Method 8270 SIM Water Matrix

| CAS ID | Analyte | N Records | Mean | Standard Deviation | Lower Control Limit | Upper Control Limit |
|-----------|-----------------------------|-----------|-------|--------------------|---------------------|---------------------|
| 56-55-3 | Benz(a)anthracene | 3277 | 89.3 | 10.1 | 59 | 120 |
| 50-32-8 | Benzo(a)pyrene | 3284 | 86.4 | 11.2 | 53 | 120 |
| 205-99-2 | Benzo(b)fluoranthene | 3248 | 89.7 | 12.3 | 53 | 126 |
| 191-24-2 | Benzo(g,h,i)perylene | 3178 | 86 | 14.1 | 44 | 128 |
| 207-08-9 | Benzo(k)fluoranthene | 3167 | 89.3 | 11.9 | 54 | 125 |
| 111-44-4 | Bis(2-chloroethyl) ether | 775 | 77.8 | 12.6 | 40 | 116 |
| 117-81-7 | Bis(2-ethylhexyl) phthalate | 275 | 114.1 | 19.6 | 55 | 173 |
| 85-68-7 | Butyl benzyl phthalate | 159 | 90.7 | 17.3 | 39 | 143 |
| 86-74-8 | Carbazole | 631 | 84 | 13.1 | 45 | 123 |
| 218-01-9 | Chrysene | 3215 | 88.3 | 10.4 | 57 | 120 |
| 84-74-2 | Di-n-butyl phthalate | 153 | 102.5 | 14.2 | 60 | 145 |
| 117-84-0 | Di-n-octyl phthalate | 157 | 103.3 | 19 | 46 | 160 |
| 53-70-3 | Dibenzo(a,h)anthracene | 3233 | 87.2 | 14.5 | 44 | 131 |
| 132-64-9 | Dibenzofuran | 864 | 77.5 | 14.1 | 35 | 120 |
| 84-66-2 | Diethyl phthalate | 142 | 94.5 | 13.5 | 54 | 135 |
| 206-44-0 | Fluoranthene | 3242 | 89.1 | 10.4 | 58 | 120 |
| 86-73-7 | Fluorene | 3232 | 84.1 | 11.3 | 50 | 118 |
| 118-74-1 | Hexachlorobenzene | 947 | 84.8 | 13 | 46 | 124 |
| 87-68-3 | Hexachlorobutadiene | 187 | 84.5 | 14.7 | 40 | 129 |
| 193-39-5 | Indeno(1,2,3-cd)pyrene | 3244 | 88.7 | 13.7 | 48 | 130 |
| 62-75-9 | N-Nitrosodimethylamine | 162 | 62.5 | 10 | 33 | 92 |
| 91-20-3 | Naphthalene | 3277 | 78.8 | 11.9 | 43 | 114 |
| 4165-60-0 | Nitrobenzene-d5 | 444 | 83.1 | 9.2 | 55 | 111 |
| 87-86-5 | Pentachlorophenol | 808 | 88.4 | 17.6 | 36 | 141 |
| 85-01-8 | Phenanthrene | 3240 | 83.6 | 10.3 | 53 | 115 |
| 129-00-0 | Pyrene | 3252 | 87.1 | 11.3 | 53 | 121 |
| 1718-51-0 | Terphenyl-d14 | 642 | 95.1 | 12.4 | 58 | 132 |

24.13 Table 12 – 1,4-Dioxane water LCS Acceptance Criteria

LCS Acceptance Range for Water for Method SIM 8270

| Analyte | Acceptable % Recovery |
|-------------|-----------------------|
| 1,4-Dioxane | 59-139 |



STANDARD OPERATING PROCEDURE

For

Method EPA Draft Method 1633 and PFAS Compliant with Table B-24, DOD, QSM,
Version 5.4 Requirements

DETERMINATION OF PER- AND POLYFLUOROALKYL SUBSTANCES IN DRINKING WATER,
WATER, OR SOILS BY ISOTOPE DILUTION ANION EXCHANGE SOLID PHASE EXTRACTION
AND LIQUID CHROMATOGRAPHY/TANDEM MASS SPECTROMETRY

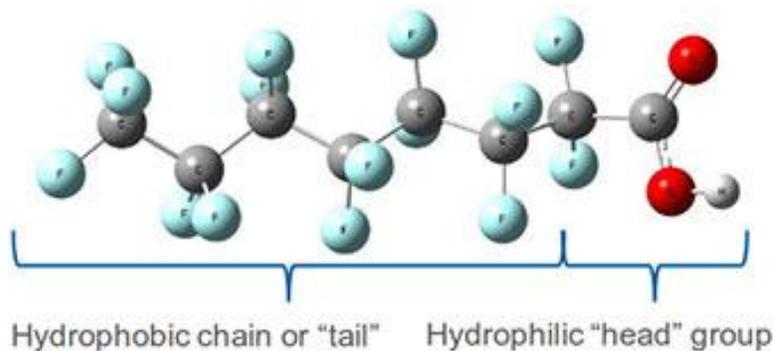




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1.0 Identification of Test Method

- 1.1 This is a solid phase extraction (SPE) liquid chromatography-tandem mass spectrometry (LC-MS/MS) method for the determination of select per- and polyfluoroalkyl substances (PFAS) in non-potable water and solids (soil, biosolids and sediment). Method 1633 requires the use of MS/MS in Multiple Reaction Monitoring (MRM) mode to enhance selectivity. Accuracy and precision data have been generated in reagent water and Ottawa sand for the compounds included in the Analyte List. Sample concentrations are determined by isotope dilution or extracted internal standard quantification using isotopically labeled compounds added to the samples before extraction. Analytes are reported in their acid form concentration.
- 1.2 This method is intended for use by analysts skilled in the performance of solid phase extractions, the operation of LC-MS/MS instrumentation, and the interpretation of the associated data.
- 1.3 This SOP deviates from the EPA 1633 referenced method in the following ways:
 - 1.3.1 The SPE procedure for aqueous samples was modified so that instead of cartridge reservoirs, a large volume sample delivery system is used for sample filtration.
 - 1.3.2 The hand-shake stage for the carbon clean-up is not performed for waters or soils extraction, and the centrifuge time for the carbon exposed sample has been reduced for the soil extraction. During method development, the carbon-clean-up SOP conditions for vortexing and centrifuging the samples produced the most optimal extraction efficiency.
 - 1.3.3 The NIS solution is added to a filtered 1.0mL aliquot of each batch QC, matrix QC, and field sample extract instead of the entire 5mL eluent; this modification saves on material cost and does not interfere with the NIS quality criteria.
 - 1.3.4 The HPLC run program was optimized for laboratory performance, efficiency, and materials cost. Including but not limited to the injection volume, columns, mobile phase, gradients, MS optimization conditions, etc. – all of which are further defined in this SOP. Note: the gradient and initial temperatures may require slight adjustments to accommodate the bile salt retention time criteria; such adjustments will not significantly affect the sensitivity or instrument performance (accuracy and precision).

Note: These deviations from the referenced test method are the best conditions for our instrumentation or client's needs and represent improved performance over the referenced test method conditions. Modifications may be made to improve performance (e.g., overcome interferences, or improve the sensitivity, accuracy, or precision of the results) provided that all performance criteria in this method are met and all modifications are well documented. Requirements for establishing equivalency are in Section 12.5 and 12.6.

2.0 Applicable Matrix or matrices

- 2.1 Method 1633 is applicable to non-potable water samples and solids (soil, biosolids and sediment).



3.0 Detection Limit

- 3.1 The method detection limit is determined in accordance with AEL SOP ADMIN-012, referencing 40CFR136, appendix B.
- 3.2 For the current MDLs, see the electronic version specific for the method, instrument, and matrix in the “AEL-QA” folder on the lab server.

4.0 Scope and Application, including components to be analyzed.

- 4.1 Both branched and linear PFAS isomers may be found in the environment. This method includes procedures for summing the contribution of multiple isomers to the final reported concentration. In those cases where standard materials containing multiple isomers are commercially available, laboratories should obtain such standards for the method analytes.
- 4.2 See Table 3 in Section 24 for a list of compounds and their characteristic ions that have been evaluated.
- 4.3 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatograph/mass spectrometers and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method.

4.4 Table 4.4 – Analyte List by Method

| Analyte* | Abbreviation | CAS Number | Acid Group |
|--|------------------|------------|---|
| 1H, 1H, 2H, 2H-Perfluorooctanesulfonic acid (6:2 Fluorotelomersulfonate) | 6:2FTS | 27619-97-2 | Fluorotelomer sulfonic acids |
| Perfluorooctanesulfonic acid | PFOS | 1763-23-1 | Perfluoroalkyl sulfonic acids |
| Perfluorooctanoate (Perfluorooctanoic acid) | PFOA | 335-67-1 | Perfluoroalkyl carboxylic acids |
| Perfluoropentanoate (Perfluoropentanoic acid) | PFPeA | 2706-90-3 | Perfluoroalkyl carboxylic acids |
| Perfluoropentanesulfonic acid | PFPeS | 2706-91-4 | Perfluoroalkyl sulfonic acids |
| Perfluoroundecanoate (Perfluoroundecanoic acid) | PFUnA | 2058-94-8 | Perfluoroalkyl carboxylic acids |
| Perfluorotetradecanoate (Perfluorotetradecanoic acid) | PFTeDA (PFTA) | 376-06-7 | Perfluoroalkyl carboxylic acids |
| Perfluorotridecanoate (Perfluorotridecanoic acid) | PFTriA (PFTriDA) | 72629-94-8 | Perfluoroalkyl carboxylic acids |
| Perfluorooctane sulfonamide | FOSA (PFOSA) | 754-91-6 | Perfluorooctane sulfonamides |
| N-Methylperfluorooctane sulfonamido acetic acid | NMeFOSAA | 2355-31-9 | Perfluorooctane sulfonamidoacetic acids |



Table 4.4 – Analyte List by Method (Continued)

| Analyte* | Abbreviation | CAS Number | Acid Group |
|--|--------------------|--------------|---|
| N-Ethylperfluorooctane sulfonamido acetic acid | NEtFOSAA | 2991-50-6 | Perfluorooctane sulfonamidoacetic acids |
| Perfluorononanesulfonate (Perfluorononane sulfonic acid) | PFNS | 68259-12-1 | Perfluoroalkyl sulfonic acids |
| Perfluorodecane sulfonate (Perfluorodecane sulfonic acid) | PFDS | 335-77-3 | Perfluoroalkyl sulfonic acids |
| 3-Perfluoropropyl propanoic acid | 3:3FTCA | 356-02-5 | Fluorotelomer carboxylic acids |
| 2H,2H,3H,3H-Perfluorooctanoic acid | 5:3FTCA | 914637-493-3 | Fluorotelomer carboxylic acids |
| 3-Perfluoroheptyl propanoic acid | 7:3FTCA | 812-70-4 | Fluorotelomer carboxylic acids |
| N-ethyl perfluorooctanesulfonamide | NEtFOSA | 4151-50-2 | Perfluorooctane sulfonamides |
| N-methyl perfluorooctanesulfonamide | NMeFOSA | 31506-32-8 | Perfluorooctane sulfonamides |
| N-ethyl perfluorooctanesulfonamidoethanol | NEtFOSE | 1691-99-2 | Perfluorooctane sulfonamide ethanols |
| N-methyl perfluorooctanesulfonamidoethanol | NMeFOSE | 24448-09-7 | Perfluorooctane sulfonamide ethanols |
| Perfluorododecanesulfonic acid | PFDoS | 79780-39-5 | Perfluoroalkyl sulfonic acids |
| 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid | 11C1-PF3OUdS | 763051-92-9 | Ether sulfonic acids |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid | 9C1-PF3ONS | 756426-58-1 | Ether sulfonic acids |
| 4,8-Dioxa-3H-perfluorononanoic acid | ADONA | 919005-14-4 | Per- & Polyfluoroether carboxylic acids |
| Hexafluoropropylene oxide dimer acid (GenX or Propanoic acid) | HFPO-DA (PFPrOPrA) | 13252-13-6 | Per- & Polyfluoroether carboxylic acids |
| Nonafluoro-3,6-dioxaheptanoic acid | NFDHA | 151772-58-6 | Per- & Polyfluoroether carboxylic acids |
| Perfluorobutanoate (Perfluorobutyric acid, Perfluorobutanoic acid) | PFBA | 375-22-4 | Perfluoroalkyl carboxylic acids |
| Perfluorobutane sulfonate (Perfluorobutanesulfonic acid) | PFBS | 375-73-5 | Perfluoroalkyl sulfonic acids |
| 1H, 1H, 2H, 2H-Perfluorodecanesulfonic acid (8:2) | 8:2FTS | 39108-34-4 | Fluorotelomer sulfonic acids |



Table 4.4 – Analyte List by Method (Continued)

| Analyte * | Abbreviation | CAS Number | Acid Group |
|---|--------------|-------------|---|
| Perfluorodecanoate (Perfluorodecanoic acid) | PFDA | 335-76-2 | Perfluoroalkyl carboxylic acids |
| Perfluorododecanoate (Perfluorododecanoic acid) | PFDoA | 307-55-1 | Perfluoroalkyl carboxylic acids |
| Perfluoro(2-ethoxyethane)sulfonic acid | PFEESA | 113507-82-7 | Ether sulfonic acids |
| Perfluoroheptane Sulfonate (Perfluoroheptane sulfonic acid) | PFHpS | 375-92-8 | Perfluoroalkyl sulfonic acids |
| Perfluoroheptanoate (Perfluoroheptanoic acid) | PFHpA | 375-85-9 | Perfluoroalkyl carboxylic acids |
| 1H, 1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2 Fluorotelomersulfonate) | 4:2FTS | 757124-72-4 | Fluorotelomer sulfonic acids |
| Perfluorohexane sulfonate (Perfluorohexanesulfonic acid) | PFHxS | 355-46-4 | Perfluoroalkyl sulfonic acids |
| Perfluorohexanoate (Perfluorohexanoic acid) | PFHxA | 307-24-4 | Perfluoroalkyl carboxylic acids |
| Perfluoro-3-methoxypropanoic acid | PFMPA | 377-73-1 | Per- & Polyfluoroether carboxylic acids |
| Perfluoro-4-methoxybutanoic acid | PFMBA | 863090-89-5 | Per- & Polyfluoroether carboxylic acids |
| Perfluorononanoate (Perfluorononanoic acid) | PFNA | 375-95-1 | Perfluoroalkyl carboxylic acids |

*Some PFAS are commercially available as ammonium, sodium, and potassium salts. This method measures all forms of the analytes as anions while the identity of the counterion is inconsequential. Analytes may be purchased as acids or as any of the corresponding salts.

5.0 Summary of Method

- 5.1 For aqueous samples, a 500 mL sample is fortified with isotopically labeled analogues of the method analytes that function as isotope dilution standards. The sample is passed through an SPE cartridge containing weak anion exchange, mixed-mode polymeric sorbent that has been pre-conditioned with washes of 1% methanolic ammonium hydroxide and 0.3M formic acid, to extract the method analytes and isotope dilution analogues. The cartridge is rinsed with sequential washes of reagent water and followed by washes of 1:1 0.1M formic acid/methanol, and then the compounds are eluted from the solid phase sorbent with 1% methanolic ammonium hydroxide. Acetic acid is added to the extract and vortexed. The extract is then cleaned with carbon, centrifuged and filtered through a 0.2um syringe filter. 1.0mL of the extract is then added to a 2.0mL polyethylene autosampler vial and 10uL of the injected internal standard is added.
- 5.2 For solid samples, 5.0g sample is fortified with isotopically labeled analogues of the method analytes that function as isotope dilution standards. The sample is extracted on an autosampler table with 0.3% methanolic ammonium hydroxide 3 times, decanting and collecting the supernatant in a



clean 50mL centrifuge tube. The extract is then cleaned with carbon, centrifuged and decanted into another clean 50mL centrifuge tube. The extract is then concentrated using a N-Evap nitrogen blowdown to a known volume (approximately 12-19mL). The extract volume is then brought up to 50mL with reagent water and pH adjusted as needed. The extract is then extracted using a SPE column following the same procedure used for aqueous samples. The cartridge is then eluted with 1% methanolic ammonium hydroxide, acetic acid added, vortexed and filtered using a 0.2um syringe filter. 1.0 ml of the extract is added to a 2.0mL polyethylene autosampler vial and 10uL of the injected internal standard is added.

- 5.3 Extracts are analyzed by LC-MS/MS in the MRM detection mode. The concentration of each analyte is calculated using the isotope dilution technique. For QC purposes, the percent recoveries of the isotope dilution analogues are calculated using the integrated peak areas of isotope performance standards, which are added to the final extract and function as traditional internal standards, exclusively applied to the isotope dilution analogues.

6.0 Definitions

See also AEL ADMIN SOP-039 Laboratory Definitions

- 6.1 Analyte – A PFAS compound included in this method. The analytes are listed in Table 4.4.
- 6.2 Compound – One of many variants or configurations of a common chemical structure. Individual compounds are identified by the number of carbon atoms and functional group attached at the end of the chain.
- 6.3 Class A glassware – Volumetric glassware that provides the highest accuracy. Class A volumetric glassware complies with the Class A tolerances defined in ASTM E694, must be permanently labeled as Class A, and is supplied with a serialized certificate of precision.
- 6.4 Extraction Batch – A group of field samples with similar matrices, which are prepared at the same time in the same location using the same procedure and processed as a unit. A Method Blank, a Laboratory Control Sample, a Matrix Spike, and a Duplicate Matrix Spike must accompany each extraction batch of 20 or fewer field samples.
- 6.5 Continuing Calibration Verification (CCV); also known as a Continuing Calibration Check (CCC) – A known interference free matrix spiked with a known concentration (near the mid-point of the Initial Calibration) of the target analytes. The CCV is analyzed 1) at the beginning of a 24-hour analytical run at the MRL concentration, and 2) every 10 field samples, and 3) at the end of the analytical sequence at alternating concentrations between mid and high calibration levels. The CCV is used to verify that the instrument calibration is in control before and after sample analysis.
- 6.6 DoD (DOD) – Acronym for Department of Defense.
- 6.7 Extracted internal standard (EIS) quantification – The response of the target compound is compared to the response of the labeled analog of another compound in the same LOC.
- 6.8 Internal standard quantitation – A means of determining the concentration of (1) a naturally occurring (native) compound by reference to a compound other than its labeled analog and (2) a labeled compound by reference to another labeled compound



- 6.9 Initial Calibration (ICAL) – Analysis of analytical standards at different concentrations that are used to determine and calibrate the quantitation range of the response of the analytical detector or method. The ICAL levels are solutions of the method analytes, isotope dilution analogues, and isotope performance standards prepared from the working standards and intermediate standards.
- 6.10 Calibration Curve – A calibration or standard curve is a set of solutions, prepared from a secondary and/or stock solution, used to calibrate the response of the LC-MS/MS instrument. The calibration curve plots known standard concentrations of an analyte versus the instrument response for that analyte.
- 6.11 Initial Calibration Verification (ICV); also known as a Quality Control Standard (QCS) – A known interference free matrix spiked with a known concentration (near the mid-point of the Initial Calibration) of the target analytes and isotope analogues. ICV standards are made from a stock solution that is different from the stock used to prepare calibration standards. This standard is analyzed immediately after the calibration to confirm the usability of the calibration.
- 6.12 Instrument Blank (IB) – An instrument blank is an aliquot of the method solvent, containing no analytes of interest. The purpose of an IB is to ensure that the analytical system is free from contamination associated with the instrument analysis. An IB also provides one way of determining the level of noise and baseline rise attributable solely to the analytical system, in the absence of any other analytes or non-analytical related contaminants. The blank should contain the internal standard.
- 6.13 Internal Standard (IS) – An internal standard is pure analytes added to a sample, extract, or standard solution in a known amount. The IS is used to measure the relative responses of other method analytes that are components of the same sample of solution. The internal standard must be an analyte that is not a sample component.
- 6.14 Field Reagent Blank (FRB) – An aliquot of reagent water that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are introduced into the sample from shipping, storage, and the field environment.
- 6.15 Method Blank (MB); also referred to as Laboratory Reagent Blank (LRB) – An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is fortified with the isotope dilution analytes and then carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process: the laboratory environment, the reagents, glassware, or extraction apparatus.
- 6.16 Laboratory Control Sample (LCS); also referred to as Laboratory Fortified Blank (LFB), and Ongoing Precision and Recovery Standard (OPR) – A known interference free matrix spiked prior to sample extraction with a known concentration of the method analytes and isotope dilution analogues. The LCS is analyzed exactly like a sample; the purpose of the LCS is to monitor analytical control for the batch. Percent recoveries are calculated for each of the analytes.



- 6.17 Laboratory Control Sample Duplicate (LCSD) – A second known interference free matrix spiked prior to sample extraction with a known concentration of standard. Analyses of duplicates indicate precision associated specifically with the laboratory procedures, removing any associated variables that might occur during sample collection, preservation, or storage procedures.
- 6.18 Sample Duplicate (DUP); also known as Field Duplicate (FD) – Separate samples collected at the same time and sampling location, shipped, and stored under identical conditions. Method precision, including the contribution from sample collection procedures, is estimated from the analysis of Field Duplicates. Field Duplicates are used to prepare Laboratory Fortified Sample Matrix and Laboratory Fortified Sample Matrix Duplicate QC samples. For the purposes of this method, Field Duplicates are collected to support potential repeat analyses (if the original field sample is lost or if there are QC failures associated with the analysis of the original field sample).
- 6.19 Matrix Spike/Matrix Spike Duplicate (MS/MSD); also referred to as Laboratory Fortified Matrix/ Laboratory Fortified Matrix Duplicate (LFM/LFMD) – An aliquot of a client supplied sample that is chosen at random, to which known quantities of the method analytes and isotope dilution analogues are added in the laboratory. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the methods used for the analyses. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the values in the MS corrected for the background concentration. Matrix Spikes are analyzed with every batch or per 20 samples. The LFSMD is used instead of the Field Duplicate to assess method precision when the method analytes are rarely found at concentrations greater than the MRL.
- 6.20 Matrix – The substrate (e.g., water, drinking water, etc.), which may contain the analyte of interest.
- 6.21 Liquid Sample – A sample classified as a groundwater, surface water, wastewater or other water-soluble liquid.
- 6.22 Solid Sample – A sample classified as soil, solid, or sludge.
- 6.23 Representative Sample: A well-mixed aliquot of sample that constitutes an accurate representation of contents within the container. Methods used to achieve a representative sub-sample are described below:
- 6.23.1 Aqueous/Liquid samples.
 - 6.23.1.1 Sample is shaken until homogeneous and then poured or pipetted into appropriate container.
 - 6.23.2 Soil Samples:
 - 6.23.2.1 If container size is sufficient, sample is mixed within until homogenous. If container size is insufficient, the entire sample is transferred to an appropriate container and mixed.
 - 6.23.3 Miscellaneous Solid Samples:



6.23.3.1 Sample is crushed, pulverized, shaken, and stirred as appropriate to ensure the aliquot used for analysis represents the entire contents of the original sample container as accurately as possible.

- 6.24 PFAS – Per- and Polyfluoroalkyl substances – A group of man-made fluorinated compounds that are hydrophobic and lipophobic, manufactured and used in a variety of industries globally. These compounds are persistent in the environment as well as in the human body. This method analyzes for the PFAS listed in Table 4.4.
- 6.25 Isotopically labeled compound – An analog of a target analyte in the method which has been synthesized with one or more atoms in the structure replaced by a stable (non-radioactive) isotope of that atom. Common stable isotopes used are ^{13}C (Carbon-13) or Deuterium (D or ^2H). These labeled compounds do not occur in nature, so they can be used for isotope dilution quantitation or other method-specific purposes.
- 6.26 Isotope Dilution Technique – An analytical technique for measuring analyte concentration using the ratio of the peak area of the native analyte to that of an isotopically labeled analogue, added to the original sample in a known amount and carried through the entire analytical procedure. The isotopically enriched PFAS are spiked into each sample and allow identification and correction of the concentration of the native compounds in the analytical process.
- 6.27 Isotope Performance Standards (IPS); also known as Non-extracted Internal Standards (NIS) – Quality control compounds that are added to all standard solutions and extracts in a known amount and used to measure the relative response of the isotopically labelled analogues (ISA or EIS) that are components of the same solution. The isotope performance standards are indicators of instrument performance and are used to calculate the recovery of the isotope dilution analogues through the extraction procedure. In this method, the isotope performance standards are not used in the calculation of the recovery of the native analytes.
- 6.28 Precursor Ion – The gas-phase species corresponding to the method analyte that is produced in the electrospray ionization interface. During tandem mass spectrometry, or MS/MS, the precursor ion is mass selected and fragmented by collision-activated dissociation to produce distinctive product ions of smaller mass to charge (m/z) ratio. For this method, the precursor ion is usually the deprotonated molecule ($[\text{M} - \text{H}]^-$) of the method analyte, except for HFPO-DA. For this analyte, the precursor ion is formed by decarboxylation of HFPO-DA.
- 6.29 Product Ion – One of the fragment ions that is produced in MS/MS by collision-activated dissociation of the precursor ion.
- 6.30 Isotope Dilution Analogues (Extracted Internal Standard (EIS) Analytes) – Isotopically labeled analogues of the method analytes that are added to the sample prior to extraction in a known amount. Note: Not all target PFAS currently have an isotopically labelled analogue. In these cases, an alternate isotopically labelled analogue is used. All listed isotope dilution analogues must be used, if available. Linear isomers are recommended to simplify peak integration. These analogues were chosen during method development because they encompass most of the functional groups, as well as the molecular weight range of the method analytes.
- 6.31 Stock Standards Solution, also known as Quantitative Standard – A concentrated solution of one or more target analytes at a known concentration, purchased from a reputable commercial



vendor, and having Certificates of Analysis. Stock standard solutions are used to prepare working calibration standards. Stock standards once opened must be replaced after 1 year or sooner if routine QC indicates a problem.

- 6.32 Intermediate Standard, also known as Primary Dilution Standard (PDS) – A solution of target analytes at known concentration prepared from one or more Stock standards. The Intermediate Standard may be used to prepare the Working Calibration Standard.
- 6.33 Working Calibration Standard (WS) – A solution of all the target analytes at a known concentration prepared either from one or more intermediate calibration standards and/or from one or more stock standard solutions. Working standards once made must be replaced after 6 months or sooner if routine QC indicates a problem.
- 6.34 Technical-Grade Standard – As defined for this method, a technical-grade standard includes a mixture of the branched and linear isomers of a method analyte. For the purposes of this method, technical-grade standards are used to identify retention times of branched and linear isomers of method analytes.
- 6.35 Analysis Window or Analysis Batch – Samples are analyzed in a time frame referred to as a “window.” The window is initiated with the analysis of the continuing calibration verification (CCV) standard. If the CCV passes the specific criteria, then samples, bracketing CCV, and a closing CCV are analyzed until the 24-hour time limit expires.
- 6.36 Surrogate Analyte (Surr) – A surrogate analyte is an organic compound that is similar to the analytes of interest in chemical composition, extraction characteristics, and chromatography, but is not normally found in environmental samples. The purpose of the surrogate analyte is to evaluate the preparation and analysis of the samples. These compounds are spiked into blanks, standards, samples, and matrix spiked samples prior to analysis. Percent recoveries are calculated for each surrogate and are used to evaluate the method performance.
- 6.37 LC-MS/MS; also known as LC QQQ (triplequad) - used as an abbreviation for liquid chromatograph with a tandem mass spectrometer. When MS abbreviated alone, it is always defined as matrix spike, when MS used in conjunction with LC as in LC-MS/MS, it is always defined as mass spectrometer.
- 6.38 Multiple Reaction Monitoring (MRM) – Application of selected reaction monitoring to multiple product ions from one or more precursor ions. Method 1633 requires the use of MS/MS in Multiple Reaction Monitoring (MRM) mode to enhance selectivity.
- 6.39 Signal-to-noise ratio (S/N) – The height of the signal as measured from the mean (average) of the noise to the peak maximum divided by the mean height of the noise.
- 6.40 SPE – Solid-phase extraction; a technique in which an analyte is extracted from an aqueous solution or a solid/tissue extract by passage over or through a material capable of reversibly adsorbing the analyte. Also termed liquid-solid extraction.
- 6.41 Initial Demonstration of Capability (IDOC), also known as Initial precision and recovery (IPR) – four aliquots of a reference matrix spiked with the analytes of interest and labeled compounds and analyzed to establish the ability of the laboratory to generate acceptable precision and recovery. An IDOC is performed prior to the first time this method is used and any time the



method or instrumentation is modified.

6.42 Case Narrative (CN) – A case narrative is simply a means of describing exactly what transpired with the samples during the analytical process. Case narratives are required for variances that occur within a project.

6.43 Non-Conformity Form (NCF) – A form which will be completed and processed for each QC failure or deviation from normal protocol that occurs outside the scope of normal operation as defined by the AEL QM Section 10, AEL SOP Admin-016, and Method SOP.

6.44 Semi-volatile department abbreviations used in sample preparation logbooks, data printouts and other Semi-volatile areas:

- 6.44.1 RR/RA – Rerun/Reanalyze
- 6.44.2 CF – Confirmation
- 6.44.3 NR – Not a Real Hit
- 6.44.4 NAP – Not a Peak
- 6.44.5 DNC – Does Not Confirm
- 6.44.6 STR – Straight/No Dilution
- 6.44.7 NT – Not Target
- 6.44.8 WRT – Wrong Retention Time
- 6.44.9 DNR – Do Not Report
- 6.44.10 BDL –Below Detection Limit
- 6.44.11 FH – Fails High
- 6.44.12 FL – Fails Low
- 6.44.13 DF – Dilution Factor
- 6.44.14 DIL – Dilution
- 6.44.15 STD – Standard
- 6.44.16 IS – Internal Standard
- 6.44.17 Surr – Surrogate
- 6.44.18 OOT – Out of Tune/CCV Window
- 6.44.19 WS – Working Standard



- 6.45 Safety Data Sheets (SDS) Safety Data Sheets (SDS) – Written information provided by vendors concerning a chemical’s toxicity, health hazards, physical properties, fire, and reactivity data, including storage, spill, and handling precautions.
- 6.46 Limit of Detection (LOD) – The LOD is **not** synonymous with the MDL. The LOD is an estimate of the minimum amount of a substance that an analytical process can reliably detect with a high level of confidence (99% Confidence; that is a false negative rate of 1%). The LOD is at the level of the MDL. The LOD must go through all the same processes that a sample will go through and be detected above instrument noise level.
- 6.47 Limits of Quantitation (LOQ) – The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specific degree of confidence. It is also the lowest concentration that produces a quantitative result within specified limits of precision and bias. For DOD work the LOQ will be set at or above the concentration of the lowest initial calibration standard. For DOD work the LOQ will be the concentration at which the PQL is verified. The LOQ can equal the PQL but is not synonymous with the PQL.
- 6.48 Limits of Quantitation (LOQ) Verification – LOQ verifications are a spiked clean matrix sample that must go through all the same processes which regular samples will go through and be within the precision and bias acceptance criteria of the method. The LOQ verifications are spiked at the concentrations of the LOQ.
- 6.49 Method Detection Limit (MDL) – The MDL is an estimate of the minimum amount of a substance that an analytical process can readily detect. The MDL is the minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined most often as 3.14 times the standard deviation of a low level, seven replicate study; however, it can be determined for some methods as the lowest increment measurable with confidence. MDLs are determined for each analyte, matrix, prep method, cleanup method, analysis method, and instrument. (See each method’s requirements). The MDL is one way to establish a Detection Limit, not a Limit of Detection.
- 6.50 Method Detection Limit (MDL) Verification – MDL verifications are spiked clean matrix samples that must go through all the same processes that a regular sample will go through and be detected above instrument noise level. For labs accredited under DOD ELAP, the MDL verifications must be performed immediately after the initial MDL study and on a quarterly basis thereafter. For labs accredited under NELAC (TNI) Standards only, MDL verifications are to be performed immediately after the initial MDL study and on a yearly basis thereafter.
- 6.51 Minimum Reporting Level (MRL) – The minimum concentration that may be reported by a laboratory as a quantified value for a method analyte. For each method analyte, the concentration of the lowest calibration standard must be at or below the MRL and the laboratory must demonstrate its ability to meet the MRL per the criteria defined in this method.
- 6.52 Practical Quantitation Level (PQL); also known as the Method Reporting Limit (MRL or RL) – the lowest calibration standard or lowest quantitation level for the method and matrix. The concentration below which data is to be qualified as having less certainty. PQLs are at a concentration greater than that of the MDL.



6.53 Qualifier Codes (For Florida and FDEP work)

- 6.53.1 A - Value reported is the mean (average) of two or more determinations. This code shall be used if the results of two or more discrete and separate samples are averaged. These samples shall have been processed and analyzed (e.g., laboratory replicate samples, field duplicates, etc.) independently. Do not use this code if the data is the result of replicate analyses on the same sample aliquot, extract or digestate. Under most conditions, replicate values shall be reported as individual analyses.
- 6.53.2 I - The reported Value is between the laboratory method detection limit (MDL) and the laboratory practical quantitation limit (PQL).
- 6.53.3 K- Off scale low.
- 6.53.4 L- Off scale high. Use if reporting above the acceptable level of quantitation.
- 6.53.5 U- Indicates that a compound was analyzed for but not detected. The value associated with the qualifier will be the MDL.
- 6.53.6 V- Indicates that the analyte was detected in both the sample and the associated method blank. NOTE: The method blank value **cannot** be subtracted from the associated sample to give a result. The sample result will be reported as is with the "V" qualifier.
- 6.53.7 H - Value based on field kit determination; results may not be accurate. This code shall be used if a field-screening test (i.e., field gas chromatograph data, immunoassay, vendor-supplied field kit, etc.) was used to generate the value and the field kit or method has not been recognized by the Department as equivalent to laboratory methods.
- 6.53.8 O - Sampled, but analysis lost or not performed. NOTE: if reporting data to STORET, a numerical value must be entered. Such values are not meaningful and shall not be used.
- 6.53.9 Q - Sample held beyond the accepted holding time. This code shall be used if the value is derived from a sample that was prepared and/or analyzed AFTER the approved holding time restrictions for sample preparation and analysis.
- 6.53.10 Y - The laboratory analysis was from an unpreserved or improperly preserved sample. The data may not be accurate.
- 6.53.11 REJ - Data is rejected and should not be used. Some or all of the quality control data for the analyte were outside criteria, and the presence or absence of the analyte cannot be determined from the data.
- 6.53.12 NAI - Not analyzed due to interference.
- 6.53.13 J - Estimated value; value not accurate.
- 6.53.13.1 This code shall be used in the following instances:



6.53.13.1.1 "1" Surrogate recovery limits have been exceeded.

6.53.13.1.2 "2" No known quality control criteria exists for the component.

6.53.13.1.3 "3" The reported value failed to meet the established quality control criteria for either precision or accuracy.

6.53.13.1.4 "4" The sample matrix interfered with the ability to make any accurate determination; or

6.53.13.1.5 "5" The data is questionable because of improper laboratory or field protocols (e.g., composite sample was collected instead of a grab sample).

6.53.13.2 A "J" value shall be accompanied by justification for its use (ex. J(4)).

6.53.13.3 A "J" value shall not be used if another code applies (ex. K, L, M, T, V, Y, PQL).

6.53.14 If more than one code applies, and the data is to be entered into STORET, only one code shall be reported. The code shall be selected based on the following hierarchy: REJ, NAI, O, Y, V, H, J, B, K, L, M, PQL, T, Z, A.

7.0 Interferences

7.1 Sources of interference in this method can be grouped into three broad categories.

7.1.1 Contaminated solvents, reagents, or sample processing hardware.

7.1.2 Contaminated desolvation or collision gas, LC flow path components, column surfaces, MS source surfaces, capillary or MS/MS detector surfaces.

7.1.3 Compounds extracted from the sample matrix to which the detector will respond.

7.2 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, deactivated syringes, SPE sample transfer lines, etc. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by running method blanks. Reagents of sufficient quality are used to reduce this possibility and purchased in accordance with ADMIN-013.

7.3 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. Typical cleaning solvents used include water, methanol, and methanolic ammonium hydroxide. This should be followed by detergent washing with hot water, and rinsing with tap water, followed by organic-free reagent water. Drain the glassware and dry it in an oven at 130°C for several hours, or rinse with methanolic ammonium hydroxide (1%), IPA, and methanol and drain. Store dry glassware in a clean environment.



- 7.3.1 The residual PFAS content of disposable plasticware and filters must be verified by batch/lot number and may be used without cleaning if PFAS levels are below the Method Detection Limit (MDL).
- 7.4 Aqueous samples should not come in contact with any glass containers or pipettes as PFAS analytes can potentially adsorb to glass surfaces. Standards dissolved in organic solvent may be purchased in glass ampoules. These standards in organic solvent are acceptable and subsequent transfers may be performed using glass syringes and pipets. Following extraction, the eluate must be collected in a polypropylene tube prior to concentration to dryness. Concentration to dryness in glass tubes may cause poor recovery.
- 7.5 All parts of the SPE manifold must be cleaned between samples with methanolic ammonium hydroxide (1%) and air dried prior to use. Sonication with methanolic ammonium hydroxide (1%) may be used for components that will fit in an ultrasonic bath. Smaller parts, like the needles, adapters, reservoirs, and stopcocks associated with the manifold, require rinsing with tap water prior to manual cleaning or sonicating with methanolic ammonium hydroxide (1%) and air drying. When in use, after loading the samples but prior to elution procedures, the chamber must be rinsed with methanolic ammonium hydroxide (1%).
- 7.6 Solid phase extraction cartridges may be a source of interferences. The analysis of LRBs provides important information regarding the presence or absence of such interferences. Each brand and lot of SPE devices must be monitored to ensure that contamination does not preclude analyte identification and quantitation. SPE cartridges should be sealed while in storage to prevent ambient contamination of the SPE sorbent.
- 7.7 Each of the three telomer sulfonates in the analyte list (4:2FTS, 6:2FTS, and 8:2FTS) are referenced to their $^{13}\text{C}_2$ isotope dilution analogue. The mass difference between the telomer sulfonates and the isotope dilution analogues is 2 mass units. The single sulfur atom in each of the unlabeled molecules has a naturally occurring M+2 isotope (^{34}S) at 4.25%. Thus, the precursor ions of the $^{13}\text{C}_2$ isotopically labeled analogues and the naturally occurring ^{34}S analogues present in the native analytes have the same nominal masses. The product ions of the telomer sulfonate isotope dilution analogues listed in Table 6 would contain a small contribution from the ^{34}S analogue of the native telomer sulfonates. At the concentrations used in this study, the contribution of the ^{34}S analogue to the isotope dilution analogue was not greater than 2.7%. Alternate product ions may be used if there is sufficient abundance.
- 7.8 LC system components, as well as the mobile phase constituents, may contain many of the analytes in this method. Thus, these PFAS will build up on the head of the LC column during mobile phase equilibration. To minimize the background PFAS peaks and to keep baseline levels constant, the time the LC column sits at initial conditions must be kept constant and as short as possible (while ensuring reproducible retention times). In addition, priming the mobile phase and flushing the column with at least 90% methanol before initiating a sequence may reduce background contamination.
- 7.9 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled.



7.9.1 Humic and fulvic material may be co-extracted during SPE and high levels may cause enhancement or suppression in the electrospray ionization source. Under the LC conditions used during method development, matrix effects due to co-extracted organic material enhanced the ionization of 4:2 FTS appreciably. Total organic carbon (TOC) is a good indicator of humic content of the sample.

7.9.2 The most frequently encountered interferences are fluoropolymers; however, bile salts (e.g., Taurodeoxycholic Acid [TDCA]) can interfere in the chromatography. For this reason, analysis of a standard containing TDCA is required as part of establishing the initial chromatographic conditions.

7.10 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is rinsed automatically with solvent between sample injections. Whenever an unusually concentrated sample (10 times the upper limit of the curve) is encountered, it should be followed by the analysis of solvent to check for cross-contamination.

8.0 Safety

8.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

8.1.1 PFOA has been described as likely to be carcinogenic to humans. Pure standards should be handled by trained personnel, with suitable protection to skin and eyes, and care should be taken not to breathe the vapors or ingest the materials.

8.1.2 It is recommended that the laboratory purchase dilute standard solutions of the analytes in this method. However, if primary solutions are prepared, they must be prepared in a hood, following universal safety measures.

8.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Safety Data Sheets (SDS) should be made available to all personnel involved in the chemical analysis. These are stored in the common areas of the labs.

8.3 Refer to the AEL Chemical Hygiene Plan and Safety Manual for safety precautions and for the Hygiene Plan and Emergency Response Plan.

8.4 See Standard Methods, 22nd and Online Editions, Section 1090 Laboratory Occupational Health and Safety.

9.0 Equipment and Supplies

9.1 Sample equipment, for discrete or composite sampling. Only HDPE tubing must be used.

9.2 HDPE bottles with an unlined polyethylene screw cap (for example, 250 mL bottles, Environmental Express, Cat. No. BPC1410 or equivalent).



9.3 Liquid Chromatograph (LC)- Mass Spectrometer (MS)/Mass Spectrometer (MS) system:

Note: For all labs, see the Quality Manual, Section 7, for the current listing by room location and letter designation for each piece of major equipment (by make, model, and serial number) and a full inventory of all major pieces of equipment in each lab.

- 9.3.1 Agilent 1260 Liquid Chromatograph coupled with an Agilent Model 6470 QQQ – An analytical system complete with liquid chromatograph; suitable for sample introduction and all required accessories, including pump, detector, column supplies, nebulizer, and collision cell. The 1260 LC provides consistent sample injection volumes and is capable of performing binary linear gradients at a constant flow rate. All PTFE transfer lines, or components were replaced with PEEK or polypropylene components and/or bypassed by the manufacturer upon instrument installation.
 - 9.3.1.1 Agilent 1260 Infinity II Binary Pump (Part No. G7112BR or equivalent)
 - 9.3.1.2 Agilent 1260 Infinity II Multi-Sampler (Part No. G7167AR or equivalent)
 - 9.3.1.3 Agilent 1260 Infinity II Multi-Column Thermostat (Part No. G7116A or equivalent)
- 9.3.2 Delay or Trap Column – GL Sciences, PFAS Delay column, 3.0X30mm (Part No. 5020-90005 or equivalent). The delay column is placed in the mobile phase flow path immediately before the injection valve. This direct connect column reduces the co-elution of PFAS originating from sources prior to the sample loop from the PFAS injected in the sample.
- 9.3.3 Analytical or Chromatographic Column – GL Sciences, InertSustain AQ-C18, 100 x 2.1mm, 1.9µm (Part No. 5020-89939, or equivalent).
- 9.3.4 Detector - Electrospray Ionization Tandem Mass Spectrometer (ESI-MS/MS)
 - 9.3.4.1 Agilent
 - 9.3.4.2 The mass spectrometer must be capable of electrospray ionization in the negative ion mode. The system must be capable of performing MS/MS to produce unique product ions for the method analytes within specified retention time segments. A minimum of 10 scans across the chromatographic peak is needed to ensure adequate precision.
 - 9.3.4.3 Rough Pump

9.4 Data System –

- 9.4.1 Agilent Mass Hunter Workstation LC/MS Data Acquisition for 6400 Series Quadrupole Version 10.0 SR1 Build 10.0.142 or equivalent.
- 9.4.2 An interfaced data system is required to acquire, store, and output MS data. The computer software must have the capability of processing stored data by recognizing a chromatographic peak within a given retention time window. The software must



allow integration of the abundance of any specific ion between specified time or scan number limits. The software must be able to construct a linear regression or quadratic regression calibration curve and calculate analyte concentrations using the internal standard technique.

9.5 Graduated Cylinders, Class A – Appropriate sizes (25mL, 100mL and 1000mL)

9.6 Polypropylene Graduated Cylinders – Appropriate sizes (1000mL, 100mL, 50mL, 25mL)

9.7 Centrifuge Tubes – Conical polypropylene centrifuge tubes (15 mL or 50mL) with polypropylene screw caps for storing standard solutions and for collection of the eluate during the extraction procedure (VWR. Part No. 21008-656 & 10025-698 or equivalent).

9.8 Auto-sampler vials – Polypropylene autosampler vials (VWR, Part No. 82030-982) with polypropylene caps (VWR, Part No. 89239-430 or equivalent).

Note: Polypropylene vials and caps are necessary to prevent contamination of the sample from PTFE coated septa. However, polypropylene caps do not reseal, creating the potential for evaporation to occur after injection. Multiple injections from the same vial are not permissible unless the cap is replaced immediately after injection.

9.9 Polyethylene Disposable Pipettes – 7.7mL capacity (VWR Part No. 14670-130 or equivalent).

9.10 Repeater pipette.

9.11 Micro-Syringes – 10 μ L, 25 μ L, 50 μ L, 100 μ L, 250 μ L, 500 μ L, and 1000 μ L.

9.12 Balance –

9.12.1 Top-loading, capable of weighing to 0.01g. For reagent (mobile phase etc.) preparation and the gravimetric determination of sample volume. (Ohaus, Model SPX2202, or equivalent).

9.12.2 Analytical, capable of weighing to 0.0001g. For percent solids determination. (VWR-220B2T, Mettler Toledo XS205, or equivalent).

9.13 Desiccator (Bel-Art Products, Secador Cat# 4207411116, or equivalent).

9.14 Glass fiber filter (Environmental Express, F93447MM-X, or equivalent).

9.15 Disposable 10mL syringe (Agilent, P/N 9301-6474, or equivalent).

9.16 Disposable syringe filter, 25-mm, 0.2 μ M Nylon Membrane (Agilent, P/N 5190-5110, or equivalent).

Note: EPA 1633 calls for an analytical balance capable of weighing to 0.0001g; that level of accuracy is required for weighing neat standards for preparation of calibration solutions. The balance use at AEL is only for reagent (mobile phase etc.) preparation and the gravimetric determination of sample volume.

9.17 Polystyrene Weigh Boats (VWR Part No. 76312-328 or equivalent).



9.18 Solid Phase Extraction (SPE) Apparatus

9.18.1 SPE Cartridges – For water/solids extraction: SPE cartridges containing weak anion exchange, particle size approximately 33 μm . The SPE sorbent must have a pKa above 8 so that it remains positively charged during extraction. SPE cartridges containing 150 mg sorbent (Phenomenex Cat. No. 8B-S038-SCH or equivalent). Use of 500 mg cartridges is allowed but the lab must perform demonstration of capability study to demonstrate the bed size does not negatively affect extraction and elution. The cartridges are for single use only and may not be reconditioned for subsequent analyses.

9.18.2 Vacuum Extraction Manifold – Equipped with flow and vacuum control (Restek Resprep QR-12 (Part No. 28298-VM) or equivalent system). Automated devices designed for use with SPE cartridges may be used; however, all extraction and elution steps must be the same as in the manual procedure. Care must be taken with automated SPE systems to ensure that Teflon tubing and other PTFE components commonly used in these systems, do not contribute to unacceptable analyte concentrations in LRBs.

9.18.3 Sample Delivery System – Use of large volume sampling lines, constructed with polyethylene tubing, are recommended, but not mandatory. Large volume sample transfer lines, constructed with PTFE tubing, are commercially available for standard extraction manifolds (Restek Cat. No. 26250 or equivalent). The PTFE tubing can be replaced with 1/8" o.d. x 1/16" i.d. polyethylene tubing (Freelin-Wade (McMinnville, Oregon) LLDPE or equivalent) cut to an appropriate length. This prevents potential contamination from PTFE transfer lines. Other types of non-PTFE tubing may be used provided it meets the LRB and LFB QC requirements. PTFE tubing may be used, but an LRB must be run on each individual transfer line and the QC requirements must be met. Empty 60mL SPE tubes are used during the SPE portion of the soil prep as the extract will have a 50mL volume, and transfer lines are not necessary.

9.18.4 Pump – Sufficient capacity to maintain a vacuum of approximately 15 to 20 inches of mercury for extraction cartridges.

9.19 Shaker table – for extracting the soil samples during the pre-extraction steps.

9.20 Vortex apparatus.

9.21 Centrifuge apparatus capable of centrifuging 50mL centrifuge tubes.

9.22 Extract Concentration System – Extracts are concentrated by evaporation with high-purity nitrogen using a water bath set no higher than 60°C (Organomation Associates, Meyer N-Evap, Model 11155, or equivalent).

9.23 pH paper – Used to verify the pH of the phosphate buffer and to measure the pH of the aqueous sample prior to anion exchange SPE. ColorpHast® pH Strips, Narrow, one square with single indicator on each strip, 6.5-10 pH range (VWR Part No. AA35227-LQ or equivalent).



10.0 Reagents and Standards

Note: Although sources of the reagents and standards noted in this SOP may be provided, they may also change based on availability, quality, and cost. The use of a different source or concentration is acceptable without modification of the procedures, provided the products are equivalent. Reagent grade or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If other grades are used, the reagent must be demonstrated to be free of analytes and interferences and all requirements of the IDC must be met when using these reagents.

Note: All reagents and standards must be labeled with their unique ID, the name of the material, the concentration, the date prepared, and the expiration date.

Note: Stock standard solutions are ordered from NELAC (TNI) approved vendors. Stock standards are received from the vendor in sealed amber ampoules. Once opened, store the stock standard solutions in polypropylene vials with polypropylene screw tops. All stock standards are stored at 0°C to 6°C and protected from light (unless otherwise instructed by the manufacturer). Check stock standards frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards. Opened stock standards should be replaced after 6 months or sooner, if comparison with check standards indicates a problem.

Note: Prior to making any laboratory prepared standards, allow the stock standard to warm to room temperature. Store intermediate and working standards in screw-top vials at 0°C to 6°C and protect from light. Working standards should have an expiration date of six months (unless the manufacturer's expiration date is sooner). Allow working standards to warm to room temperature prior to use.

Note: Modifying the solvent composition of the standard or extract by increasing the aqueous content to better focus early eluting compounds on the column is not permitted. A decrease in methanol concentration could lead to lower or imprecise recovery of the more hydrophobic method analytes, while higher methanol concentration could lead to the precipitation of salts in some extracts.

10.1 Reagents and Standards are ordered and tracked internally in accordance with SOPs ADMIN-013 and ADMIN-031.

10.2 The mixing of reagents shall be tracked in a reagent logbook kept in the LC room. The mixing of intermediate and/or working standards shall be tracked in a standard logbook kept in the LC room. Both logbooks will contain the following information:

10.2.1 For parent material:

10.2.1.1 The manufacturer lot number.

10.2.1.2 The manufacturer name.

10.2.1.3 The chemical name and/or chemical description.

10.2.1.4 The expiration date.



10.2.1.5 The AEL receiving lot number.

10.2.2 For any laboratory prepared reagents and/or standards:

10.2.2.1 The recipe (the amount of parent material used and how the standard was mixed) is included in the logbook.

10.2.2.2 The creation date.

10.2.2.3 The expiration date.

10.2.2.4 The standard concentration.

10.3 Reagents:

10.3.1 Reference Matrices:

10.3.1.1 Organic-free reagent water — all references to water in this method refer to organic-free reagent water from the de-ionized water tap, unless ultrapure is needed then the ELGA 18mO system is utilized. Siemens Company in accordance with Admin-032 maintains our present de-ionized water system. Organic-free reagent water is defined as purified water which does not contain any measurable quantities of any method analytes or interfering compounds greater than one-third of the MRL for each method analyte. It may be necessary to flush the water purification unit to rinse out any build-up of PFAS in the system prior to collection of reagent water.

10.3.1.2 Ottawa sand – Restek, part# 26137, 20/30 mesh, 5 KG.

10.3.2 Reagent grade or HPLC grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.3.3 Methanol, CH₃OH, CASRN 67-56-1 – HPLC grade or better in accordance with ADMIN-013 (VWR, Part No. BDH85800.100E or equivalent). LC/MS grade required for mobile phase. Purge and Trap grade used for methanolic ammonium hydroxide solutions.

10.3.4 Ammonium Acetate, NH₄C₂H₃O₂, CASRN 631-61-8, molecular weight equals 77.08 g/mole – HPLC grade or better in accordance with ADMIN-013. **LC-MS grade is required for mobile phase.**

10.3.4.1 Store at 2-8°C.

10.3.4.2 Replace two years after opening date.



- 10.3.5 Concentrated Ammonium Hydroxide Reagent, NH_4OH , CASRN 1336-21-6, – approximately 56.6% in water as ammonium hydroxide (w/w), approximately 29% in water as ammonia, approximately 14.8 N (VWR, Part No. 470300-212, Certified ACS Plus grade, or equivalent).
- 10.3.6 Carbon – Supelclean ENVI-Carb SPE Bulk, part#57210-U.
- 10.3.7 Formic acid – 96%, or greater. Store at room temperature.
- 10.3.8 Salinized glass wool – Supelco, part# 20411, or equivalent. Rinsed twice with methanol prior to use.
- 10.3.9 LC/MS grade Acetic Acid – used to add to sample extracts.
- 10.3.10 Nitrogen – Ultra-high-purity grade. Used as a nebulizer gas in the ESI interface, as collision gas, and to concentrate sample extracts.
- 10.3.11 Laboratory Prepared Reagents:
- 10.3.11.1 20 mM LC-MS grade Ammonium Acetate, Chromatographic mobile phase – To prepare 0.5 L, add 0.77 g ammonium acetate to 0.5 L of ultrapure reagent water. **This solution is volatile and must be replaced at least once per week.** More frequent replacement may be necessary if unexplained losses in sensitivity or retention time shifts are encountered.
- 10.3.11.2 0.3% ammonium hydroxide in methanol – used for soil extraction. Dilute 1.0mL of concentrated ammonium hydroxide up to 100mL of P&T methanol. **Method 1633 gives a 1-month expiration date; however, it is recommended that this solution should be made fresh on the day of extraction.**
- 10.3.11.3 1% ammonium hydroxide in methanol – Used for pre-conditioning of SPE cartridges and for elution of SPE cartridges during water and solid prep. Dilute 3.3 mL of concentrated ammonium hydroxide up to 100 mL P&T methanol. **Method 1633 gives a 1-month expiration date; however, it is recommended that this solution should be made fresh on the day of extraction.**
- 10.3.11.4 0.3M formic acid – aqueous, dissolve 13.8 grams of formic acid into 1L of ultrapure reagent water. Replace after 2 years. Store at room temperature.
- 10.3.11.5 0.1M formic acid – aqueous, dissolve 4.6 grams into 1L of ultrapure reagent water. Replace after 2 years. Store at room temperature.
- 10.3.11.6 50% aqueous formic acid – mix 50mL formic acid and 50mL of reagent water. Replace after 2 years. A smaller amount may be made to conserve reagents and reduce waste, (ie. 5ml formic acid and 5 ml reagent water).



- 10.3.11.7 3% aqueous ammonium hydroxide – mix 10mL of ammonium hydroxide (30%; 10.3.5) and 90mL of reagent water. A smaller amount may be made to conserve reagents and reduce waste, ie. 1ml of ammonium hydroxide and 9 ml reagent water. Replace after 3 months.
- 10.3.11.8 1:1 0.1M formic acid/methanol solution – mix equal amounts of 0.1M formic acid (10.3.12.6) and methanol. Replace after 1 year. Store at room temperature.
- 10.3.11.9 1633 IB/Standard reagent – used to make instrument blanks, for standard (ICAL/CCV/ICV) make-up and if sample dilutions are needed. Mix 3.3mL of ammonium hydroxide (10.3.5), 1.7 mL of ultrapure reagent water and 0.625mL of LC/MS acetic acid up to 100mL of LC/MS grade methanol. Store at room temperature. Replace after 1 month.
- 10.4 Stock standards - Stock solutions purchased as certified solutions. Opened stock standards must be replaced after 1 year or sooner; if comparison with check standards indicates a problem (common problems occur from degradation or evaporation of the standard). Certificates of analysis are stored in the SVOC lab in accordance with Quality Manual 5.0. Store purchased standards according to manufacturer specifications. All purchased stock standards solutions must be replaced after reaching the manufacturer's expiration date assigned to the standard.

Note: Alternative standards from the ones listed below may be used, provided they are from a certified vendor.

Note: Fluorinated carboxylic acids will esterify in anhydrous acidic methanol. To prevent esterification, standards must be stored under basic conditions. If base is not already present, this may be accomplished by the addition of sodium hydroxide (approximately 4 mole equivalents) when standards are diluted in methanol. When calculating molarity for solutions containing multiple PFAS, the molecular weight can be estimated as 250 atomic mass units (amu). It is necessary to include sodium hydroxide in solutions of both isotopically labeled and native analytes. The amount of sodium hydroxide needed may be calculated using the equation in Section 15. The standards purchased from Wellington and Absolute already contain the appropriate amount of sodium hydroxide to prevent esterification.

Note: When a compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Sorption of PFAS analytes in methanol solution to glass surfaces after prolonged storage has not been evaluated. PFAS analyte and isotopically labeled analogues commercially purchased in glass ampoules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be stored in polypropylene containers. Standards for sample fortification generally should be prepared in the smallest volume that can be accurately measured to minimize the addition of excess organic solvent to aqueous samples.

Note: When not being used, store standard solutions in the dark at less than 4 °C unless the vendor recommends otherwise in screw-capped vials with foiled-lined caps.



- 10.4.1 Tuning Mix – ESI-L Low Concentration Tuning Mix (100mL, Agilent part # G1969-85000), or equivalent. Use as received; no prep required.
- 10.4.2 Bile Salts
 - 10.4.2.1 TDCA – Taurodeoxycholic acid, sodium salt – EMD Millipore part# 580221-5GM, or equivalent.
 - 10.4.2.2 TUDCA – Tauroursodeoxycholic acid, sodium salt – EMD Millipore part# 580549-1GM, or equivalent.
 - 10.4.2.3 TCDCA – Sodium taurochenodeoxycholate, EMD Millipore part#T6260-100MG, or equivalent.
- 10.4.3 Mass-labelled PFAS Injection Standard Solution/Mixture (Non-extracted Internal standard - NIS) – Wellington part# MPFAC-HIF-IS, 1.2 mL, various concentrations, or equivalent.
- 10.4.4 Mass-labelled PFAS Extraction Standard Solution/Mixture (Extracted Internal Standards -EIS) – Wellington part# MPFAC-HIF-ES, 1.2 mL, various concentrations, or equivalent.
- 10.4.5 Analyte Standard Materials
 - 10.4.5.1 Technical Grade Standards (for evaluation of the retention time of branched isomers). At the time of this SOP revision only 4 technical grade standards were found by the lab:
 - 10.4.5.1.1 PFOA (Wellington Part No. T-PFOA, or equivalent)
 - 10.4.5.1.2 N-Et-FOSA - Sigma-aldrich, CDS010729-250MG, neat, or equivalent.
 - 10.4.5.1.3 N-Me-FOSE – Toronto Research Chemicals – M327345, neat, or equivalent.
 - 10.4.5.1.4 N-Et-FOSE – Toronto Research Chemicals – E917650, neat, or equivalent.

Note: This method measures all forms of the analytes as anions while the identity of the counterion is inconsequential. Analytes may be commercially available as neat materials or as certified stock standards as their corresponding ammonium, sodium, or potassium salts. These salts are acceptable standards provided the measured mass, or concentration, is corrected for the salt content. The equation for this correction is provided in Section 15. The correction for the salt content is calculated on the vendor's Certificate of Analysis; this predetermined corrected value is used to quantify the target analytes in salt form.

10.4.5.2 Primary Stock Standards

- 10.4.5.2.1 Native 30 PFAS mix; 30 analytes; 1000ng/mL (Wellington Part No. PFAC30PAR, or equivalent).



- 10.4.5.2.2 Native MXG PFAS mix; 2 analytes, 2000ng/mL (Wellington Part No. PFAC-MXG, or equivalent).
- 10.4.5.2.3 L-PFDoS solution; 1 analyte, 50ug/mL (Wellington part No. L-PFDoS, or equivalent)
- 10.4.5.2.4 Native N-Me/EtFOSA and N-Me/Et FOSE Solution/Mixture; 4 analytes, 1.0ug/mL/10ug/mL (Wellington part No. PFAC-MXJ, or equivalent)
- 10.4.5.2.5 Native X:3 Fluorotelomer Carboxylic Acid Solution/Mixture, 3 analytes, 4.0ug/mL/20ug/mL (Wellington part No. PFAC-MXJ, or equivalent)

10.4.5.3 Secondary Stock Standards

- 10.4.5.3.1 EPA method 533 analytes; 25 analytes; 500ng/mL (Wellington Part No. EPA-533PAR, or equivalent)
- 10.4.5.3.2 Native PFAS Solution/Mixture; 25 analytes, various concentrations (Wellington Part No. PFAC-MXH, or equivalent)
- 10.4.5.3.3 N-Et-FOSE stock, 50ug/mL (Wellington part No. N-EtFOSE-M, or equivalent)
- 10.4.5.3.4 N-MeFOSE stock, 50ug/mL (Wellington part No. N-MeFOSE-M, or equivalent)
- 10.4.5.3.5 N-EtFOSA stock, 50ug/mL (Wellington part No. N-EtFOSA-M, or equivalent)
- 10.4.5.3.6 N-MeFOSA stock, 50ug/mL (Wellington part No. N-MeFOSA-M, or equivalent)
- 10.4.5.3.7 3:3FTCA stock, 50ug/mL (Wellington part No. FPrPA, or equivalent)
- 10.4.5.3.8 5:3FTCA stock, 50ug/mL (Wellington part No. FPePA, or equivalent)
- 10.4.5.3.9 7:3FTCA stock, 50ug/mL (Wellington part No. FHpPA, or equivalent)

10.5 Laboratory Prepared Standards

- 10.5.1 All laboratory prepared standard solutions should be prepared in a hood and must be replaced after 1 year or sooner if routine QC indicates a problem or the method requires a shorter expiration date. An assigned expiration date of a lab prepared standard cannot exceed the manufacturer's expiration date for any component used in



the standard formulation.

10.5.2 Bile salts: 1) In to a tared 10.0 mL volumetric flask, weigh approximately 0.01 grams of the bile salt (either TDCA, TUDCA and/or TCDCA). Add LC/MS methanol to volume, thus making a standard approximately 1.0mg/mL. Transfer and store in a 12 mL glass vial. 2) Dilute 100uL of the stock solution (1.0mg/mL) up to 1.0mL of LC/MS methanol to make a 100ug/mL intermediate solution. Make and store in a 2.0mL glass autosampler vial. 3) Dilute 10uL of all 3 intermediate solutions (100ug/mL) up to 1.0mL of the 1633 Blank MeOH solution (10.3.12.9) thus making a 1.0ug/mL working standard containing all 3 bile salts.

10.5.3 1633 Non-extracted Internal standard Working Solution (1633NIS WS)

10.5.3.1 Prepare the non-extracted internal standard working solution in methanol by diluting 100uL of IS stock (10.4.3) up to 1.0mL of LC/MS methanol. During collection of method performance data, 1.0mL of the final extracts were fortified with 10 μ L of the 1633NIS WS. 10uL of the 1633NIS WS are added to the 1.0mL ICAL and ICV standard solutions.

10.5.4 1633 Mass-labeled Extracted Internal Standard solution (1633EIS WS)

10.5.4.1 Prepare the isotope dilution analogue EIS by diluting 100uL of the 1633 EIS stock (10.4.4) in 1.0mL of LC/MS methanol. 50uL of the EIS is added to each batch QC item and sample, which equates to 10uL per 1.0mL. Thus 10uL of the EIS is added to each 1.0mL ICAL and ICV solution.

10.5.4.1.1 Note that the concentrations of sulfonates in the isotope dilution analogue PDS is based on the weight of the salt. It is not necessary to account for difference in the formula weight of the salt compared to the free acid for sample quantitation.

10.5.5 Analyte PDS – Primary and Secondary source

10.5.5.1 The primary analyte stock solutions listed in 10.4.5.2 are used to prepare High intermediate and a Low intermediate. Before the intermediates can be made the stock solution for L-PFDoS must be diluted to a working level. These intermediates are used to prepare the calibration standards and are used for batch spiking:

10.5.5.2 L-PFDoS Working Intermediate solution (PFDoS INT) – 100uL of 10.4.5.2.3 is brought up to 1.0mL of LC/MS methanol. This yields a working solution of 5000 ng/mL.

10.5.5.2.1 Select nominal analyte concentrations for the intermediates such that between 5 and 100 μ L of the PDS is used to fortify samples and prepare standard solutions. The analyte INTs are prepared at 2 different concentrations:

10.5.5.2.1.1 100ng/mL (1633 High Int): Prepared by diluting 100uL of PFAC30PAR, 50uL of PFAC-MXG, 20uL of PFDoS



INT, 100uL of PFAC-MXI and 100uL of PFAC-MXJ into 1.0mL of LC/MS methanol.

10.5.5.2.1.2 10ng/mL (1633 Low Int): Prepared by diluting 100uL of the 100ng/mL 1633 High Int (Section 10.5.5.2.1.1) into 1.0mL of LC/MS methanol.

10.5.5.2.2 These INTs are used to spike batch QC (LCS/LCSD/MS/MSD) to allow for all analytes to be contained within the spikes. The user may modify the concentrations of the individual analytes based on the confirmed MRLs and the desired monitoring range.

10.5.5.2.3 The INTs are stored cold; warm the vials to room temperature and vortex prior to use.

10.5.5.3 The Secondary source stocks are used to make the Initial Calibration Verification (ICVs). Due to limited availability of source standards; several solutions are used to make ICV intermediates, which are then combined to make a working ICV standard. The 50ug/mL stocks must be diluted to a lower concentration prior to making the ICV Intermediate:

10.5.5.3.1 ICV Intermediates:

10.5.5.3.1.1 533 ICV INT: Prepared by diluting 50uL of the EPA-533PAR (Section 10.4.5.3.1) into 100% methanol and adding sodium hydroxide if not already present to prevent esterification. The final volume of the standard is 1.0mL.

10.5.5.3.1.2 1633 MXH INT: 100uL of the MXH stock (10.4.5.3.2) brought up to 1.0mL of LC/MS grade methanol.

10.5.5.3.1.3 1633 ICV1 INT: 20uL of N-Me-FOSA stock (10.4.5.3.6) + 20uL of N-Et-FOSA stock (10.4.5.3.5) + 200uL of N-Me-FOSE stock (10.4.5.3.4) + 200uL of N-Et-FOSE stock (10.4.5.3.3) up to 1.0mL of LC/MS grade methanol.

10.5.5.3.1.4 1633 ICV2 INT: 80uL of 3:3FTCA (FPrPA) (10.4.5.3.7) up to 1.0mL of LC/MS grade methanol.

10.5.5.3.1.5 1633 ICV3 INT: 400uL of 5:3FTCA (FPePA) (10.4.5.3.8) up to 1.0mL of LC/MS grade methanol.

10.5.5.3.1.6 1633 ICV4 INT: 400uL of 7:3FTCA (FHpPA) (10.4.5.3.9) up to 1.0mL of LC/MS methanol.



10.5.5.3.2 1633 ICV INT Working Stock

10.5.5.3.2.1 Combine 100uL of ICV1 INT (10.5.5.3.1.3) + 100uL of ICV2 INT (10.5.5.3.1.4) + 100uL of ICV3 INT (10.5.5.3.1.5) + 100uL of ICV4 INT (10.5.5.3.1.6) up to 1.0mL of LC/MS grade methanol.

10.6 Initial Calibration (ICAL) and Initial Calibration Verification (ICV) Standards

PFAS Calibration Standards – Prepared as described below in 1633 Blank MeOH (10.3.12.7).

| ICAL (ng/mL) | Amount of PFAS Int1 to use in uL | Amount of PFAS Int2 to use in uL | Final Volume (mL) | Amount of NIS WS to Use (uL)* | Amount of EIS Spike to Use (uL) |
|--------------|----------------------------------|----------------------------------|-------------------|-------------------------------|---------------------------------|
| ICAL1 0.1 | - | 10 | 1 | 10 | 10 |
| ICAL2 0.2 | - | 20 | 1 | 10 | 10 |
| ICAL3 0.5 | - | 50 | 1 | 10 | 10 |
| ICAL4 0.8 | - | 75 | 1 | 10 | 10 |
| ICAL5 1.0 | 10 | - | 1 | 10 | 10 |
| ICAL6 1.5 | 15 | - | 1 | 10 | 10 |
| ICAL7 2.0 | 20 | - | 1 | 10 | 10 |
| ICAL8 5.0 | 50 | - | 1 | 10 | 10 |
| ICAL9 10.0 | 100 | - | 1 | 10 | 10 |

*NIS is added to the 1.0mL final volume

1633 Initial Calibration Verification (ICV) - combined

| ICV (ng/mL) | Amount of 1) ICV INT WS + 2) MXH INT + 3) 533 ICV INT (uL) | Final Volume (mL) | Amount of NIS WS to Use (uL)* | Amount of EIS Spike to Use (uL) |
|-------------|--|-------------------|-------------------------------|---------------------------------|
| ICVC | 1)20uL + 2)10uL + 3)10uL | 1 | 10 | 10 |

*NIS is added to the 1.0mL final volume

Note: During the multi-laboratory validation study, laboratories reported that NMeFOSA was an impurity in the branched isomer qualitative standard for NMeFOSE and NEtFOSA was an impurity in the branched isomer qualitative standard for NEtFOSE supplied for the study. Those impurities did not preclude the use of these standards, but laboratories should be aware of the possibility.

11.0 Sample collection, preservation, shipment, and storage

11.1 See AEL Admin-005 and Admin-023.

11.2 See AEL Quality Manual Section 6.0 for sample acceptance policy.

11.3 See FDEP SOP FS1000 for preservation requirements, shipping conditions, and holding time requirements.



- 11.4 Samples must be collected in HDPE containers fitted with unlined polyethylene screw-caps, Discard sample bottles after a single use. The bottle volume should approximate the volume of the sample. Subsampling from a single bottle is not permitted except as described in Section 14.4.3.5.7.
- 11.5 Samples must be shipped on ice. Samples are valid if any ice remains in the cooler when it is received at the laboratory or bottles are received within 2 days of collection and are confirmed to be between 0 - 6 °C. Once at the laboratory, samples should be stored at or below -20 °C until extraction for both waters and solids.
- 11.6 Aqueous samples (including leachates) should be analyzed as soon as possible; however, samples may be held in the laboratory:
- 11.6.1 For up to 90 days from collection, when stored at ≤ -20 °C and protected from the light, or
- 11.6.2 For up to 28 days when stored at 0 - 6 °C and protected from the light, with the caveat that issues were observed with certain perfluorooctane sulfonamide ethanols and perfluorooctane sulfonamidoacetic acids after 7 days. These issues are more likely to elevate the observed concentrations of other PFAS compounds via the transformation of these precursors if they are present in the sample.
- 11.7 Solid samples (soils and sediments) and tissue samples may be held for up to 90 days, if stored by the laboratory in the dark at either 0 - 6 °C or ≤ -20 °C, with the caveat that samples may need to be extracted as soon as possible if NFDHA is an important analyte.
- 11.8 Store sample extracts in the dark at less than 0 - 4 °C until analyzed. If stored in the dark at less than 0 - 4 °C, sample extracts may be stored for up to 90 days, with the caveat that issues were observed for some ether sulfonates after 28 days. These issues may elevate the observed concentrations of the ether sulfonates in the extract over time. Samples may need to be extracted as soon as possible if NFDHA is an important analyte.
- 12.0 Quality Control
- 12.1 For every analytical sequence (not to exceed 30hours of runtime), the LC-MS/MS system must pass the QC Check Tune for the unit, wide, and widest settings.
- 12.2 There must be an initial calibration of the LC-MS/MS system – see Section 13.
- 12.3 The LC-MS/MS system must meet the calibration verification acceptance criteria – see Section 13.
- 12.4 The Retention Time position shall be set using the midpoint of the ICAL when a calibration curve is performed. On days when the ICAL is not performed, the initial CCV is used.
- 12.5 Initial Demonstration of Capability – each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff is trained or



significant changes in instrumentation are made.

12.5.1 For water and soil IDOC prep will consist of a method blank and four replicates for each analyte spiked with the EIS and the native analytes (see section 14.1.1 for the spike standards and volumes for water IDOCs, and section 14.2.1 for the spike standards and volumes for soil IDOCs).

12.5.2 The average recovery for IDOC analytes shall fall within 80-120% of the expected value and the percent relative standard deviation for the IDOC analytes shall be less than 20%.

Note: Once Table 5 in the EPA Draft 1633 method is revised for the multi-laboratory study and finalized, the laboratory will adopt those specific OPR limits.

12.5.3 The Demonstration of Capability is not considered complete until the analyst has documentation saying that they have read, understood, and agreed to follow the AEL-SOP for this method and the associated EPA and/or Standard Methods on which the AEL-SOP was based.

12.5.4 Initial DOC's must be successfully performed by each analyst in accordance with the Quality Manual and ADMIN-030.

12.6 Method Detection Limit (MDL) - MDLs must be established for all analytes, following the procedures outlined in ADMIN SOP-012, which conforms to EPA CFR 40 Part 136.6 appendix B, updated October 2017. Below is a summary of the steps for performing MDLs. Please refer to the ADMIN SOP-012 for the full procedures.

12.6.1 New MDL Study: Most MDL determinations are for already established analyte, matrix, and instrument combinations. In those cases where a new analyte is to be introduced, an initial MDL study will have to be implemented. Select a spiking level, typically 2 to 10 times the estimated MDL. Process a minimum of seven spiked samples and seven method blank samples through all steps of the method. The samples used for the MDL must be prepared in at least three batches on three separate calendar dates and analyzed on three separate calendar dates.

12.6.2 New Instrument: To bring on a new instrument for an already established method a full set of seven low-level replicates is not needed. Only two spiked samples and two method blank samples prepared and analyzed on different calendar dates are required for the new instrument. The resulting values shall be compared against existing MDLs for validity. If both method blank results are below the existing MDL, then the existing MDL_b is validated. Combine the new spiked sample results to the existing spiked sample results and recalculate the MDLs. If the recalculated MDL_s is within 0.5 to 2.0 times the existing MDL and fewer than 3% of the MB have results above the existing MDL, the existing MDL can be left unchanged and the new instrument is validated.

12.6.3 Existing Instrument, Major Maintenance: Follow the procedures for bringing on a new instrument.



12.6.4 Ongoing Data Collection: During any quarter in which samples are being analyzed, prepare, and analyze a minimum of two spiked samples on each instrument, in separate batches (separate prep batches and separate analytical batches), using the same spiking concentration used with established MDLs. If any analytes are repeatedly not detected in the quarterly spiked sample analyses, or do not meet the qualitative identification criteria of the method then this is an indication that the spiking level is not high enough and should be adjusted upward. Note that it is not necessary to analyze additional method blanks together with the spiked samples; the method blank population should include all of the routine method blanks analyzed with each batch during the course of sample analysis.

12.6.4.1 At least once per year, re-evaluate the spiking level. If more than 5% of the spiked samples do not return positive numerical results that meet all method qualitative identification criteria, then the spiking level must be increased, and the initial MDL re-determined following the procedure for establishing a new MDL.

12.6.5 Ongoing Annual Verification: At least once every thirteen months, re-calculate MDLs and MDL_b from the collected spiked samples and method blank results using the equations in the ADMIN SOP-012. These calculations shall be performed by the QA department along with updating and maintaining all chart and LIMs entry of any MDL changes.

12.7 Calibration Verification: Analyze an ICV to confirm the accuracy of the primary calibration standards.

12.7.1 An ICV must be analyzed during the IDOC and then quarterly thereafter.

12.7.2 For this method, the laboratory is not required to obtain standards from a source independent of the primary calibration standards. Instead, the laboratory should acquire the best available quantitative standards and use these to prepare both the primary calibration standards and the ICV.

12.7.3 The ICV must be an independent dilution beginning with the common starting materials.

12.7.4 The acceptance criterion for the ICV is 70–130% of the true value. If the accuracy for any analyte fails the recovery criterion, prepare fresh standard dilutions, and repeat the Calibration Verification.

12.8 On Going QC requirements

12.8.1 The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank (MB), a matrix spike (MS), a duplicate (MSD, DUP, or LCSD), a laboratory control sample (LCS), a low-level laboratory control sample (LLCS) and the addition of isotope dilution analogues to each field sample and QC sample.



- 12.8.1.1 Each sample, MB, LCS, LCSD, LLCS, MS, and MSD must be spiked with the isotope dilution analogues spike.
- 12.8.1.2 Recovery of isotopically labeled compounds from sample matrices must be assessed and records maintained.
- 12.8.2 Every analytical sequence (not to exceed 30 hours of runtime), prior to sample analysis, the laboratory must run a CheckTune. See Section 10.4.1 for the identification of the Tuning solution. If any of the masses do not Pass criteria for Wide or Widest, then the analyst may choose to manually tune to bring those masses back in acceptance criteria; otherwise, a new Autotune must be ran. If any of the masses do not Pass for Unit, then a new Autotune must be analyzed.
- 12.8.3 Every analytical sequence, prior to sample analysis, after the high standard of the ICAL, after every CCV and after any field sample with an analyte detection above the range of the calibration curve (if observed in time to edit/update the analytical sequence) the laboratory must analyze an Instrument Blank (IB).
- 12.8.3.1 The IB contains only the isotope dilution analogues and the non-extracted internal standards in the blank MeOH solution used for calibration standards.
- 12.8.3.2 The IB must not contain any analyte of interest above $\frac{1}{2}$ the MRL/LOQ, or else the instrument system is contaminated. A new IB may be made and analyzed to verify the contamination.
- 12.8.4 Every calibration sequence, and every sequence in which DOD sample are analyzed, a bile salt check standard (Section 10.5.2) must be analyzed. Each bile salt must not elute within 1 minute of all PFOS isomers.
- 12.8.4.1 DOD QSM 5.4 requires the bile salt check be analyzed at the beginning of every analytical sequence regardless of the matrix to be analyzed.
- 12.8.4.2 EPA Draft 1633 requires an initial bile salt interference when establishing chromatographic conditions (regardless of matrix). A daily salt check is not required unless running tissue samples.
- 12.8.5 Daily, at the beginning of the analytical sequence, qualitative identification standards must be analyzed to Confirm the RT of each linear and known branched isomer or isomer group. Quantitative standards containing isomeric mixtures for an analyte are commercially available for PFOS, PFHxS, NMeFOSAA, and NEtFOSAA; the isomer RTs for those quantitative standards can be verified during the instrument calibration and in the CCVs. Qualitative/technical standards are available for PFOA, NEtFOA, NMeFOSE, and NEtFOSE; those standards should be analyzed daily for RT verification.
- 12.8.6 Every analytical shift, prior to sample analysis, after every tenth field sample, and at the end of the analysis sequence, the laboratory must analyze a CCV (see Section 13.3 for acceptance criteria).
- 12.8.7 With every batch of 20 samples (or less) the laboratory must analyze the following:



12.8.7.1MB -1 per batch of 20 or less.

12.8.7.1.1 For non-DOD EPA 1633: Any analyte of interest must not be detected above the MRL, greater than 1/3rd any regulatory compliance limits, or greater than 1/10th the concentration found in any sample within the prep batch – whichever is greater.

12.8.7.1.2 For DOD QSM: Any analyte of interest must not be detected above one-half of the LOQ, or greater than 1/10th the amount found in any sample, or greater than 1/10th the regulatory limit – whichever is greater. The laboratory will strive to have no detections of target analytes at concentrations above the MDL.

12.8.7.2LCS/LLCS – 1 per batch of 20 or less.

12.8.7.2.1 A Laboratory Control Sample (LCS) and Low-Level Laboratory Control Sample (LLCS) are included with each analytical batch and once every twenty samples. The LCS consists of an aliquot of a clean (control) matrix corresponding with the sample matrix and of the same weight or volume. A low-level LCS (LLCS) is required for every batch, and must be spiked at, or below, the MRL. The LCS concentration must be spiked near the mid-point of the calibration curve. A LCS Duplicate must be prepped if there is not enough field sample to prep a MS/MSD.

12.8.7.2.2 For non-DOD EPA 1633: Standard AEL limits of 80-120% have been assigned until the multi-laboratory evaluation is completed by the EPA. The lab may also evaluate LCS against EPA Draft Method Table 5 (see section 24.9 Table); criteria subject to change upon finalization of the draft method

12.8.7.2.3 For DOD QSM: Recovery must be within 40-150%, until in-house limits can be created, or if project limits are not provided. In-house limits cannot be lower than 40%.

12.8.7.3MS/MSD – May be analyzed with every batch of 20 samples or less per matrix. An MS/MSD set may not be required for non-DOD 1633 projects but are required for DOD projects.

12.8.7.3.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate un-spiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an un-spiked field sample. If samples are not expected to contain target analytes, the laboratories should use a matrix spike and matrix spike duplicate pair. A matrix



spike is required for every twenty samples and every extraction batch.

12.8.7.3.2 The MS/MSD should be fortified at a concentration close to the mid-point of the ICAL.

12.8.7.3.3 The spike recoveries must be within 40-150% of the true value if spiked at a level greater than 2X the MRL, if project limits are not provided, or until in-house limits are established.

12.8.7.3.4 The RPD for all analytes shall be less than, or equal to, 30% for MS/MSDs spiked at a level greater than 2X the MRL.

12.8.8 The laboratory must maintain performance records to document the quality of data that is generated.

12.8.8.1 Isotope dilution analogue recoveries: The laboratory must calculate the percent recovery for the isotope dilution analogues spiked before sample/QC prep. The recoveries for the isotopes must be within the limits below, if project specific limits are not provided, or until in-house limits are established.

12.8.8.1.1 For non-DOD EPA 1633: Standard AEL limits of 20-150% have been assigned until the multi-laboratory evaluation is completed by the EPA. The lab may also evaluate the EIS against EPA Draft Method Table 9 (see section 24.8 Table); criteria subject to change upon finalization of the draft method

12.8.8.1.2 For DOD QSM: Recovery must be within 20-150%, until in-house limits can be created, or if project limits are not provided. In-house limits cannot be lower than 20%.

12.8.8.2 If an isotope dilution analogue fails to meet the recovery criterion, evaluate the area of the isotope performance standard to which the analogue is referenced and the recovery of the analogues in the CCCs.

12.8.8.2.1 If necessary, recalibrate and service the LC-MS/MS system. Take corrective action, then analyze the failed extract in a subsequent Analysis Batch.

12.8.8.2.1.1 If the repeat analysis meets the 20–150% recovery criterion, report only data for the reanalyzed extract.

12.8.8.2.1.2 If the repeat analysis fails the recovery criterion after corrective action, extraction of the sample must be repeated provided a sample is available and still within the holding time.



12.8.9 Non-extracted internal standard (NIS) recovery: The laboratory must monitor the recovery of the non-extracted internal standards in all injections of the analysis sequence.

12.8.9.1 For non-DOD EPA 1633: Standard AEL criteria of greater than 30% of the average area measured during the initial calibration have been assigned until the multi-laboratory evaluation is completed by the EPA. The lab may also evaluate area recoveries against EPA Draft 1633 Table 10 (see section 24.7 Table); criteria subject to change upon finalization of the draft method. *(optional): NIS areas in field samples and QC samples should be 50-200% of the mean area of the ICAL.*

12.8.9.2 For DOD QSM: Area recoveries must be greater than 30% of the average of the ICAL.

12.8.9.3 NIS areas corrected for the dilution factor must be greater than 30% of the average area of the calibration standards in diluted samples when additional NIS was not added post dilution of the extract.

12.8.9.4 Random evaporation losses have been observed with the polypropylene caps causing high- biased NIS areas. The cap(s) must be replaced after an injection to minimize solvent loss.

12.8.9.4.1 If a non-extracted internal standard area for a sample does not meet these criteria, reanalyze the extract in a subsequent analytical sequence.

12.8.9.4.2 If the NIS fails to meet the acceptance criteria in the repeat analysis, extraction of the sample must be repeated, provided the sample is still within holding time.

12.8.9.4.3 If a dilution is required, add appropriate amount of NIS based on the dilution performed. The area for the NIS must be greater than 30% of the average of the ICAL for the diluted sample.

12.8.10 The experience of the analyst performing LC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. If the peaks look normal, if the response obtained is comparable to the response from previous calibrations, etc., the instrument is considered still in calibration.

13.0 Calibration and Standardization

Note: See also Section 10 for initial calibration curve standard preparation and curve concentration levels.

Note: See also AEL SOP ADMIN-038 for Calibration, Manual Integrations, and Rules for Chromatography, which outlines the procedures for choosing curve type, calculations performed, integrations allowed, and associated statistics.



13.1 Initial Calibration

13.1.1 The LC/MS operating conditions –

13.1.1.1 The LC operating conditions listed in Table 3 Section 24 serve as guidelines only. Changes to the chromatographic conditions can be made by the analyst in order to improve the speed of analysis, lower the cost of analysis, and/or improve the separation or lower the detection limit as long as the changes meet the initial and continuing calibration criteria and quality assurance criteria listed in this SOP.

Note: The gradient and initial temperatures may require slight adjustments to accommodate the bile salt retention time criteria; such adjustments will not significantly affect the sensitivity or instrument performance (accuracy and precision).

13.1.1.2 Once these operating conditions are established they will be used to calibrate the instrument. All samples, blanks and quality assurance samples must be analyzed with the same operating conditions.

13.1.1.3 MS Optimization: During the development of this method, instrumental parameters were optimized for the precursor and product ions listed in Table 3 Section 24. Product ions other than those listed may be selected; however, the analyst should avoid using ions with lower mass or common ions that may not provide sufficient discrimination between the analytes of interest and co-eluting interferences.

13.1.1.3.1 There have been reports that not all product ions in the linear PFOS are produced in all branched PFOS isomers. (This phenomenon may exist for many of the PFAS.) For this method, the m/z 80 product ion must be used for PFOS and PFHxS to minimize this problem and promote comparability between laboratories.

13.1.1.3.2 Upon instrument installation, the response of the precursor ion ($[M - H]^-$ or $[M - CO_2 - H]^-$) and production ion for each analyte was optimized as per manufacturer's guidance. Analyte concentrations of 5.0 ng/mL were used for this step during method development. The MS parameters (source voltages, source and desolvation temperatures, gas flows, etc.) were varied by the Mass Hunter software until optimal analyte responses were determined. The analytes exhibited different optimal parameters, requiring some compromise on the final operating conditions.

13.1.1.3.3 The peak shape of the early eluting compounds may be improved by increasing the volume of the injection loop or increasing the aqueous content of the initial mobile phase composition.



13.1.1.4 Prepare and analyze the technical-grade standard of PFOA, discussed in Section 10.4.5.1, at a mid- to high- level concentration. Identify the retention times of the branched isomers of PFOA present in the technical-grade PFOA standard. When PFOA is chromatographed on a reversed-phase column, the branched isomers elute prior to the linear isomer. Repeat the procedure in this section for PFH_xS, PFOS, N-Et-FOSA, N-Me-FOSE, and N-Et-FOSE discussed in Section 10.4.5.1, and any other analytes for which technical-grade standards have been acquired. The branched isomer identification checks must be repeated any time chromatographic changes occur that alter analyte retention times.

13.1.1.5 Inject a mid- to high-level calibration standard under optimized LC-MS/MS conditions to obtain the retention times of each method analyte. Divide the chromatogram into segments that contain one or more chromatographic peaks. For maximum sensitivity, minimize the number of MRM transitions that are simultaneously monitored within each segment. Ensure that the retention time window used to collect data for each analyte is of sufficient width to detect earlier eluting branched isomers.

13.1.1.6 Retention Time window position establishment occurs once per ICAL and at the beginning of the analytical sequence. The position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.

13.1.1.6.1 The RT of each analyte and EIS analyte must fall within 0.4 minutes of the predicted retention times from the daily calibration verification or, on days when ICAL is performed, from the midpoint standard of the ICAL.

13.1.1.6.2 Analytes must elute within 0.1 minutes of the associated EIS. This criterion applies only to analyte and labeled analog pairs.

13.1.1.6.3 When establishing the chromatographic conditions, it is important to consider the potential interference of bile salts during analyses. Inject the bile salt interference check standard containing TDCA, TCDCA and TUDCA (section 10.4.2). Ensure that the bile salts do not coelute with any of the target analytes, EIS, or NIS standards. Analytical conditions must be set to allow a separation of at least 1 minute between the bile salts and the retention time window of PFOS. This evaluation is required when establishing the chromatographic conditions for the method, regardless of the sample matrices to be analyzed.

13.1.2 Mass spectrometer tuning and LC performance requirements

13.1.2.1 For every analytical sequence (not to exceed 30 hours of runtime), the LC-MS/MS system must pass the QC Check Tune for the unit, wide, and widest settings prior to any additional analysis.



13.1.2.2 Before injecting calibration standards, the IB should be analyzed at the beginning of a run to confirm that the analytical system is free from contamination. The IB is used to determine the level of noise and baseline rise attributable solely to the analytical system, in the absence of any other analytes or non-analytical related contaminants. The IB is considered to be passing if the results for all target compounds are below the method detection limit (MDL).

13.1.3 Initial Calibration Curve (ICAL) – The isotope dilution/internal standard calibration technique is used for this method. The internal standard approach assumes that variations in instrument sensitivity, amount injected, etc. can be corrected by determining the ratio of the response of the analyte to the response of an internal standard that has been added to the extract.

13.1.3.1 This method has 7 Non-extracted Internal Standards that are used as reference compounds for the internal standard quantitation of the isotope dilution analogues. The suggested isotope performance standard reference for each isotope dilution analogue is listed in Table 1 Section 24. The 24 isotope dilution analogues are used as reference compounds to quantitate the native analyte concentrations. The suggested isotope dilution analogue references for the native analytes are listed in Table 2 Section 24.

13.1.3.2 Nine standards are used to calibrate the instrument for PFAS analysis; a minimum of six standards must be used. The lowest concentration standard (Practical Quantitation Limit (PQL)) is at the MRL; the highest concentration is at the end of the linear range (Upper Quantitation Limit (UQL)). See Section 10 for appropriate dilutions to prepare the calibration curve.

13.1.3.3 Analyze the Calibration Standards following the same GC operating procedures as the client samples (recommended procedures are listed in Table 3 Section 24.4).

13.1.3.4 Calibrate the LC-MS/MS and fit the calibration points with either a linear or quadratic regression. Weighting may be used. **Forcing the calibration curve through the origin is mandatory for this method. Forcing zero allows for a better estimate of the background levels of method analytes.**

13.1.3.4.1 When using an average response factor (RF) calibration (using a calibration factor (CF) fit), for the curve evaluation, a %RSD of <20% for the CF's over the working range verifies acceptance of the calibration curve. A minimum of six calibration points are required for an average RF calibration model.

13.1.3.4.2 When using a linear regression model, a minimum of 6 calibration points are required, and the coefficient of determination (r^2) must be equal to or greater than 0.990 (or the correlation coefficient (r) must be equal to or greater than 0.995).

13.1.3.4.3 When using a quadratic regression model, a minimum of 7 calibration points are required, and the coefficient of



determination (r^2) must be equal to or greater than 0.990 (or the correlation coefficient (r) must be equal to or greater than 0.995).

13.1.3.4.4 The qualifier ion ratios must be updated within the quantitation method using the average of the ICAL levels. This is accomplished by using the MassHunter function “Average Qualifier Ratios” found in the “Method” tab. The method must be in “Edit” mode for this feature to be active. Select the “Cals” button and Select All, then select OK. Once the ratios have been updated to the average and the calibration curves are set up as desired, save the new calibration method using the format “J11A-PFAS-YYMMDD”.

13.1.3.5 Quantitate

13.1.3.5.1 Native Analytes: Quantitate the native analytes using the internal standard calibration technique. The internal standard technique calculates concentration based on the ratio of the peak area of the native analyte to that of the isotope dilution analogue.

13.1.3.5.2 Isotope Dilution Analogues: The isotope dilution analogues are quantified using the internal standard calibration technique. Because isotope dilution analogues are added at a single concentration level to the calibration standards, calibrate for each of these using an average response factor.

13.1.3.5.3 Non-extracted Internal Standards: Because Isotope performance standards are added at a single concentration level to the calibration standards, calibrate for each of these using an average response factor.

13.1.3.6 Evaluate the initial calibration by calculating the concentration of each analyte as an unknown against its regression equation. The MassHunter software automatically calculates these recoveries.

13.1.3.6.1 Instrument sensitivity: Sufficient instrument sensitivity is established if a signal-to-noise ratio $\geq 3:1$ can be achieved when analyzing the lowest concentration standard within the quantitation range that the laboratory includes in its assessment of calibration linearity.

13.1.3.6.2 All calibration points should be within 70– 130% of their true value.

13.1.3.6.3 If these criteria cannot be met, the analyst could have difficulty meeting ongoing QC criteria. In this case, corrective action is recommended such as reanalyzing the calibration standards, restricting the range of calibration, or performing instrument maintenance. If the cause for failure to meet the criteria is due to



contamination or standard degradation, prepare fresh calibration standards and repeat the initial calibration.

13.1.3.7 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

13.1.4 Bile salts interference check

13.1.4.1 The laboratory must analyze a bile salt interference check standard, containing TDCA, TCDCA and TUDCA (section 10.4.2), after the initial calibration as a check on the chromatographic conditions, even if tissue samples are not going to be run. If an interference is present, the chromatographic conditions must be modified to eliminate the interference from the bile salts (e.g., changing the retention time of the bile salts such that they fall outside the retention time window for any of the linear or branched PFOS isomers in the standard by at least one minute), and the initial calibration repeated.

13.2 Initial Calibration Verification (ICV) – Before sample analysis can begin, the integrity of the standard used to prepare the calibration curve must be verified by the analysis of a second source (see Section 10.7).

13.2.1 For the ICV to be valid, the recovery for all analytes must be between 70-130%.

13.3 Continuing Calibration Verification (CCV) – Analyzed prior to sample analysis, after the IB, and ICAL/ICV (if a calibration was performed). A CCV must be run at the beginning of every 24-hour analysis window, after every tenth field sample, and at the end of the analytical sequence.

13.3.1 The beginning CCV for each Analysis Batch must be at, or below, the MRL for each analyte. This CCV verifies instrument sensitivity prior to the analysis of samples and meets the DOD requirement for an Instrument Sensitivity Check (ISC). If standards have been prepared such that all low calibration levels are not in the same solution, it may be necessary to analyze two standards to meet this requirement. Alternate subsequent CCVs between the mid and high calibration levels. The acquisition start time of the final CCC must be within 24 hours of the acquisition start time of the low-level CCC at the beginning of the Analysis Batch. More than one Analysis Batch within a 24-hour period is permitted.

13.3.2 CCV Criteria:

13.3.2.1 Verify Retention Times: The analyst must ensure that each method analyte elutes entirely within the assigned window during each Analysis Sequence.

13.3.2.1.1 Make this observation by viewing the quantitation ion for each analyte in the CCCs analyzed during an Analysis Batch.



13.3.2.1.2 If an analyte peak drifts out of the assigned window, then data for that analyte is invalid in all injections acquired since the last valid CCC.

13.3.2.1.3 In addition, all peaks representing multiple isomers of an analyte must elute entirely within the same MRM window.

13.3.2.2 Non-extracted Internal Standard Area: The absolute area of the quantitation ion for each of the 7 NIS must be greater than 30% of the average area measured during the initial calibration.

13.3.2.3 Isotope Dilution Analogue Recovery: Using the average response factor determined during the initial calibration and the internal standard calibration technique, calculate the percent recovery of each isotope dilution analogue in the CCC. The recovery for each analogue must be within a range of 70–130%.

13.3.2.4 Native Analyte Recovery:

13.3.2.4.1 The concentration of the analytes in CCVs must be within 70–130%.

13.3.2.4.2 If these limits are exceeded, then all data for the failed analytes must be considered invalid. Any field samples analyzed since the last acceptable CCV that are still within holding time must be reanalyzed after an acceptable calibration has been restored.

13.3.2.4.3 If the CCV fails because the calculated concentration is greater than 130% for a method analyte, and field sample extracts show no concentrations above the MRL for that analyte, non-detects may be reported without re-analysis.

14.0 Procedure

Note: Some of the PFAS adsorb to surfaces, including polypropylene. During the elution step of the procedure, sample bottles must be rinsed with the elution solvent whether extractions are performed manually or by automation. For water samples containing particles and for solids % solids need to be determined. Aqueous samples containing up to 50mg solids may be prepped using this procedure. For samples with greater than 50mg, a smaller aliquot may be prepped if necessary; however, subsampling should be avoided whenever possible. The sample should be processed in its entirety and should not be filtered.

14.1 Determination of % Solids

14.1.1 For aqueous samples using the following procedure:

14.1.1.1 Desiccate and weigh a glass-fiber filter to 3 significant figures.

14.1.1.2 Filter 10mL of well-mixed sample through the filter.



14.1.1.3 Dry the filter for a minimum of 12 hours at 110 +/- 5C, and cool in a desiccator.

14.1.1.4 Calculate % solids

$$\% \text{ solids} = \frac{\text{weight of sample aliquot after drying (g)} - \text{weight of filter (g)}}{10 \text{ g}} \times 100$$

14.1.2 Percent solids for solid samples are determined using the laboratory's typical SM2540 procedure.

14.2 Aqueous Liquid Extraction Procedure: A default sample volume of 500mL is used for this SOP. Leachate samples are analyzed using a 100-mL sample volume. Therefore, they must not be included in the same sample preparation batch as aqueous samples analyzed which are analyzed using 500-mL sample volumes.

14.2.1 Sample Preparation:

14.2.1.1 Homogenize the sample by inverting 3 to 4 times, and then allowing to settle. Do not filter.

14.2.1.2 Determine sample volume:

14.2.1.2.1 An indirect measurement is accomplished by weighing the sample and bottle to the nearest 0.1 gram. After extraction, weigh the empty bottle to the nearest 0.1 gram and subtract this value from the weight recorded prior to extraction. Assume a sample density of 1.0g/mL. Record the sample volumes for use in the final calculations of analyte concentrations.

14.2.1.2.2 Some of the PFAS adsorb to surfaces, thus the sample may not be transferred to a graduated cylinder for volume measurement.

14.2.1.2.3 The MB, LCS and LLCS must have the same volume as that of the field samples and may be prepared by measuring reagent water with a graduated cylinder.

14.2.1.3 Verify that the sample has a pH between 6.0 and 7.0. If adjustment is necessary, then 50% formic acid (10.3.12.6) or 3% aqueous ammonium hydroxide (10.3.12.7) may be added.

14.2.1.4 Fortify the QC Samples: Fortify LCS/LCSD (LFBs), LLCS (LLFB), MS (LFSMs), and MSD (LFSMDs), with an appropriate volume of Analyte PDS (Section 10.5.5.2.1.1) – 25uL for the LLCS and 100uL for the LCS/D, MS and MSD. Spike the solution directly into the original bottle. Cap and invert each sample several times to mix.



14.2.1.5 Add a 50 μ L aliquot of the isotope dilution EIS (Section 10.5.4) to each batch QC (MB, LCS, LCSD, LLCS), Matrix QC (MS and MSD), and field sample. Spike the solution directly into the original bottle, then cap and invert to mix.

14.2.2 Solid Phase Extraction for aqueous samples:

14.2.2.1 Cartridge Cleaning and Conditioning: Do not allow cartridge packing material to go dry during any of the conditioning steps. If the cartridge goes dry during the conditioning phase, the conditioning must be repeated. Do not use vacuum during the conditioning.

14.2.2.1.1 Pack each cartridge to half height with methanol washed salinized glass wool. Rinse each cartridge with 15 mL of 1% ammonium hydroxide in methanol (10.3.12.3).

14.2.2.1.2 Next, rinse each cartridge with 5 mL of 0.3M formic acid (10.3.12.4) without allowing the rinse to drop below the top edge of the packing.

14.2.2.1.3 Discard the wash solvents.

14.2.2.1.4 Close the valve and fill the tube with reagent water.

14.2.2.2 Cartridge Loading:

14.2.2.2.1 Attach the sample transfer tubes to the SPE cartridges and adjust the vacuum to approximately 5 inches Hg.

14.2.2.2.2 Begin adding sample to the cartridge. Adjust the vacuum and control valves so that the approximate flow rate is 5 mL/min.

14.2.2.2.3 Do not allow the cartridge to go dry before all the sample has passed through.

14.2.2.2.4 Flow rates above 5 mL/min during loading may cause low analyte recovery.

14.2.2.3 Sample Bottle Rinse:

14.2.2.3.1 After the entire sample has passed through the cartridge, but not allowing the sample level to drop below the top edge of the packing material, rinse the sample bottle with a 10 mL aliquot of reagent water using the Class A graduated cylinder or a repeater pipette.

14.2.2.3.2 Draw the rinse through the sample transfer tubes and the cartridges. Add 5 mL of 1:1 0.1M formic acid/methanol to the sample bottle and draw through the transfer tube and SPE cartridge. As the rinses near completion, do not allow the cartridges to go dry and shut the valve. Once all samples and QC



have reached the same step, then open all valves, allow the remaining liquid to elute and dry the cartridges for approximately 15 seconds.

14.2.2.4 Sample Bottle and Cartridge Elution and Extract completion/storage:

- 14.2.2.4.1 After the drying step, release the vacuum on the extraction manifold, ensure the valves are closed and place labeled 15mL collection tubes under each sample position.
- 14.2.2.4.2 Rinse the sample bottles with 5 mL of 1% ammonium hydroxide in methanol, then elute the analytes from the cartridges by pulling the elution solvent through the sample transfer tubes and the cartridges.
- 14.2.2.4.3 Use a low vacuum such that the solvent exits the cartridge in a dropwise fashion.
- 14.2.2.4.4 After elution is complete, add 25uL of the LC/MS acetic acid (10.3.10) and approximately 10mg of the loose carbon (10.3.6) to each extract. Vortex for approximately 15 to 30 seconds, and then centrifuge at 4000 rpm for approximately 5 minutes. It is important to minimize the amount of time the extracts are in contact with the carbon.
- 14.2.2.4.5 Label clean 15mL centrifuge tubes, remove the plunger from a clean 10mL disposable syringe and attach a 25mm 0.2 um syringe filter. Carefully decant the extract into the syringe leaving the carbon behind and gently replace the plunger while filtering the extract into the clean collection tube.
- 14.2.2.4.6 Once all extracts are filtered, use a 1.0mL pipette to add an aliquot of the extract to a 2.0mL polypropylene autosampler vial and add 10uL of the NIS working solution (10.5.3.1).

14.2.2.5 Recap vials as soon as possible after injection to prevent evaporation losses; the polypropylene caps do not reseal after puncture.

14.2.2.6 The remaining extracts can be stored in the 15 mL collection tubes after extraction. Store extracts at 0-4°C and protected from light.

14.3 Solid Extraction Procedure:

14.3.1 Setup and Initial extraction:

14.3.1.1 Perform the daily calibration verification of the open top balance.

14.3.1.2 Homogenize the sample and weigh 5.0 +/- 0.5g of sample in a labeled 50mL polypropylene centrifuge tube. Ottawa sand is used for the MB, LCS,



LCSD, and LLCS QC samples. Add 2.5mL of reagent water to the Ottawa sand in the QC samples.

- 14.3.1.3 Add 50uL of the Isotope EIS standard to each sample and QC (MB, LCS, LCSD, LLCS, MS, and MSD).
- 14.3.1.4 Add 100uL of the 1633 High Int standard to the LCS, LCSD, MS, and MSD. Add 25uL of the 1633 High Int standard to the LLCS. Vortex to mix well. Let stand for 30 minutes.
- 14.3.1.5 Add 10mL of the 0.3% Ammonium hydroxide in methanol solution to each tube using the repeater pipette.
- 14.3.1.6 Cap and vortex the contents to mix.
- 14.3.1.7 Shake on the shaker table at 300-350 rpm for approximately 30 minutes. Use a NIST timer to measure the 30min.
- 14.3.1.8 Centrifuge for approximately 10 minutes at 2800rpm. Decant the supernatant to a clean labeled 50mL centrifuge tube.
- 14.3.1.9 Add 15mL of the 0.3% ammonium hydroxide in methanol to the centrifuge tube containing the soil for a second extraction. Vortex to mix and shake on the shaker table for approximately 30 minutes.
- 14.3.1.10 Centrifuge for approximately 10 minutes at 2800rpm. Decant the supernatant into the tube containing the extract from the first shake.
- 14.3.1.11 Add 5mL of the 0.3% ammonium hydroxide in methanol to the tube containing the soil and vortex for approximately 30 seconds. Centrifuge for approximately 10 minutes at 2800rpm. And decant the supernatant into the tube containing the first 2 extractions.
- 14.3.1.12 Add approximately 10mg of the loose carbon to the combined extract in each tube, vortex for approximately 15 to 30 seconds and then centrifuge for approximately 5 minutes at 4000rpm. It is important to minimize the amount of time the extracts are in contact with the carbon. Decant into a clean 50mL centrifuge tube leaving the carbon behind.

14.3.2 Extract concentration:

- 14.3.2.1 At this step the analyst has the option of adding reagent water to the extract to dilute to approximately 35mL; this may be necessary for dry samples; however, the analyst should use their best judgement as to if adding water is necessary.
- 14.3.2.2 Concentrate each extract using an N-Evap with the water bath set to 55-60°C and N₂ flow of approximately 1.2L/min. Allow the extracts to concentrate for approximately 25 minutes and then remove and vortex for 5-10 seconds. Continue blow down, stopping approximately every 10 minutes and



vortexing, until the desired final volume is reached as outlined below:

| Water Content in Sample* | Concentrated Final Volume** |
|--------------------------|-----------------------------|
| < 5 g | 12mL |
| 5 – 8 g | 12-15mL |
| 8 – 9 g | 15-18mL |
| 9– 10 g | 16-19mL |

*the water content in the sample is determined from the % solids plus if any water was added in step 14.3.2.1. Per the method – a good rule of thumb is to make the “concentrated final volume” 7-10mL above the “water content in sample.”

**Note: the concentrated final volumes used were obtained from Alyssa Wingard’s email Memo on 05/18/2022 06:08AM EDT: “Clarification of draft method 1633 extract concentration procedures.”

Note: Slowly concentrating extracts, in 1-mL increments, is necessary to prevent excessive concentration and the loss of neutral compounds (methyl and ethyl FOSEs and FOSAs) and other highly volatile compounds. The extract must be concentrated to remove the methanol as excess methanol during SPE clean-up results in poor recovery of C13 and C14 carboxylic acids and C10 and C12 sulfonates. If all of the methanol is evaporated, the aforementioned neutral compounds are likely to have poor recovery; if too much methanol is in the final extract, then the aforementioned longer-chain compounds are likely to have poor recovery.

14.3.2.3 Add reagent water to the extract to bring the volume to approximately 50mL and vortex. Check that the pH is 6.5 ± 0.5 and adjust as necessary with 50% formic acid or 30% ammonium hydroxide (or with 5% formic acid and 3% aqueous ammonium hydroxide. The extracts are ready for SPE and cleanup.

14.3.3 SPE Cartridge Conditioning: Do not allow the cartridge(s) to go dry between steps. Do not use vacuum during the conditioning steps.

14.3.3.1 Pack each cartridge to half height with methanol washed salinized glass wool. Attach a 60mL reservoir to each cartridge. Rinse each reservoir and cartridge with 15 mL of 1% ammonium hydroxide in methanol (10.3.12.3).

14.3.3.2 Next, rinse each reservoir and cartridge with 5 mL of 0.3M formic acid (10.3.12.4) without allowing the rinse to drop below the top edge of the packing. Close the valve and fill the tube with reagent water.

14.3.3.3 Discard the wash solvents.

14.3.3.4 Pour the sample extracts into the reservoir being careful not to spill or splash. Pull the extracts through the cartridges using the vacuum at approximately 5mL/min. Retain the 50mL centrifuge tubes for later rinsing.

14.3.3.5 Discard the eluate.

14.3.3.6 As the extracts near filter completion, rinse the reservoir sides with 2 x 5mL rinses of reagent water. Follow with a 5mL rinse using 1:1 0.1M formic



acid/methanol and stop the flow without allowing the cartridge to go dry. Once all of the extracts have reached the same point, open the valves and elute the remaining liquid and dry the cartridges for approximately 15 seconds.

14.3.3.7 Discard the rinse solution.

14.3.4 Elution and Extract Prep and Storage:

14.3.4.1 After the drying step, release the vacuum on the extraction manifold, ensure the valves are closed and place a labeled 15mL collection tube under each sample position.

14.3.4.2 Add 5mL of 1% ammonium hydroxide in methanol to the 50mL centrifuge tubes retained from step 14.3.3.4, cap and shake to rinse the walls.

14.3.4.3 Pour the 5mL aliquot of 1% ammonium hydroxide/methanol into the respected reservoir for each sample, being careful not to spill or splash. Elute the cartridges pulling the 1% ammonium hydroxide/methanol through the cartridges in a drop-wise fashion.

14.3.4.4 Add 25uL of LC/MS grade acetic acid and vortex for approximately 15-30 seconds.

14.3.4.5 Label a clean set of 15mL polypropylene tubes, remove the plunger from a 10mL disposable syringe and attach a 25mm 0.2um syringe filter. Carefully pour the sample extract into the syringe and gently replace the plunger while filtering the extract into a clean tube.

14.3.4.6 Once all extracts are filtered, use a 1.0mL pipette to add an aliquot of the extract to a 2.0mL polypropylene autosampler vial and add 10uL of the NIS working solution (10.5.3.1).

14.3.4.7 Recap the autosampler vials after analysis to prevent evaporation; the polypropylene caps do not reseal. The remaining extract should be stored at 0-4°C and protected from light.

14.4 LCS-MS/MS analysis of samples:

14.4.1 Inject a 6 µL aliquot of the sample extract into the LC-MS/MS system, using the same operating conditions that were used for the calibration (Table 3 Section 24). The injection volume must be the same volume used for the calibration standards.

14.4.2 Qualitative analysis:

See also ADMIN SOP-038: Calibration, Manual Integration, and Rules for Chromatography.
See also TECH SOP-009 Multi-peak Compound Identification for Organics.
See also TECH SOP-010 Establishing and Maintaining Retention Time Windows.
(These three SOPs are required reading for any analyst performing this method).



- 14.4.2.1 Because environmental samples may contain both branched and linear isomers of the method analytes, but quantitative standards that contain branched isomers do not exist for all method analytes, integration and quantitation of the PFAS is dependent on the type of standard materials available.
- 14.4.2.2 Identify peaks by retention times. At the conclusion of data acquisition, use the same software settings established during the calibration procedure to identify analyte peaks in the predetermined retention time windows. Confirm the identity of each analyte by comparison of its retention time with that of the corresponding analyte peak in an initial calibration standard or CCV.
- 14.4.2.3 A native or isotopically labeled compound is identified in a standard, blank, sample, or QC sample when all of the following criteria are met:
- 14.4.2.3.1 Peak responses must be at least three times the background noise level (S/N 3:1). If the S/N ratio is not met due to high background noise, the laboratory must correct the issue (e.g., perform instrument troubleshooting to check and if needed, replace, the transfer line, column, detector, liner, filament, etc.). If the S/N ratio is not met but the background is low, then the analyte is to be considered a non-detect.
 - 14.4.2.3.2 Target analyte, EIS analyte, and NIS analyte RTs must fall within ± 0.4 minutes of the predicted retention times from the midpoint standard of the ICAL or initial daily CV, whichever was used to establish the RT window position for the analytical batch. The retention time window used must be of sufficient width to detect earlier-eluting branched isomers. For all method analytes with exact corresponding isotopically labeled analogs, method analytes must elute within ± 0.1 minutes of the associated EIS.
 - 14.4.2.3.3 For concentrations at or above the method LOQ, the total quantification ion (Q1) response to the total confirmation ion (Q2) response ratio must fall within $\pm 50\%$ of the ratio observed in the mid-point initial calibration standard. If project-specific requirements involve reporting sample concentrations below the LOQ or ML, the response ratio must also fall within $\pm 50\%$ of the ratio observed in the initial daily CCV.
 - 14.4.2.3.3.1 The total response of all isomers (branched and linear) in the quantitative standards should be used to define ratio. In samples, the total response should include only the branched isomer peaks that have been identified in either the quantitative or qualitative standard. If standards (either quantitative or qualitative) are not available for purchase, only the linear isomer can be identified and quantitated in samples. The ratio requirement does not apply for PFBA, PFPeA, NMeFOSE, NEtFOSE,



PFMPA, and PFMBA because suitable (not detectable or inadequate S/N) secondary transitions (Q2) are unavailable.

14.4.2.4 If the field sample result does not all meet the criteria above, and all sample preparation avenues (e.g., extract cleanup, sample dilution, etc.) have been exhausted, the result may only be reported with a data qualifier alerting the data user that the result could not be confirmed because it did not meet the method-required criteria and therefore should be considered an estimated value. If the criteria listed above are not met for the standards, the laboratory must stop analysis of samples and correct the issue.

14.4.2.5 For DOD work:

- 14.4.2.5.1 The chemical derivation of the ion transitions must be documented. A minimum of two ion transitions (Precursor → quant ion and precursor → confirmation ion) and the ion transitions ratio per analyte are required for confirmation.
- 14.4.2.5.2 Exception is made for analytes where two transitions do not exist (PFBA, PFPeA, and FOSA).
- 14.4.2.5.3 Documentation of the primary and confirmation transitions and the ion ratio is required.
- 14.4.2.5.4 In-house acceptance criteria for evaluation of ion ratios must be used and must not exceed 50-150% of the ion abundance ration in the midpoint calibration standard or daily CCV. The average qualifier ion ratios must be updated whenever a new calibration curve is ran; see section 13.1.3.4.4.
- 14.4.2.5.5 Signal to Noise Ratio (S/N) must be ≥ 10 for all ions used for quantification and must be ≥ 3 for all ions used for confirmation.
- 14.4.2.5.6 Quant ion and confirmation ion must be present and must maximize simultaneously (± 2 seconds).

14.4.3 Quantitative analysis

14.4.3.1 Once a compound has been identified, the analyte concentrations will be automatically quantified by the MassHunter software using the multipoint calibration and the measured sample volume. Report only those values that fall between the MDL and the highest calibration standard.

14.4.3.2 Proceed with quantitation based on the type of standard available for each method analyte.

- 14.4.3.2.1 If standards containing the branched and linear isomers cannot be purchased (i.e., only the linear isomer is available), only the linear isomer can be identified and quantitated in field samples



and QC samples because the retention time of the branched isomers cannot be confirmed.

14.4.3.2.2 Multiple chromatographic peaks, representing branched and linear isomers, are likely to be observed for standards of PFHxS, PFOS, N-Me-FOSAA and N-Et-FOSAA. For these analytes, all the chromatographic peaks observed in the standard must be integrated and the areas summed. Chromatographic peaks in all field samples and QC samples must be integrated in the same way as the calibration standard for analytes with quantitative standards containing the branched and linear isomers.

14.4.3.2.3 For **PFOA**, identify the branched and linear isomers by analyzing a technical-grade standard that includes both linear and branched isomers and ensure that all isomers elute within the same acquisition segment. **Quantitate field samples and fortified matrix samples by integrating the total response, accounting for peaks that are identified as linear and branched isomers. Quantitate based on the initial calibration with the quantitative PFOA standard containing just the linear isomer.**

14.4.3.3 Calculate the concentration of each isotope dilution analogue using the multipoint calibration and the measured sample volume. Verify that the percent recovery is within 20–150% of the true value.

14.4.3.4 The non-extracted internal standard area counts must be greater than 30% of the average of the ICAL for each sample and QC item.

14.4.3.5 The analyst must not extrapolate beyond the established calibration range. If an analyte result exceeds the range of the initial calibration curve, the extract may be diluted up to 10X and re-analyzed.

14.4.3.5.1 Dilute the extract accordingly using the methanol blank solution in section 10.3.11.9 as the final solvent, making a 1.0 mL volume. Select the dilution performed based on ensuring the EIS recovery is greater than 5%. For example, if the EIS recovery was 50% in the initial analysis, then a 10X dilution may be performed.

14.4.3.5.2 Add the appropriate amount of the Non-extracted internal standard to compensate for the dilution performed.

14.4.3.5.3 Report all concentrations measured in the original sample that do not exceed the calibration range.

14.4.3.5.4 Report concentrations of analytes that exceeded the calibration range in the in the original sample based on measurement in a diluted sample.



- 14.4.3.5.5 Incorporate the dilution factor into final concentration calculations and the resulting data must be annotated as a dilution. This is the only circumstance when subsampling is permitted.
- 14.4.3.5.6 If the EIS responses in the diluted extract do not meet the S/N and retention time requirements, then the compound cannot be measured reliably by isotope dilution in the diluted extract. In such cases, the laboratory must take a smaller aliquot of any affected aqueous sample and dilute it to 500 mL with reagent water and analyze the diluted aqueous sample or analyze a smaller aliquot of soil sample. Adjust the compound concentrations, detection limits, and minimum levels to account for the preparation dilution.
- 14.4.3.5.7 If a dilution greater than 10x is indicated, then the laboratory must prepare and analyze a diluted aqueous sample or a smaller aliquot of a solid sample.
- 14.4.3.5.8 If the recovery of any isotopically labeled compound is outside of the acceptance limits, a diluted aqueous sample or smaller aliquot (for solids and tissue) must be analyzed. If the recovery of any isotopically labeled compound in the diluted sample is outside of the normal range, the method does not apply to the sample being analyzed and the result may not be reported or used for permitting or regulatory compliance purposes.

14.4.4 Data reporting

14.4.4.1 Report results for aqueous samples in ng/L. Report results for solid samples in ng/g, on a dry-weight basis, and report the percent solids for each sample separately. Other units may be used if required in a permit or for a project. Report all QC data with the sample results.

15.0 Calculations

- 15.1 Multiply all concentrations by any dilutions incurred during extraction to obtain the final result.
- 15.2 Calculation for Stability of Methanolic Solutions:

$$\frac{\text{Total PFAS mass (g)} \times 160 \left(\frac{\text{g}}{\text{mol}}\right)}{250 \left(\frac{\text{g}}{\text{mol}}\right)} = \text{Mass of NaOH Required (g)}$$



15.3 Correction for Analytes Obtained in the Salt Form

$$mass(acid\ form) = mass(salt\ form) \times \frac{MW_{acid}}{MW_{salt}}$$

15.4 Calculation of Isotope Dilution Analogue Recovery

$$\%R = \frac{A}{B} \times 100$$

Where,

A = measured concentration of the isotope dilution analogue, and

B = fortification concentration of the isotope dilution analogue.

15.5 Average (% RSD) Calibration Calculations:

15.5.1 Calculate the Response factors (RF) or Relative response factor (RRF); for each compound:

The response ratio (RR) for each compound calibrated by isotope dilution is calculated according to the equation below, separately for each of the calibration standards, using the areas of the quantitation ions (Q1) with the m/z shown in Table 2. RR is used for the 24 compounds quantified by true isotope dilution.

$$RR = \frac{Area_n M_l}{Area_l M_n}$$

where:

Area_n = The measured area of the Q1 m/z for the native (unlabeled) PFAS

Area_l = The measured area at the Q1 m/z for the corresponding isotopically labeled PFAS added to the sample before extraction

M_l = The mass of the isotopically labeled compound in the calibration standard

M_n = The mass of the native compound in the calibration standard

Similarly, the response factor (RF) for each unlabeled compound calibrated by extracted internal standard is calculated according to the equation below. RF is used for the 16 compounds quantified by extracted internal standard.

$$RF = \frac{Area_s M_{EIS}}{Area_{EIS} M_s}$$

where:

Area_s = The measured area of the Q1 m/z for the target (unlabeled) PFAS

Area_{EIS} = The measured area at the Q1 m/z for the isotopically labeled PFAS used as the extracted internal standard (EIS)

M_{EIS} = The mass of the isotopically labeled PFAS used as the extracted internal standard (EIS) in the calibration standard

M_s = The mass of the target (unlabeled) PFAS in the calibration standard



A response factor (RF_s) is calculated for each isotopically labeled compound in the calibration standard using the equation below. RF_s is used for the 24 isotopically labeled compounds quantified by non-extracted internal standard.

$$RF_s = \frac{Area_1 M_{NIS}}{Area_{NIS} M_1}$$

where:

$Area_1$ = The measured area of the Q1 m/z for the isotopically labeled PFAS standard added to the sample before extraction

$Area_{NIS}$ = The measured area at the Q1 m/z for the isotopically labeled PFAS used as the non-extracted internal standard (NIS)

M_{NIS} = The mass of the isotopically labeled compound used as the non-extracted internal standard (NIS) in the calibration standard

M_1 = The mass of the isotopically labeled PFAS standard added to the sample before extraction

15.5.2 Calculate the mean response factor (RF) for each compound:

$$\overline{RF} = \frac{\sum_{i=1}^n RF_i}{n}$$

Where: n = The number of standards analyzed.

15.5.3 The processing software will calculate the standard deviation (SD) and the RSD of the calibration factors for each analyte as:

$$SD = \sqrt{\frac{\sum_{i=1}^n [RF_i - \overline{RF}]^2}{(n - 1)}} \quad RSD = \frac{SD}{\overline{RF}} * 100$$

Where: n = The number of standards analyzed.

15.5.4 Calculation of the percent difference (%D) for all analytes in the ICV and/or CCV:

$$\%D = \frac{C_{expected} - C_{found}}{C_{expected}} * 100 = \frac{RF_{ccv} - \overline{RF}}{\overline{RF}} * 100$$

Where:

$C_{expected}$ = the true value of the analyte or surrogate.

C_{found} = the on-column analyte or surrogate result

15.5.5 Determine the concentration of individual compounds in the sample using the following equation:



$$\text{Concentration (ug/L)} = \frac{(A_s) (I_s)}{(A_{is}) (\overline{RF})}$$

Where:

A_s = response of the analyte in the sample

I_s = concentration of internal standard present (in ug/L).

A_{is} = response of the internal standard

RF = Average Response Factor (unitless)

15.6 Linear Calibration Calculations (using a least squares regression (first-order) calibration fit):

15.6.1 Option 1: X_s is the concentration of the analyte in the calibration standard aliquot introduced into the instrument and Y_s is the ratio of response of the analyte to the response of internal standard times the mass of the internal standard in the calibration standard aliquot introduced into the instrument.

$$X_s = C_s \quad \text{and} \quad Y_s = A_s \times \frac{C_{is}}{A_{is}}$$

Where:

C_s = concentration of analyte in the volume of calibration standard introduced into the instrument.

C_{is} = concentration of internal standard in the volume of calibration standard injected into the instrument.

A_s = Peak response of analyte.

A_{is} = Peak response of internal standard.

15.6.2 Option 2: x is the ratio of the analyte concentration in the calibration standard aliquot introduced into the instrument to the internal standard concentration in the calibration standard aliquot introduced into the instrument and y is the ratio of response of the analyte to the response of internal standard.

$$x = \frac{C_s}{C_{is}} \quad \text{and} \quad y = \frac{A_s}{A_{is}}$$

Where:

C_s = concentration of analyte in the volume of calibration standard introduced into the instrument.

C_{is} = concentration of internal standard in the volume of calibration standard injected into the instrument.

A_s = Peak response of analyte.

A_{is} = Peak response of internal standard.

15.6.3 The linear least squares regression equation is:

$$y = ax + b$$



Where:

a = The slope of the linear regression.

b = The intercept of the linear regression.

15.6.4 The processing software will calculate the coefficient of determination (r^2) for each analyte by squaring the correlation coefficient as:

$$r^2 = \left[r = \frac{n \sum_{i=1}^n x_i y_i - \sum_{i=1}^n x_i \sum_{i=1}^n y_i}{\sqrt{(n \sum_{i=1}^n x_i^2 - (\sum_{i=1}^n x_i)^2)(n \sum_{i=1}^n y_i^2 - (\sum_{i=1}^n y_i)^2)}} \right]^2$$

15.6.5 Calculation of the Percent Drift for all analytes in the ICV and/or CCV:

$$\% \text{ Drift} = (\text{Analyte Result} / \text{True Value}) * 100$$

15.6.6 Calculations of sample amounts when using a linear regression calibration fit:

$$\text{Option 1} \quad X_s = \frac{\left(\frac{A_s \times C_{is}}{A_{is}} \right) - b}{a}$$

$$\text{Option 2} \quad X_s = \frac{\left(\frac{A_s}{A_{is}} \right) - b}{a} \times C_{is}$$

Where:

X_s = calculated concentration of the analyte or surrogate in the sample aliquot introduced into the instrument

A_s = peak response of the analyte or surrogate in the sample

A_{is} = peak response of the internal standard in the sample

C_{is} = concentration of the internal standard in the sample aliquot introduced into the instrument.

a = The slope of the linear regression.

b = The intercept of the linear regression.

15.7 Percent Recovery for standards and LCS/LCSD

$$\% \text{ Recovery} = (\text{LCS Result} / \text{True Value}) * 100$$

15.8 Percent Recovery for MS and MSD samples

$$\% \text{ Recovery} = [(\text{MS/D Result} - \text{Parent Sample Result}) / \text{Spike True Value}] * 100$$



15.9 Dry weight determination:

$$\text{mg/dry kg PH} = \frac{C_s}{1 - (\% \text{moisture}/100)}$$

Where:

C_s = Concentration of Pesticides (in mg/L or mg/kg)

15.10 Relative Percent Difference (RPD)

$$\% \text{ RPD} = \frac{|\text{Difference b/w Dups}|}{\text{Average of Dups}} * 100$$

15.11 Qualifier Ion Ratio: Mass Hunter calculates this value as the response(area) for the qualifier ion divided by the response(area) of the quantification ion times 100:

$$\text{Ratio} = (\text{qualifier ion area}/\text{quantifier ion area}) * 100$$

15.12 Non-extracted Internal Standard Mean area:

$$\text{Mean Area}_{\text{NIS}_i} = (\sum \text{Area}_{\text{NIS}_i})/n$$

Where:

$\text{Area}_{\text{NIS}_i}$ = Area counts for the i th NIS, where i ranges from 1 to 7, for the seven NIS compounds.

n = The number of ICAL standards (the default value is $n = 6$). If a different number of standards is used for the ICAL, for example, to increase the calibration range or by dropping a point at either end of the range to meet the linearity criterion, change 6 to match the actual number of standards used)

15.13 Method Detection Limit: The MDL is typically calculated as 3.143 times the standard deviation (SD).

$$\bar{X} = \frac{\sum_{i=1}^n X_i}{n} \quad SD = \sqrt{\frac{\sum_{i=1}^n [X_i - \bar{X}]^2}{(n - 1)}} \quad \text{MDL} = \text{SD} \times 3.143$$

Where: n = The number of standards analyzed.

X = One of the seven replicate MDL values.

16.0 Method Performance (See Sections 12.0 Quality Control and 18.0 Data Assessment and acceptance criteria for quality control measures).



17.0 Pollution Prevention

17.1 See Standard Methods section 1100, 22nd Edition or On-Line edition – Waste Minimization and Disposal.

17.2 See SOP Admin-018 and the AEL Safety Manual.

18.0 Data Assessment and acceptance criteria for quality control measures

18.1 It is the responsibility of the analyst to assess all data for quality control acceptance criteria.

18.2 It is the responsibility of the analyst to review all data/data entry for adherence to quality control criteria and any transcription or typographical errors prior to peer or supervisor review.

18.3 It is the responsibility of the analyst and/or supervisor to initiate and/or recommend correction action for out of control data.

18.4 All out of control data will be qualified according to SOP Admin-008.

18.5 See AEL QM Section 10.0.

18.6 All calibration will have a minimum of a six-point curve (Note: a minimum seven-point curve is required for a quadratic calibration fit). Points at the ends of the curve may be dropped if they are non-linear. If a data point is not usable or unacceptable within the curve, it will be re-analyzed. A data point for the ICAL can be dropped only if it can be documented as a non-repeatable error.

18.7 Each analytical batch at a minimum will follow the “24 hour rule,” where, at the beginning of each 24-hour period, an IB, and low level CCV must be analyzed. The following CCV’s will vary between mid and high levels. The final CCV must be injected within 24 hours of the opening CCV.

18.7.1 The instrument must be free of analytes as exhibited by an instrument blank at the beginning of each analysis window.

18.7.2 The Check Tune must pass the method criteria for any following data to be reported.

18.7.3 The CCV must meet method acceptance criteria to report unqualified data.

18.7.3.1 If any analyte fails with a low recovery, no data results can be reported for that analyte.

18.7.3.2 If any analyte fails with a high recovery, non-detects can be reported if qualified or properly noted in the case narrative. Corrective action (ex: remixing standard, cleaning injection liner, etc.) will be taken before the next analysis window (or next analytical sequence if the instrument is running multiple analysis windows in one night).

18.7.3.3 Failure to meet the CCV QC performance criteria requires corrective action.



Following a minor remedial action, such as servicing the autosampler or flushing the column, check the calibration with a mid-level CCV and a CCV at the MRL, or recalibrate according to Section 13. If isotope performance standard and calibration failures persist, maintenance may be required, such as servicing the LC-MS/MS system or replacing the LC column.

- 18.8 Method Blank (MB) – The MB must be clean of analytes of interest. If there are hits in the method blank, these hits must be below the method detection limit.
- 18.8.1 If the hits are above the method reporting level, and the samples themselves are clean of those hits (below the MDL), the sample results are fine to report.
- 18.8.2 If the MB results are above the MDL, and samples have the same hits, then sample results will not be accurate, and the MB and samples must be re-analyzed and/or re-extracted to prove that the hits were not contamination.
- 18.9 Laboratory Control Spikes (LCS) – The LCS must fall within control chart limits for percent recoveries of both analytes for valid data reporting. If the LCS fails, first check the instrument for possible problems. Document any issues, and then re-analyze the LCS. If the LCS still fails, then all samples in the extraction batch must be re-extracted or re-analyzed.
- 18.10 Matrix Spike/Matrix Spike Duplicate (MS/MSD) – Failure to meet control limits for analyte and surrogate recoveries does not in itself require data to be rejected. Data can be flagged (J(4)) for matrix interference.
- 18.11 At any point in the analytical batch, an analyst may use his/her discretion to fail or reject data he/she feels to be suspicious or in error. At this point, the analyst should seek the help of a supervisor or the QC officer.
- 18.12 Data should be rejected for individual sample runs if the chromatogram looks odd, retention times shift outside retention time windows, or surrogates fail due to reasons other than matrix interferences.
- 18.13 Any and all QC failures must be reported to a supervisor and the QC officer.
- 18.14 Extraction personnel should be informed of any failures immediately. They should also be informed of any trends that develop in sample recoveries.
- 19.0 Corrective action for out of control data
- 19.1 See Section 20.0 for out of control or unacceptable data.
- 19.2 See SOP ADMIN-016 and ADMIN-028.
- 20.0 Contingencies for handling out of control or unacceptable data
- 20.1 If a blank failure occurs, any sample containing that analyte will get a ‘V’ qualifier and an NCF is required.



20.1.1 If the MB exhibits contamination, but the samples are BDL for the analytes in question, there is no need for any qualifier nor does an NCF need to be written.

20.2 LCS failure

20.2.1 If the LCS fails the lower criteria and there is insufficient sample to re-extract, the sample will be reported as is; however, every analyte that failed will get a “J(3)” qualifier and an NCF is required.

20.2.2 If the LCS fails the upper criteria and the sample is BDL for the analyte in question, the failure will be case narrated. The sample will not be qualified nor does an NCF need to be written.

20.2.3 If the LCS fails the upper criteria and the sample contains a hit for the analyte in question and there is insufficient sample to re-extract, the sample will be reported as is; however, the analyte in question will get a “J(3)” qualifier and an NCF is required.

20.3 If the native sample that is used for the MS/MSD has a target analyte detected at a concentration greater than 4 times the spike value, the spike recovery in the MS and MSD will not be calculated. The MS/MSD failure will be case narrated and the sample will not be qualified.

20.4 See ADMIN-016 for the NCF writing process.

20.5 See ADMIN-028 for the Case Narrative writing process.

21.0 Waste Management

21.1 Refer to SOP for Waste Management (ADMIN-018) for any other questions.

21.2 See Standards Method section 1090, 22nd Edition or On-Line edition– Waste Minimization and Disposal.

22.0 Cautions/Preventative Maintenance

22.1 Routine, preventative instrument maintenance must be performed and documented in a maintenance logbook to assure optimum instrument performance. All maintenance is documented in the maintenance log in accordance with Quality Manual 8.0.

22.2 System Carryover – Highly concentrated calibration standards and client samples containing high concentrations of target analytes can be retained in the GC systems and bleed out (carryover) into subsequent QC and client samples. Blanks must be analyzed after the initial calibration and “hot” client samples to prove the system is free of such contamination before more batches QC (MB, etc.) are analyzed. It is not acceptable to delete carryover from batch QC or client samples. Client samples must be rerun to confirm suspected hits from carryover.

22.3 Liquid Chromatogram

22.3.1 A regular schedule of maintenance is dictated more by the instrument checks and a visual check of the chromatography more than by any set schedule. Most



maintenance is done in response to a failure of one of the QC checks done during the course of normal operation or poor chromatographic performance. These checks ensure that the instrument is working at top performance and is proof that the instrument is in good working order.

22.3.2 “Hot” or “dirty” samples or the cumulative effect of many samples can cause the chromatography to degrade as well. Performing routine maintenance can bring performance back to normal operation. However, in some cases when the chromatography is not improved with this maintenance, the column will require replacement.

22.3.3 A dirty detector can result with use over time. If the baseline is seen as becoming erratic or the signal response is seen to degrade, this may indicate that the detector needs cleaning. Cleaning the source will most times restore full signal response.

22.4 When any maintenance is performed, the system should be carefully inspected for leaks prior to beginning analysis. Any parts of the instrument that have been recently taken apart and re-assembled are the most likely places for a leak to develop. It is also important to periodically check for leaks at the detectors where the columns are inserted.

22.5 Typical Instrument Preventative Maintenance Schedule – LC-MS/MS

22.5.1 Daily:

22.5.1.1 Keep wash/syringe rinse filled with appropriate reagent.

22.5.1.2 Check the Nitrogen tank pressure, replace tank as needed.

22.5.2 Monthly:

22.5.2.1 Clean auto-samplers and check that needles are clean and in working condition (clean or replace the syringes if needed).

22.5.2.2 Clean any accumulated dust and dirt off of the instrumentation.

22.5.2.3 Check the fore line pump oil level. Add pump fluid as needed until the oil level in the window is near, but not above, the upper fill line.

22.5.2.4 Clean the spray shield and the surrounding areas within the source. Remove and inspect the nebulizer, checking the tip for proper position and for build up and/or clogging.

22.5.3 Semi-Annual:

22.5.3.1 Schedule a preventative maintenance visit from Agilent.

22.5.3.2 Vent the system and change and/or clean the capillary.

22.5.4 Yearly:



22.5.4.1 Replace oil in rotary pump.

22.5.4.2 Replace column (or earlier if needed).

22.5.5 The laboratory must be cleaned regularly to prevent background contamination of the analyte PFBA. During method development it was found that PFBA was coming from the room's ventilation system. The duct coverings must be wiped down at least every 2 weeks. Also, HEPA filters are used in the system intake, and the intake grate should also be wiped down every 2 weeks. The HEPA filter should be replaced at a minimum of every 6 months.

23.0 References

- 23.1 *DETERMINATION OF PER- AND POLYFLUOROALKYL SUBSTANCES IN DRINKING WATER BY ISOTOPE DILUTION ANION EXCHANGE SOLID PHASE EXTRACTION AND LIQUID CHROMATOGRAPHY/TANDEM MASS SPECTROMETRY*, Method 533, November 2019, U.S. EPA Document No. 815-B-19-020.
- 23.2 *2nd DRAFT method 1633 – Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS*. June 2022.
- 23.3 Standard Methods 22nd Edition or On-Line edition.
- 23.4 EPA CFR 40 Part 136.6, Appendix B.
- 23.5 TNI Standards 2016
- 23.6 ISO 17025: 2005 & 2017 Standards
- 23.7 DoD ELAP QSM Rev. 5.4 October 2021
- 23.8 AEL Health and Safety Manual.
- 23.9 AEL Policy and Procedures Manual.
- 23.10 AEL Quality Manual, latest revision.
- 23.11 AEL Admin SOPs.

24.0 Tables, Diagrams, flowcharts, and validation data

- 24.1 SOP Validation Data. See the employee file for the following individuals for an acceptable initial demonstration of capability, which serves as validation data for this method at AEL.



24.2 Table 1 – NIS and EIS mass labeled analytes and Retention Times, and Reference NIS for each EIS.

| Non-extracted Internal Standards (NIS) | | RT(min) |
|---|--------------|---------|
| Full Name | Abbreviation | |
| Perfluoro-n-[2,3,4-13C3]butanoic acid | 13C3-PFBA | 6.68 |
| Perfluoro-n-[1,2-13C2]hexanoic acid | 13C2-PFHxA | 13.14 |
| Perfluoro-n-[1,2,3,4-13C4]octanoic acid | 13C4-PFOA | 18.36 |
| Perfluoro-n-[1,2,3,4,5-13C5] nonanoic acid | 13C5-PFNA | 20.66 |
| Perfluoro-n-[1,2-13C2]decanoic acid | 13C2-PFDA | 23.99 |
| Perfluoro-1-hexane[18O2]sulfonic acid | 18O2-PFHxS | 16.26 |
| Perfluoro-n-[1,2,3,4-13C4]octanesulfonic acid | 13C4-PFOS | 20.731 |

| Extracted Internal Standards (EIS) | | RT (min) | Reference NIS |
|--|---------------------------------------|----------|-------------------------------------|
| Full Name | Abbreviation | | |
| Perfluoro-n-[13C4]butanoic acid | ¹³ C ₄ -PFBA | 6.67 | ¹³ C ₃ -PFBA |
| Perfluoro-n-[13C5]pentanoic acid | ¹³ C ₅ -PFPeA | 13.14 | ¹³ C ₂ -PFHxA |
| Perfluoro-1-[2,3,4-13C3]butanesulfonic acid | ¹³ C ₃ -PFBS | 10.63 | ¹⁸ O ₂ -PFHxS |
| 1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]hexane sulfonic acid | ¹³ C ₂ -4:2FTS | 12.79 | ¹⁸ O ₂ -PFHxS |
| Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid | ¹³ C ₅ -PFHxA | 13.14 | ¹³ C ₂ -PFHxA |
| Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid | ¹³ C ₃ -HFPO-DA | 14.07 | ¹³ C ₂ -PFHxA |
| Perfluoro-n-[1,2,3,4-13C4]heptanoic acid | ¹³ C ₄ -PFHpA | 16.00 | ¹³ C ₂ -PFHxA |
| Perfluoro-1-[1,2,3-13C3]hexanesulfonic acid | ¹³ C ₃ -PFHxS | 16.25 | ¹⁸ O ₂ -PFHxS |
| 1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]octane sulfonic acid | ¹³ C ₂ -6:2FTS | 18.18 | ¹⁸ O ₂ -PFHxS |
| Perfluoro-n-[13C8]octanoic acid | ¹³ C ₈ -PFOA | 18.36 | ¹³ C ₄ -PFOA |
| Perfluoro-n-[13C9]nonanoic acid | ¹³ C ₉ -PFNA | 20.66 | ¹³ C ₅ -PFNA |
| Perfluoro-1-[13C8]octanesulfonic acid | ¹³ C ₈ -PFOS | 20.73 | ¹³ C ₄ -PFOS |
| 1H,1H,2H,2H-Perfluoro-1-[1,2-13C2]decane sulfonic acid | ¹³ C ₂ -8:2FTS | 23.78 | ¹⁸ O ₂ -PFHxS |
| Perfluoro-n-[1,2,3,4,5,6-13C6]decanoic acid | ¹³ C ₆ -PFDA | 23.98 | ¹³ C ₂ -PFDA |
| Perfluoro-n-[1,2,3,4,5,6,7-13C7]undecanoic acid | ¹³ C ₇ -PFUnA | 27.69 | ¹³ C ₂ -PFDA |
| Perfluoro-n-[1,2-13C2]dodecanoic acid | ¹³ C ₂ -PFDoA | 28.37 | ¹³ C ₂ -PFDA |
| N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid | d5-N-Et-FOSAA | 27.69 | ¹³ C ₄ -PFOS |
| N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid | d3-N-Me-FOSAA | 26.12 | ¹³ C ₄ -PFOS |
| Perfluoro-n-[1,2-13C2]tetradecanoic acid | M2PFTeDA | 28.97 | ¹³ C ₂ -PFDA |
| Perfluoro-1-[13C8]octanesulfonamide | ¹³ C ₈ -PFOSA | 27.27 | ¹³ C ₄ -PFOS |
| N-methyl-d3-perfluoro-1-octanesulfonamide | D3-NMeFOSA | 28.90 | ¹³ C ₄ -PFOS |
| N-ethyl-d5-perfluoro-1-octanesulfonamide | D5-NEtFOSA | 29.20 | ¹³ C ₄ -PFOS |
| N-methyl-d7-perfluorooctanesulfonamidoethanol | D7-NMeFOSE | 28.85 | ¹³ C ₄ -PFOS |
| N-ethyl-d9-perfluorooctanesulfonamidoethanol | D9-NEtFOSE | 29.15 | ¹³ C ₄ -PFOS |



24.3 Table 2 – Method analytes, Retention times, and Suggested Isotope Dilution Analogue References

| Analyte | RT (min) | Isotope Dilution Analogue |
|------------|----------|---------------------------------------|
| PFBA | 6.68 | ¹³ C ₄ -PFBA |
| PFMPA | 7.86 | ¹³ C ₅ -PFPeA |
| 3:3FTCA | 9.79 | ¹³ C ₅ -PFPeA |
| PFPeA | 9.92 | ¹³ C ₅ -PFPeA |
| PFBS | 10.63 | ¹³ C ₃ -PFBS |
| PFMBA | 10.92 | ¹³ C ₅ -PFPeA |
| PFEESA | 11.90 | ¹³ C ₅ -PFHxA |
| NFDHA | 12.61 | ¹³ C ₅ -PFHxA |
| 4:2FTS | 12.80 | ¹³ C ₂ -4:2FTS |
| PFHxA | 13.14 | ¹³ C ₅ -PFHxA |
| PFPeS | 13.61 | ¹³ C ₃ -PFHxS |
| HFPO-DA | 14.07 | ¹³ C ₃ -HFPO-DA |
| PFHpA | 16.01 | ¹³ C ₄ -PFHpA |
| PFHxS | 16.26 | ¹³ C ₃ -PFHxS |
| ADONA | 16.35 | ¹³ C ₃ -HFPO-DA |
| 5:3FTCA | 16.32 | ¹³ C ₅ -PFHxA |
| 6:2FTS | 18.19 | ¹³ C ₂ -6:2FTS |
| PFOA | 18.36 | ¹³ C ₈ -PFOA |
| PFHpS | 18.48 | ¹³ C ₈ -PFOS |
| PFNA | 20.67 | ¹³ C ₉ -PFNA |
| PFOS | 20.74 | ¹³ C ₈ -PFOS |
| 7:3FTCA | 21.33 | ¹³ C ₅ -PFHxA |
| 9Cl-PF3ONS | 22.72 | ¹³ C ₃ -HFPO-DA |
| 8:2 FTS | 23.77 | ¹³ C ₂ -8:2FTS |
| PFNS | 24.03 | ¹³ C ₈ -PFOS |
| PFDA | 23.99 | ¹³ C ₆ -PFDA |
| N-Me-FOSAA | 26.20 | d ₃ -N-MeFOSAA |

| Analyte | RT (min) | Isotope Dilution Analogue |
|--------------|----------|---------------------------------------|
| PFDS | 27.67 | ¹³ C ₈ -PFOS |
| FOSA | 27.27 | ¹³ C ₈ -PFOSA |
| PFUnA | 27.68 | ¹³ C ₇ -PFUnA |
| N-Et_FOSAA | 27.71 | d ₅ -N-EtFOSAA |
| 11Cl-PF3OUdS | 28.19 | ¹³ C ₃ -HFPO-DA |
| PFDoA | 28.37 | ¹³ C ₂ -PFDoA |
| PFDoS | 28.67 | ¹³ C ₈ -PFOS |
| PFTTrDA | 28.71 | <i>Average*</i> |
| N-Me-FOSE | 28.88 | D ₇ -NMeFOSE |
| N-Me-FOSA | 28.91 | D ₃ -NMeFOSA |
| PFTeDA | 28.97 | M ₂ PFTeDA |
| N-Et-FOSE | 29.17 | D ₉ -NEtFOSE |
| N-Et-FOSA | 29.21 | D ₅ -NEtFOSA |

*For improved accuracy, PFTTrDA is quantitated using the average areas of the labeled analytes ¹³C₂-PFTeDA and ¹³C₂-PFDoA.



24.4 Table 3 – Recommended LC-MS/MS Operating Conditions

| HPLC Program | | |
|--------------|--------------------------|------------|
| Time (min) | % 20 mM ammonium acetate | % Methanol |
| Initial | 95.0 | 5.0 |
| 0.5 | 95.0 | 5.0 |
| 3.0 | 60.0 | 40.0 |
| 16.0 | 32.0 | 68.0 |
| 24.0 | 32.0 | 68.0 |
| 26.0 | 5.0 | 95.0 |
| 28.5 | 5.0 | 95.0 |
| 29.0 | 95.0 | 5.0 |
| 30.0 | 95.0 | 5.0 |

| LC-MS/MS Conditions ^{a,b} | | | | | |
|---------------------------------------|-------------------------------------|-------------------------------------|---------------------------|-------------------|--------------------------------------|
| Analyte | Precursor Ion ^c (m/z) | Product Ion ^{c,d} (m/z) | Confirmation Ion (m/z) | Fragmentor (v) | Collision Energy ^e (v) |
| PFBA | 213 | 169 | NA | 60 | 8 |
| ¹³ C ₃ -PFBA | 216 | 172 | NA | 65 | 8 |
| ¹³ C ₄ -PFBA | 217 | 172 | NA | 60 | 8 |
| PFMPA | 229 | 85 | NA | 60 | 12 |
| PFPeA | 263 | 219 | 68.9 | 70 | 4 |
| ¹³ C ₅ -PFPeA | 268 | 223 | NA | 60 | 8 |
| ¹³ C ₃ -PFBS | 302 | 80 | 99 | 125 | 38 |
| PFBS | 299 | 80 | 99 | 137 | 38 |
| PFMBA | 279 | 85 | NA | 70 | 12 |
| PFEESA | 315 | 134.9 | 83 | 104 | 26 |
| NFDHA | 295 | 201 | 85 | 83 | 2 |
| ¹³ C ₂ -4:2FTS | 329 | 80.9 | 309 | 135 | 34 |
| 4:2FTS | 327 | 307 | 81 | 125 | 20 |
| ¹³ C ₅ -PFHxA | 318 | 273 | 120 | 58 | 6 |
| ¹³ C ₂ -PFHxA | 315 | 270 | N/A | 68 | 6 |
| PFHxA | 313 | 269 | 119 | 53 | 6 |
| PFPeS | 349 | 80 | 99 | 142 | 50 |
| ¹³ C ₃ -HFPO-DA | 287 ^f | 169 | 185 | 68 | 2 |
| HFPO-DA | 285 ^f | 169 | 185 | 68 | 6 |
| ¹³ C ₄ -PFHpA | 367 | 322 | NA | 72 | 0 |
| PFHpA | 363 | 319 | 169 | 71 | 10 |



LC-MS/MS Conditions (Continued) ^{a,b}

| Analyte | Precursor Ion ^c (m/z) | Product Ion ^{c,d} (m/z) | Confirmation Ion (m/z) | Fragmentor (v) | Collision Energy ^e (v) |
|--|-------------------------------------|-------------------------------------|---------------------------|-------------------|--------------------------------------|
| ¹³ C ₃ -PFHxS ^g | 402 | 80 | 99 | 152 | 50 |
| ¹⁸ O ₂ -PFHxS ^g | 403 | 83.9 | NA | 134 | 42 |
| PFHxS ^h | 399 | 80 | 99 | 140 | 50 |
| ADONA | 377 | 251 | 85 | 60 | 10 |
| ¹³ C ₂ -6:2FTS | 429 | 80.9 | 309 | 130 | 38 |
| 6:2FTS | 427 | 407 | 81 | 125 | 24 |
| ¹³ C ₄ -PFOA | 417 | 172 | NA | 76 | 22 |
| ¹³ C ₈ -PFOA | 421 | 376 | NA | 69 | 4 |
| PFOA | 413 | 369 | 169 | 125 | 6 |
| PFHpS | 449 | 80 | 99 | 140 | 58 |
| ¹³ C ₉ -PFNA | 472 | 427 | NA | 66 | 4 |
| ¹³ C ₅ -PFNA | 468 | 423 | NA | 81 | 6 |
| PFNA | 463 | 419 | 219 | 91 | 6 |
| ¹³ C ₄ -PFOS ^g | 503 | 80 | 99 | 180 | 52 |
| ¹³ C ₈ -PFOS ^g | 507 | 80 | 99 | 155 | 60 |
| PFOS ^h | 499 | 80 | 99 | 160 | 58 |
| 9Cl-PF3ONS ⁱ | 531 | 351 | 532.8→353.0 | 130 | 30 |
| ¹³ C ₂ -8:2FTS | 529 | 80 | 509 | 170 | 54 |
| 8:2FTS | 527 | 507 | 81 | 170 | 28 |
| ¹³ C ₆ -PFDA | 519 | 474 | NA | 81 | 4 |
| ¹³ C ₂ -PFDA | 515 | 470 | NA | 81 | 4 |
| PFDA | 513 | 469 | 219 | 98 | 8 |
| ¹³ C ₇ -PFUnA | 570 | 525 | NA | 73 | 5 |
| PFUnA | 563 | 519 | 269 | 132 | 10 |
| ¹¹ C ₁ -PF3OUds ^j | 631 | 451 | 632.9→452.9 | 160 | 30 |
| ¹³ C ₂ -PFDoA | 615 | 570 | NA | 79 | 5 |
| PFDoA | 613 | 569 | 319 | 89 | 10 |
| ¹³ C ₈ -PFOSA | 506 | 78 | NA | 125 | 40 |
| FOSA | 498 | 78 | 478 | 150 | 42 |
| PFNS | 549 | 80 | 99 | 127 | 54 |
| PFDS | 599 | 80 | 99 | 145 | 60 |
| N-Et-FOSAA | 584 | 419 | 526 | 117 | 18 |
| N-Me-FOSAA | 570 | 419 | 438 | 129 | 18 |
| d5-N-Et-FOSAA | 589 | 419 | NA | 115 | 24 |
| d3-N-Me-FOSAA | 573 | 419 | NA | 115 | 16 |
| PFTeDA | 713 | 669 | 169 | 101 | 14 |
| PFTrDA | 663 | 619 | 169 | 109 | 10 |
| M2PFTeDA | 715 | 670 | NA | 100 | 13 |
| PFDoS | 699 | 80 | 99 | 132 | 60 |



LC-MS/MS Conditions (Continued) ^{a,b}

| Analyte | Precursor Ion ^c (<i>m/z</i>) | Product Ion ^{c,d} (<i>m/z</i>) | Confirmation Ion (<i>m/z</i>) | Fragmentor (<i>v</i>) | Collision Energy ^e (<i>v</i>) |
|------------|--|--|------------------------------------|----------------------------|---|
| 3:3FTCA | 241 | 177 | 117 | 76 | 2 |
| 5:3FTCA | 341 | 237 | 217 | 83 | 10 |
| 7:3FTCA | 441 | 317 | 337 | 109 | 22 |
| N-Me-FOSA | 512 | 219 | 169 | 140 | 22 |
| N-Et-FOSA | 526 | 219 | 169 | 126 | 26 |
| N-Me-FOSE | 616 | 58.9 | NA | 89 | 14 |
| N-Et-FOSE | 630 | 58.9 | NA | 119 | 50 |
| D3-NMeFOSA | 515 | 219 | NA | 137 | 26 |
| D5-NEtFOSA | 531 | 219 | NA | 165 | 26 |
| D7-NMeFOSE | 623 | 58.9 | NA | 94 | 14 |
| D9-NEtFOSE | 639 | 58.9 | NA | 91 | 54 |

ESI Conditions for Agilent 6470 QQQ

| | |
|-----------------------------|--------------|
| Polarity | Negative ion |
| Capillary needle voltage | -2.5 kV |
| Sheath Gas Flow | 12 L/min |
| Nitrogen desolvation gas | 8 L/min |
| Desolvation gas temperature | 170°C |

NA = These analytes do not produce a confirmation ion mass

- ^{a.} An LC-MS/MS chromatogram of the analytes obtained using these parameters is shown in **Figure 1**.
- ^{b.} Segments are time durations in which single or multiple scan events occur.
- ^{c.} Precursor and product ions listed in this table are nominal masses. During MS and MS/MS optimization, the analyst determined precursor and product ion masses to one decimal place by locating the apex of the mass spectral peak (e.g., *m/z* 498.9 79.9 for PFOS). These precursor and product ion masses (with at least one decimal place) should be used in the MS/MS method for all analyses.
- ^{d.} Ions used for quantitation purposes.
- ^{e.} Nitrogen used as collision gas.
- ^{f.} HFPO-DA and NFDHA are not stable in the ESI source and the $[M - H]^-$ yields a weak signal under typical ESI conditions. The precursor ion used during method development was $[M - CO_2 - H]^-$.
- ^{g.} The isotope dilution analogue used during method development was composed of the linear isomer exclusively.
- ^{h.} Analyte has multiple resolved chromatographic peaks due to linear and branched isomers. All peaks summed for quantitation purposes. To reduce bias regarding detection of branched and linear isomers, the *m/z* 80 product ion must be used for this analyte.
- ^{i.} The qualifier ion transition used for this analyte is 532.8-353.0.
- ^{j.} The qualifier ion transition used for this analyte is 632.9-452.9.



24.5 Table 4 – Typical 1633 Analytical Sequence

| Calibration Curve Analytical Sequence |
|---|
| Rinse |
| IB |
| ICAL (1 through 9) |
| IB |
| ICV |
| IB |
| Qualitative ID standards |
| Bile salts mix |
| ISC (Low-Level CCV) |
| Mid-level CCV |
| IB |
| Batch QC, matrix QC and 10 client samples |
| Mid-Level CCV |
| 10 client samples |
| Mid-Level CCV |

| Daily Analytical Sequence |
|-----------------------------------|
| Rinse |
| IB |
| Qualitative ID standards |
| Bile salts mix |
| ISC (Low Level CCV) |
| Mid-level CCV |
| Batch QC and 10 client samples |
| Mid-Level CCV |
| 10 client samples |
| Mid-level CCV |



24.6 Table 5 – Method Required Quality Control for 1633 and DOD Table B-24

| Initial Demonstration of Capability | | |
|---|--|---|
| Requirement | Specification and Frequency | Acceptance Criteria |
| Establish retention times for branched isomers | Each time chromatographic conditions change. | All isomers of each analyte must elute within the same MRM window. |
| Demonstration of low system background | After calibration, analyze an Instrument Blank (IB) following the highest standard in the calibration range. | Demonstrate that the method analytes are less than one-half of the Minimum Reporting Level (MRL). |
| Initial Precision and Recovery (IPR): demonstration of accuracy and precision | For each matrix, extract and analyze a MB and 4 replicate Laboratory Fortified Blanks (LFBs) near the mid-range concentration. All processing steps must be included (extraction, cleanup, and concentration). IPR is required for new staff and whenever method modifications are made. | Results must be within 80–120% of the true value to meet AEL acceptance criteria. Percent relative standard deviation must be ≤20% to meet AEL acceptance criteria. <i>Note: Once Table 5 in the EPA Draft 1633 method is finalized, the laboratory will adopt those specific limits.</i> |
| Method Detection Limit | Establish MDLs as per Section 12.0 of this SOP and 40 CFR Part 136 App. B. MDL verification is required at initial instrument set-up, and whenever method modifications are made. | Initial MDL: 7 spiked samples and 7 method blank samples prepared in at least three batches on three separate calendar dates and analyzed on three separate calendar dates. New Instrument or Method Modification: 2 spiked samples and 2 method blank samples prepared & analyzed on different calendar dates & compared against existing MDLs for validity. If both method blank results are below the existing MDL, then the existing MDL _s is validated. Recalculate the MDL _s to include the two spiked samples. If the recalculated MDLs is within 0.5 to 2.0 times the existing MDL, the existing MDL is validated. |



| Quality Control Requirements | | |
|-------------------------------------|--|--|
| Requirement | Specification and Frequency | Acceptance Criteria |
| Mass Calibration | Annually and on as-needed basis perform a full Autotune. Daily before starting an analytical sequence perform a Checktune. | Follow vendor's procedure. If daily checktune fails any mass for the Negative mode (EPA 1633 specifies +/- 0.2Da), then stop and perform a full Autotune. A new ICAL will need to be ran following a new Autotune. |
| Retention time windows | Set RT windows for each analyte, EIS, and NIS during ICAL, and with each CCV ran at the beginning of the sequence. | Method analytes, EIS and NIS must be within 0.4 minutes of the RT from the mid-level standard of the ICAL, or the daily CCV. Each analyte must fall within 0.1 minute of its EIS; this only applies to analytes that have a mass-labeled analog. |
| Bile salts | DOD – Ran at the beginning of an analytical sequence for DOD only. 1633 – Requires an initial bile salt interference when establishing chromatographic conditions (regardless of matrix). A daily salt check is not required unless running tissue samples. | Each bile salt must not elute within 1 minute of all PFOS isomers. |
| Qualitative Identification Standard | Analyze daily at the beginning of the analytical sequence. | Confirm the RT of each linear and known branched isomer or isomer group. Quantitative standards containing isomeric mixtures for an analyte are commercially available for PFOS, PFHxS, NMeFOSAA, and NEtFOSAA. Qualitative/technical standards are available for PFOA, NEtFOSA, NMeFOSE, and NEtFOSE. |



| Quality Control Requirements (Continued) | | |
|---|--|--|
| Requirement | Specification and Frequency | Acceptance Criteria |
| Initial calibration | <p>Analyzed as a corrective action for CCC exceedances, after major instrument maintenance, or after mass recalibration.</p> <p>Use the isotope dilution calibration technique to generate a calibration curve using either average CF, linear or quadratic curve fits. Use at least 6 standard concentrations; 7 for Quadratic. Linear and Quadratic fits must be forced through origin. Weighted fits allowed.</p> <p>Qualifier ion ratios must be updated with each new calibration curve to the average of the calibration levels used to generate the curves.</p> | <p>When each calibration standard is calculated as a known using the calibration curve, all levels should be within 70–130% of the true value.</p> <p>1633 – Low Level signal-to-noise Ratio $\geq 3:1$ %RSD/RSE must be $\leq 20\%$; Record the mean area response for each NIS. Analyze a Bile Salt standard after calibration (see acceptance criteria above).</p> <p>DOD – %RSD $\leq 20\%$, or $R^2 > 0.990$</p> |
| Instrument Blank | Analyze an instrument blank at the beginning of a sequence, after the High-level of the ICAL (if running), after each CCC and after any field sample that has analyte detections exceeding the range of the ICAL (if possible). | DOD – All analytes must be $\leq \frac{1}{2}$ the MRL/LOQ. If any samples ran after a sample with a detection above the calibration range, had detections for that analyte above $\frac{1}{2}$ MRL, they must be re-analyzed. |
| Initial calibration verification (ICV) | Second source standard ran after Initial calibration, prior to sample analysis. May be from same vendor but must be different mix/lot. | DOD – Analyte concentrations must be 70-130% of their true value. |
| Instrument Sensitivity Check | Ran daily prior to sample analysis; DOD specifies once every 12 hours thereafter. Concentrations of analytes at, or below, MRL/LOQ. | 1633 – Signal to noise must be $\geq 3:1$. DOD – all analytes must be 70-130% of true value. |
| Continuing Calibration Check (CCC) or Calibration Verification (CV) | Verify initial calibration by analyzing a mid-level CCC at the beginning of each Analysis Batch. Subsequent CCCs are required after every tenth field sample and to complete the batch. | 1633 and DOD – all analytes and EIS must be 70-130% of true value. Set sequence IAR acceptance window as 50-150% of IAR in midpoint calibration standard or daily CCV. |



| Quality Control Requirements (Continued) | | |
|---|--|---|
| Requirement | Specification and Frequency | Acceptance Criteria |
| Non-extracted Internal Standards (NIS) | Non-extracted Internal standards are added to all standards and sample extracts. | <p>1633 – area recoveries are evaluated against EPA Draft 1633 Table 10 (see section 24.7 Table); criteria subject to change upon finalization of the draft method.</p> <p>(optional): NIS areas in field samples and QC samples should be 50-200% of the mean area of the ICAL.</p> <p>DOD – Area recoveries must be greater than 30% of the average of the ICAL. NIS areas corrected for the dilution factor must be greater than 30% of the average area of the calibration standards in diluted samples when additional NIS was not added post dilution of the extract.</p> |
| Extracted Internal Standards (EIS). | Isotope dilution analogues are added to all samples prior to extraction. | <p>1633 - % recovery for the EIS are evaluated against EPA Draft 1633 Table 9 (see section 24.8 Table); criteria subject to change upon finalization of the draft method.</p> <p>DOD – EIS recoveries must be within 20-150% until in-house limits can be created, or no project limits are provided. In-house limits cannot be lower than 20%.</p> |
| Laboratory Control Sample (LCS) and Low-Level Laboratory Control Sample (LLCS). (Equivalent to as OPR and LLOPR in method 1633) | Include one LCS per Extraction Batch. Fortify the LCS with method analytes at a concentration near the mid-point of the curve. If not enough sample is provided for MS/MSD, then a LCSD must be performed. | <p>1633 – Standard AEL limits of 80-120% have been assigned until the multi-laboratory evaluation is completed by the EPA. The lab may also evaluate LCS against EPA Draft Method Table 5 (see section 24.9 Table); criteria subject to change upon finalization of the draft method</p> <p>DOD – recovery must be within 40-150%, until in-house limits can be created, or if project limits are not provided. In-house limits cannot be lower than 40%.</p> |



Quality Control Requirements (Continued)

| Requirement | Specification and Frequency | Acceptance Criteria |
|--|---|--|
| Method Blank | Prepare a method blank with each extraction batch. Must be spiked with EIS and subjected to prep procedure. | 1633 – must be 1) less than the ML for any/all analytes; 2) less than 1/3 any regulatory compliance limits; or 3) < 1/10 the concentration found in any sample within the prep batch. DOD – Must be 1) less than ½ the MRL/LOQ, or 2) < 1/10 th the concentration found in any sample in the prep batch, or 3) < 1/10 th the regulatory limit, which ever of the three concentrations is greater. |
| Laboratory Fortified Sample Matrix (LFSM) | Include one LFSM per Extraction Batch. Fortify the LFSM with method analytes at a concentration close to but greater than the native concentrations (if known), or near the mid-point of the curve. Not required for method 1633. | DOD – recoveries must be within the LCS limits – 40-150% (or in-house limits), if project limits are not provided. |
| Laboratory Fortified Sample Matrix Duplicate (LFSMD) or Field Duplicate (FD) | Include at least one LFSMD or FD with each Extraction Batch. Not required for method 1633. | DOD – recoveries must be within the LCS limits – 40-150% (or in-house limits), if project limits are not provided. % RSD must be </= 30%. |



24.7 EPA Draft 1633 Table 10 – NIS Recoveries

Table 10. Range of Recoveries for Non-Extracted Internal Standards in the Single-laboratory Validation Study, by Matrix

| NIS Compounds | Aqueous | | | Solid | | | Tissue | | |
|-------------------------------------|------------|-----|---------|------------|-----|---------|------------|-----|---------|
| | % Recovery | | RSD (%) | % Recovery | | RSD (%) | % Recovery | | RSD (%) |
| | Min | Max | | Min | Max | | Min | Max | |
| ¹³ C ₃ -PFBA | 60 | 91 | 10.3 | 54 | 89 | 6.4 | 51 | 82 | 7.0 |
| ¹³ C ₂ -PFHxA | 43 | 94 | 18.6 | 52 | 90 | 7.4 | 41 | 80 | 19.3 |
| ¹³ C ₄ -PFOA | 59 | 87 | 9.7 | 54 | 89 | 6.4 | 51 | 82 | 9.5 |
| ¹³ C ₅ -PFNA | 64 | 87 | 7.5 | 59 | 94 | 7.1 | 52 | 88 | 11.2 |
| ¹³ C ₇ -PFDA | 57 | 86 | 10.0 | 55 | 91 | 8.6 | 47 | 85 | 19.4 |
| ¹⁸ O ₂ -PFHxS | 59 | 87 | 9.6 | 53 | 87 | 7.1 | 51 | 80 | 8.1 |
| ¹³ C ₄ -PFOS | 60 | 82 | 7.5 | 58 | 86 | 7.0 | 52 | 85 | 10.3 |

Data for this table are derived from the single-laboratory validation study, and are only provided as examples for this draft method. The data will be updated with the interlaboratory study results in a subsequent revision.



24.8 EPA Draft 1633 Table 9 – EIS Recoveries

Table 9. Range of Recoveries for Extracted Internal Standards (EIS) in the Single-laboratory Validation Study, by Matrix

| EIS Compounds | Aqueous | | | Solid | | | Tissue | | |
|---------------------------------------|------------|-----|---------|------------|-----|---------|------------|-----|---------|
| | % Recovery | | RSD (%) | % Recovery | | RSD (%) | % Recovery | | RSD (%) |
| | Min | Max | | Min | Max | | Min | Max | |
| ¹³ C ₄ -PFBA | 9 | 97 | 15.9 | 3 | 113 | 37.4 | 84 | 99 | 8.0 |
| ¹³ C ₅ -PFPeA | 39 | 103 | 13.3 | 28 | 112 | 17.2 | 86 | 107 | 11.1 |
| ¹³ C ₅ -PFHxA | 73 | 97 | 2.7 | 79 | 110 | 5.5 | 92 | 95 | 1.6 |
| ¹³ C ₄ -PFHpA | 77 | 95 | 2.4 | 73 | 111 | 6.0 | 80 | 93 | 8.2 |
| ¹³ C ₈ -PFOA | 87 | 95 | 0.8 | 86 | 115 | 4.4 | 90 | 95 | 2.8 |
| ¹³ C ₉ -PFNA | 82 | 95 | 1.6 | 87 | 110 | 4.2 | 90 | 98 | 4.3 |
| ¹³ C ₆ -PFDA | 71 | 93 | 3.3 | 87 | 112 | 4.9 | 83 | 97 | 7.7 |
| ¹³ C ₇ -PFUnA | 56 | 94 | 6.5 | 66 | 124 | 11.6 | 71 | 91 | 12.9 |
| ¹³ C ₂ -PFDoA | 34 | 87 | 13.7 | 26 | 109 | 24.3 | 54 | 96 | 29.2 |
| ¹³ C ₂ -PFTeDA | 17 | 153 | 26.2 | 18 | 110 | 30.1 | 31 | 102 | 67.8 |
| ¹³ C ₃ -PFBS | 72 | 100 | 4.7 | 89 | 120 | 5.4 | 89 | 98 | 5.1 |
| ¹³ C ₃ -PFHxS | 79 | 95 | 1.6 | 87 | 110 | 4.4 | 98 | 99 | 0.1 |
| ¹³ C ₈ -PFOS | 67 | 96 | 3.6 | 79 | 113 | 5.7 | 92 | 103 | 6.0 |
| ¹³ C ₂ -4:2FTS | 81 | 199 | 14.8 | 95 | 248 | 17.0 | 192 | 215 | 6.2 |
| ¹³ C ₂ -6:2FTS | 64 | 183 | 16.4 | 76 | 127 | 9.4 | 145 | 230 | 27.2 |
| ¹³ C ₂ -8:2FTS | 65 | 139 | 8.4 | 86 | 173 | 15.2 | 136 | 220 | 24.6 |
| ¹³ C ₈ -PFOSA | 27 | 93 | 15.4 | 61 | 123 | 10.0 | 87 | 96 | 4.5 |
| D ₃ -NMeFOSA | 14 | 74 | 16.4 | 28 | 86 | 22.7 | 8 | 38 | 61.9 |
| D ₃ -NEtFOSA | 12 | 70 | 16.5 | 21 | 70 | 25.5 | 8 | 30 | 57.8 |
| D ₃ -NMeFOSAA | 21 | 113 | 7.3 | 52 | 142 | 14.8 | 106 | 139 | 13.1 |
| D ₃ -NEtFOSAA | 12 | 106 | 8.2 | 68 | 151 | 16.9 | 79 | 151 | 31.8 |
| D ₇ -NMeFOSE | 11 | 77 | 18.6 | 13 | 107 | 27.9 | 5 | 30 | 81.1 |
| D ₉ -NEtFOSE | 8 | 73 | 19.6 | 16 | 97 | 30.4 | 0 | 29 | 103.1 |
| ¹³ C ₃ -HFPO-DA | 92 | 113 | 2.0 | 70 | 119 | 10.4 | 93 | 102 | 5.1 |

Data for this table are derived from the single-laboratory validation study, and are only provided as examples for this draft method. The data will be updated with the interlaboratory study results in a subsequent revision.



24.9 EPA Draft 1633 Table 5 – OPR and IPR Criteria

Table 5. Single-Laboratory Validation Performance Summary for Target Compounds and Extracted Internal Standards

| Compounds | Blank (ng/mL) | Aqueous Matrices ¹ | | | Solid Matrices ¹ | | | Tissue Matrices ¹ | | |
|--|---------------|-------------------------------|---------|-------------|-----------------------------|---------|-------------|------------------------------|---------|-------------|
| | | IPR Rec (%) | RSD (%) | OPR Rec (%) | IPR Rec (%) | RSD (%) | OPR Rec (%) | IPR Rec (%) | RSD (%) | OPR Rec (%) |
| Extracted Internal Standard (EIS) | | | | | | | | | | |
| ¹³ C ₄ -PFBA | N/A | 85 - 91 | 1.6 | 88 - 108 | 92 - 99 | 1.6 | 95 - 109 | 93 - 97 | 1.0 | 95 - 105 |
| ¹³ C ₃ -PFPeA | N/A | 87 - 95 | 2.4 | 84 - 111 | 86 - 106 | 5.3 | 80 - 110 | 85 - 108 | 6.0 | 89 - 103 |
| ¹³ C ₃ -PFHxA | N/A | 85 - 92 | 1.9 | 83 - 108 | 83 - 101 | 4.8 | 92 - 106 | 79 - 111 | 8.5 | 88 - 98 |
| ¹³ C ₄ -PFHpA | N/A | 78 - 100 | 6.2 | 83 - 106 | 87 - 102 | 4.1 | 90 - 100 | 88 - 93 | 1.3 | 80 - 102 |
| ¹³ C ₈ -PFOA | N/A | 77 - 98 | 6.0 | 84 - 107 | 89 - 101 | 3.2 | 92 - 104 | 91 - 98 | 1.7 | 86 - 102 |
| ¹³ C ₉ -PFNA | N/A | 82 - 96 | 3.8 | 84 - 107 | 86 - 101 | 4.1 | 90 - 106 | 91 - 104 | 3.3 | 89 - 101 |
| ¹³ C ₆ -PFDA | N/A | 81 - 98 | 4.7 | 84 - 106 | 79 - 101 | 6.0 | 86 - 109 | 89 - 104 | 4.0 | 90 - 104 |
| ¹³ C ₇ -PFUnA | N/A | 84 - 100 | 4.4 | 84 - 109 | 84 - 104 | 5.4 | 91 - 116 | 84 - 118 | 8.4 | 88 - 109 |
| ¹³ C ₂ -PFDoA | N/A | 61 - 103 | 12.9 | 73 - 101 | 70 - 93 | 7.1 | 73 - 106 | 95 - 125 | 6.8 | 70 - 108 |
| ¹³ C ₂ -PFTeDA | N/A | 72 - 89 | 5.4 | 74 - 97 | 83 - 88 | 1.5 | 74 - 107 | 81 - 114 | 8.5 | 10 - 110 |
| ¹³ C ₃ -PFBS | N/A | 87 - 94 | 2.0 | 88 - 110 | 97 - 105 | 1.8 | 96 - 109 | 87 - 114 | 6.5 | 95 - 106 |
| ¹³ C ₃ -PFHxS | N/A | 83 - 89 | 1.9 | 85 - 103 | 92 - 97 | 1.4 | 92 - 106 | 92 - 97 | 1.4 | 91 - 103 |
| ¹³ C ₈ -PFOS | N/A | 78 - 92 | 3.9 | 86 - 110 | 87 - 107 | 4.9 | 95 - 109 | 87 - 93 | 1.6 | 95 - 103 |
| ¹³ C ₂ -4:2FTS | N/A | 64 - 106 | 12.1 | 87 - 137 | 132 - 135 | 0.6 | 123 - 145 | 106 - 221 | 17.6 | 155 - 291 |
| ¹³ C ₂ -6:2FTS | N/A | 93 - 102 | 2.2 | 67 - 149 | 118 - 129 | 2.3 | 104 - 138 | 87 - 135 | 10.8 | 117 - 149 |
| ¹³ C ₂ -8:2FTS | N/A | 99 - 109 | 2.5 | 71 - 137 | 96 - 122 | 6.1 | 93 - 123 | 179 - 299 | 12.5 | 79 - 304 |
| ¹³ C ₈ -PFOSA | N/A | 60 - 107 | 14.2 | 57 - 109 | 69 - 86 | 5.4 | 66 - 100 | 104 - 153 | 9.4 | 88 - 120 |
| D ₃ -NMeFOSA | N/A | 55 - 85 | 10.8 | 39 - 84 | 47 - 59 | 5.4 | 25 - 64 | 20 - 58 | 24.5 | 3 - 34 |
| D ₅ -NEtFOSA | N/A | 54 - 91 | 12.9 | 43 - 84 | 43 - 51 | 4.5 | 18 - 58 | 30 - 56 | 15.2 | 0 - 56* |
| D ₃ -NMeFOSAA | N/A | 63 - 117 | 14.9 | 66 - 117 | 98 - 107 | 2.1 | 86 - 109 | 102 - 187 | 14.7 | 144 - 196 |
| D ₅ -NEtFOSAA | N/A | 66 - 115 | 13.7 | 63 - 115 | 98 - 104 | 1.3 | 85 - 109 | 178 - 216 | 4.9 | 175 - 223 |
| D ₇ -NMeFOSE | N/A | 61 - 106 | 13.6 | 42 - 99 | 50 - 61 | 5.1 | 35 - 76 | 3 - 5 | 11.6 | 0 - 8* |
| D ₉ -NEtFOSE | N/A | 63 - 108 | 13.2 | 44 - 90 | 46 - 57 | 5.5 | 32 - 72 | 8 - 33 | 30.0 | 0 - 33* |
| ¹³ C ₃ -HFPO-DA | N/A | 89 - 106 | 4.5 | 88 - 121 | 98 - 108 | 2.4 | 83 - 125 | 87 - 106 | 4.9 | 81 - 106 |

¹ The recovery limits are applied to all samples, method blanks, IPR, OPR samples for all matrix types.

* Ranges were determined at ± 2 standard deviations from the mean. Because of the low recoveries for these EIS, the calculated lower limits were negative values. Therefore, the lower limits have been set to 0 for these analytes.

Data for this table are derived from the single-laboratory validation study, and are only provided as examples for this draft method. The data will be updated to reflect the interlaboratory study results in a subsequent revision. Therefore, these criteria will change after interlaboratory validation. Several sections of this method state that Table 5 criteria are required, this is standard language that will be applicable when the method is finalized.



24.10 DOD/DOE QSM 5.4, Appendix B QC Requirements: Table B-15

| Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water | | | | | |
|---|--|--|--------------------------|--------------------------|---|
| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
| Aqueous Sample Preparation | Each sample and associated batch QC samples. | <p>Solid Phase Extraction (SPE) must be used unless samples are known to contain high PFAS concentrations (e.g., Aqueous Film Forming Foam (AFFF) formulations). Inline SPE is acceptable.</p> <p>Entire sample plus bottle rinsate must be extracted using SPE.</p> <p>Known high PFAS concentration samples require serial dilution be performed in duplicate.</p> <p>Documented project approval is needed for samples prepared by serial dilution as opposed to SPE.</p> | NA. | NA. | <p>Samples with > 1% solids may require centrifugation prior to SPE extraction.</p> <p>Pre-screening of separate aliquots of aqueous samples is recommended.</p> |
| Solid Sample Preparation | Each sample and associated batch QC samples. | Entire sample received by the laboratory must be homogenized prior to subsampling. | NA. | NA. | NA. |
| Biota Sample Preparation | Each sample and associated batch QC samples. | Sample prepared as defined by the project (e.g., whole fish versus filleted fish). | NA. | NA. | NA. |



Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|--|---|---|--|------------------------------|--|
| AFFF and AFFF Mixture Samples Preparation | Each sample and associated batch QC samples. | Each field sample must be prepared in duplicate (equivalent to matrix duplicate). Serial dilutions must be performed to achieve the lowest LOQ possible for each analyte. | NA. | NA. | Adsorption onto bottle is negligible compared to sample concentration so subsampling is allowed. Multiple dilutions will most likely have to be reported in order to achieve the lowest LOQ possible for each analyte. |
| Sample Cleanup Procedure | Each sample and associated batch QC samples. Not applicable to AFFF and AFFF Mixture Samples. | ENVI-Carb™ or equivalent must be used on each sample and batch QC sample. | NA. | Flagging is not appropriate. | Cleanup should reduce bias from matrix interferences. |
| Mass Calibration | Instrument must have a valid mass calibration prior to any sample analysis. Mass calibration is verified after each mass calibration, prior to initial calibration (ICAL). | Calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. Mass calibration range must bracket the ion masses of interest. The most recent mass calibration must be used for every acquisition in an analytical run. Mass calibration must be verified to be ± 0.5 amu of the true value, by acquiring a full scan continuum mass spectrum of a PFAS stock standard. | If the mass calibration fails, then recalibrate. If it fails again, consult manufacturer instructions on corrective maintenance. | Flagging is not appropriate. | Problem must be corrected. No samples may be analyzed under a failing mass calibration. The mass calibration is updated on an as-needed basis (e.g., QC failures, ion masses fall outside of the ± 0.5 amu of the true value, major instrument maintenance is performed, or the instrument is moved). |



Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|--|--|-------------------|------------------------------|---|
| Mass Spectral Acquisition Rate | Each analyte, Extracted Internal Standard (EIS) Analyte. | A minimum of 10 spectra scans are acquired across each chromatographic peak. | NA. | Flagging is not appropriate. | NA. |
| Calibration, Calibration Verification, and Spiking Standards | All analytes. | Standards containing both branched and linear isomers must be used when commercially available. PFAS method analytes may consist of both branched and linear isomers, but quantitative standards that contain the linear and branched isomers do not exist for all method analytes. For PFAS that do not have a quantitative branched and linear standard, identify the branched isomers by analyzing a qualitative standard that includes both linear and branched isomers and determine retention times, transitions and transition ion ratios. Quantitate samples by integrating the total response (i.e., accounting for peaks that are identified as linear and branched isomers) and relying on the initial calibration that uses the linear isomer quantitative standard. | NA. | Flagging is not appropriate. | Standards containing both branched and linear isomers are to be used during method validation and when reestablishing retention times, to ensure the total response is quantitated for that analyte. Technical grade standards cannot be used for quantitative analysis. |



Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|--|---|--|-------------------|---|--|
| <p>Sample PFAS Identification</p> | <p>All analytes detected in a sample.</p> | <p>The chemical derivation of the ion transitions must be documented. A minimum of two ion transitions (Precursor → quant ion and precursor → confirmation ion) and the ion transitions ratio per analyte are required for confirmation. Exception is made for analytes where two transitions do not exist (PFBA and PFPeA).</p> <p>Documentation of the primary and confirmation transitions and the ion ratio is required.</p> <p>In-house acceptance criteria for evaluation of ion ratios must be used and must not exceed 50150%.</p> <p>Signal to Noise Ratio (S/N) must be ≥ 10 for all ions used for quantification and must be ≥ 3 for all ions used for confirmation.</p> <p>Quant ion and confirmation ion must be present and must maximize simultaneously (± 2 seconds).</p> | <p>NA.</p> | <p>PFAS identified with Ion ratios that fail acceptance criteria must be flagged.</p> <p>Any quantitation ion peak that does not meet the maximization criteria shall be included in the summed integration and the resulting data flagged as “estimated, biased high”.</p> | <p>For example: Ion Ratio = (quant ion abundance/ confirm ion abundance)</p> <p>Calculate the average ratio (A) and standard deviation (SD) using the ICAL standards. An acceptance range of ratio could be within $A \pm 3SD$ for confirmation of detection.</p> |



Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|--|---|--|-------------------|-----------------------------|----------|
| Ion Transitions (Precursor->Product) | Every field sample, standard, blank, and QC sample. | <p>In order to avoid biasing results high due to known interferences for some transitions, the following transitions must be used for the quantification of the following analytes:</p> <p>PFOA: 413 → 369 PFOS: 499 → 80 PFHxS: 399 → 80 PFBS: 299 → 80 4:2 FTS: 327 → 307 6:2 FTS: 427 → 407 8:2 FTS: 527 → 507 NEtFOSAA: 584 → 419 NMeFOSAA: 570 → 419</p> <p>If these transitions are not used, the reason must be technically justified and documented (e.g., alternate transition was used due to observed interferences).</p> | NA. | Flagging is not appropriate | NA. |



Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|-----------------------------------|--|---|------------------------------------|------------------------------|---|
| Initial Calibration (ICAL) | At instrument set-up and after ICV or CCV failure, prior to sample analysis. | <p>The isotopically labeled analog of an analyte (Extracted Internal Standard Analyte) must be used for quantitation if commercially available (Isotope Dilution Quantitation).</p> <p>Commercial PFAS standards available as salts are acceptable providing the measured mass is corrected to the neutral acid concentration. Results shall be reported as the neutral acid with appropriate CAS number.</p> <p>If a labeled analog is not commercially available, the Extracted Internal Standard Analyte with the closest retention time or chemical similarity to the analyte must be used for quantitation. (Internal Standard Quantitation)</p> <p>Analytes must be within 70-130% of their true value for each calibration standard.</p> <p><i>(continued next page)</i></p> | Correct problem, then repeat ICAL. | Flagging is not appropriate. | <p>No samples shall be analyzed until ICAL has passed.</p> <p>External Calibration is not allowed for any analyte.</p> <p>Calibration can be linear (minimum of 5 standards) or quadratic (minimum of 6 standards); weighting is allowed.</p> |



Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|--|--|--|-------------------|--------------------------------------|
| Initial Calibration (ICAL) <i>(Continued)</i> | | ICAL must meet one of the two options below: Option 1: The RSD of the RFs for all analytes must be $\leq 20\%$. Option 2: Linear or non-linear calibrations must have $r^2 \geq 0.99$ for each analyte. | | | |
| Retention Time window position establishment | Once per ICAL and at the beginning of the analytical sequence. | Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used. | NA. | NA. | Calculated for each analyte and EIS. |
| Retention Time (RT) window width | Every field sample, standard, blank, and QC sample. | RT of each analyte and EIS analyte must fall within 0.4 minutes of the predicted retention times from the daily calibration verification or, on days when ICAL is performed, from the midpoint standard of the ICAL. Analytes must elute within 0.1 minutes of the associated EIS. This criterion applies only to analyte and labeled analog pairs. | Correct problem and reanalyze samples. | NA. | Calculated for each analyte and EIS. |



Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|--|--|--|--|--|--|
| Instrument Sensitivity Check (ISC) | Prior to analysis and at least once every 12 hours. | Analyte concentrations must be at LOQ; concentrations must be within $\pm 30\%$ of their true values. | Correct problem, rerun ISC. If problem persists, repeat ICAL. | Flagging is not appropriate. | No samples shall be analyzed until ISC has met acceptance criteria. ISC can serve as the initial daily CCV. |
| Initial Calibration Verification (ICV) | Once after each ICAL, analysis of a second source standard prior to sample analysis. | Analyte concentrations must be within $\pm 30\%$ of their true value. | Correct problem, rerun ICV. If problem persists, repeat ICAL. | Flagging is not appropriate. | No samples shall be analyzed until calibration has been verified. |
| Continuing Calibration Verification (CCV) | Prior to sample analysis, after every 10 field samples, and at the end of the analytical sequence. | Concentration of analytes must range from the LOQ to the mid-level calibration concentration. Analyte concentrations must be within $\pm 30\%$ of their true value. | Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV. | If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification. | Results may not be reported without valid CCVs. Instrument Sensitivity Check (ISC) can serve as a bracketing CCV. |



Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|--------------------------|---|---|---|---|--|
| Instrument Blanks | Immediately following the highest standard analyzed and daily prior to sample analysis. | Concentration of each analyte must be $\leq \frac{1}{2}$ the LOQ. Instrument Blank must contain EIS to enable quantitation of contamination. | If acceptance criteria are not met after the highest calibration standard, calibration must be performed using a lower concentration for the highest standard until acceptance criteria is met. If sample concentrations exceed the highest allowed standard and the sample(s) following exceed this acceptance criteria ($>1/2$ LOQ), they must be reanalyzed. | Flagging is only appropriate in cases when the sample cannot be reanalyzed and when there is no more sample left. | No samples shall be analyzed until instrument blank has met acceptance criteria. Note: Successful analysis following the highest standard analyzed determines the highest concentration that carryover does not occur. When the highest standard analyzed is not part of the calibration curve, it cannot be used to extend out the calibration range, it is used only to document a higher concentration at which carryover still does not occur. |



Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---|---|---|--|--|--|
| Extracted Internal Standard (EIS) Analytes | Every field sample, standard, blank, and QC sample. | <p>Added to solid sample prior to extraction. Added to aqueous samples, into the original container, prior to extraction.</p> <p>For aqueous samples prepared by serial dilution instead of SPE, added to final dilution of samples prior to analysis.</p> <p>Extracted Internal Standard Analyte recoveries must be within 50% to 150% of ICAL midpoint standard area or area measured in the initial CCV on days when an ICAL is not performed.</p> | <p>Correct problem. If required, re-extract and reanalyze associated field and QC samples.</p> <p>If recoveries are acceptable for QC samples, but not field samples, the field samples must be re-extracted and analyzed (greater dilution may be needed).</p> <p>Samples may be reextracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure.</p> | Apply Q-flag and discuss in the Case Narrative only if reanalysis confirms failures in exactly the same manner. | <p>Failing analytes shall be thoroughly documented in the Case Narrative.</p> <p>EIS should be 96% (or greater) purity. When the impurity consists of the unlabeled analyte, the EIS can result in a background artifact in every sample, standard and blank, if the EIS is fortified at excessive concentrations.</p> |
| Method Blank (MB) | One per preparatory batch. | No analytes detected $> \frac{1}{2}$ LOQ or $> 1/10^{\text{th}}$ the amount measured in any sample or $1/10^{\text{th}}$ the regulatory limit, whichever is greater. | <p>Correct problem. If required, re-extract and reanalyze MB and all QC samples and field samples processed with the contaminated blank.</p> <p>Samples may be reextracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure.</p> <p>Examine the project specific requirements. Contact the client as to additional measures to be taken.</p> | <p>If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative.</p> <p>Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.</p> | <p>Results may not be reported without a valid MB.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p> |



Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|--|---|--|--|--|--|
| Laboratory Control Sample (LCS) | One per preparatory batch. | <p>Blank spiked with all analytes at a concentration \geq LOQ and \leq the mid-level calibration concentration.</p> <p>A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p> | <p>Correct problem, then reextract and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available.</p> <p>Samples may be reextracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure.</p> <p>Examine the projectspecific requirements. Contact the client as to additional measures to be taken.</p> | <p>If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative.</p> <p>Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.</p> | <p>Results may not be reported without a valid LCS.</p> <p>Flagging is only appropriate in cases where the samples cannot be reanalyzed.</p> |
| Matrix Spike (MS) | One per preparatory batch. Not required for aqueous samples prepared by serial dilution instead of SPE. | <p>Sample spiked with all analytes at a concentration \geq LOQ and \leq the mid-level calibration concentration.</p> <p>A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified.</p> <p>If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.</p> | <p>Examine the project-specific requirements. Contact the client as to additional measures to be taken.</p> | <p>For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.</p> | <p>For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).</p> |



Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|--|--|---|--|--|--|
| Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD) | For MSD: One per preparatory batch. For MD: Each aqueous sample prepared by serial dilution instead of SPE. | For MSD: Sample spiked with all analytes at a concentration \geq LOQ and \leq the mid-level calibration concentration. A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD \leq 30% (between MS and MSD or sample and MD). | Examine the project-specific requirements. Contact the client as to additional measures to be taken. | For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative. | The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is \geq LOQ. The MD is a second aliquot of the field sample that has been prepared by serial dilution. |
| Post Spike Sample | Only applies to aqueous samples prepared by serial dilution instead of SPE that have reported value of $<$ LOQ for analyte(s). | Spike all analytes reported as $<$ LOQ into the dilution that the result for that analyte is reported from. The spike must be at the LOQ concentration to be reported for this sample as $<$ LOQ. When analyte concentrations are calculated as $<$ LOQ, the post spike for that analyte must recover within 70-130% of its true value. | When analyte concentrations are calculated as $<$ LOQ, and the spike recovery does not meet the acceptance criteria, the sample, sample duplicate, and post spike sample must be reanalyzed at consecutively higher dilutions until the criteria is met. | Flagging is not appropriate. | When analyte concentrations are calculated as $<$ LOQ, results may not be reported without acceptable post spike recoveries. |



24.11 DOD/DOE QSM 5.4, Appendix B QC Requirements: Table B-24

Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by LC/MS/MS (EPA Draft Method 1633)

****ALL OF THE REQUIREMENTS CONTAINED IN EPA DRAFT METHOD 1633 MUST BE MET. This table contains additional requirements that must be met. Where the name for the QC sample listed in this table differs from EPA Draft Method 1633 terminology, the corresponding EPA Draft Method 1633 terminology is provided in the Comments column.****

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | Comments |
|---------------------|---|--|-------------------|-------------------|--|
| AFFF samples | Each AFFF sample. Note: This does not include AFFF samples that are to be evaluated for MIL-PRF-14385 compliance. Those AFFF samples must be performed in compliance with DoD AFFF01, not EPA Draft Method 1633. | AFFF samples must be subsampled in duplicate for analysis in accordance with DoD AFFF01, Section 11.2.1 through 11.2.9. Note: In lieu of the LCSD required in Section 11.2.6 of DoD AFFF01, one MS/MSD pair must be prepared with each batch of AFFF samples. All AFFF samples must be processed in duplicate in the same manner as whole sample aqueous samples (SPE, carbon cleanup) per EPA Draft Method 1633. | NA. | NA. | A copy of the latest version of DoD AFFF can be found at https://denix.osd.mil/edqw/ |



Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by LC/MS/MS (EPA Draft Method 1633)

| | | | | | |
|---|---|--|-----|---|---|
| Ion Transitions (Precursor-> Product) | Every field sample, standard, blank, and QC sample. | In addition to the requirements of EPA Draft Method 1633, the following must be met: 1) If a qualitative or quantitative standard containing an isomeric mixture (branched and linear isomers) of an analyte is commercially available for an analyte, the quantification ion used must be the quantification ion identified in Table 2 of EPA Draft Method 1633 unless interferences render the product ion unusable as the quantification ion. 2) In cases where interferences render the product ion unusable as the quantification ion, project approval is required before using the alternative product ion. | NA. | Flagging is not appropriate. Provide technical justification in the Case Narrative. | Currently, qualitative or quantitative standards containing isomeric mixtures for an analyte are commercially available for PFOA, PFOS, PFHxS, NMeFOSAA, NEtFOSAA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE. |
|---|---|--|-----|---|---|



Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by LC/MS/MS (EPA Draft Method 1633)

| | | | | | |
|---|--|--|---|--|--|
| Ion Ratio | All analytes detected in a sample. | Must meet all of the requirements of EPA Draft Method 1633. | Must meet all of the requirements of EPA Draft Method 1633. | Document and discuss the failure in the Case Narrative. Apply I-flag to the result associated with the failure. | |
| Instrument Sensitivity Check (ISC) | Daily. At the beginning of each analytical sequence, prior to sample analysis. | In addition to the requirements of EPA Draft Method 1633, the following must be met: All analyte concentrations must be within $\pm 30\%$ of their true values. | Correct problem, rerun ISC. If problem persists, repeat ICAL. | Flagging is not appropriate. | No samples shall be analyzed until acceptance criteria for ISC has been met. |
| Initial Calibration Verification (ICV) | Once after each ICAL, prior to sample analysis. | Must be made from a second source standard. All analyte concentrations must be within $\pm 30\%$ of their true values. | Correct problem, rerun ICV. If problem persists, repeat ICAL. | Flagging is not appropriate. | No samples shall be analyzed until acceptance criteria for ICV has been met. |



Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by LC/MS/MS (EPA Draft Method 1633)

| | | | | | |
|------------------------------|--|---|--|---|---|
| Instrument Blank (IB) | Immediately following the highest standard analyzed in the calibration, daily prior to analyzing standards, after each CCV, and immediately following samples with PFAS concentrations exceeding the quantification range. | In addition to the requirements of EPA Draft Method 1633, the following must be met: Concentration of each analyte must be $\leq \frac{1}{2}$ the LOQ. | If acceptance criteria are not met after the highest calibration standard, calibration must be performed using a lower concentration for the highest standard until acceptance criteria is met. If sample concentrations exceed the highest calibration standard and the sample(s) following exceed this acceptance criteria ($> 1/2$ LOQ), they must be reanalyzed using a fresh aliquot of the sample extract. | Flagging is only appropriate in cases where the extract cannot be reanalyzed and re-extraction is not possible. | EPA Draft Method 1633 equivalent to the CCV is the Calibration Verification (CV). |
|------------------------------|--|---|--|---|---|



Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by LC/MS/MS (EPA Draft Method 1633)

| | | | | | |
|---|--|--|---|---|--|
| <p>Extracted Internal Standard (EIS) Compounds</p> | <p>Every field sample, standard, blank, and QC sample.</p> | <p>In addition to the requirements of EPA Draft Method 1633, the following must be met:</p> <ol style="list-style-type: none"> 1) Isotopically labeled analogs of analytes must be used when they are commercially available. 2) QC samples and field samples must recover within in-house limits if project limits are not provided; otherwise, project limits must be met. Preliminary inhouse acceptance criteria of 20-150% must be used until inhouse limits are generated in accordance with Sections 9.4.1 and 9.4.2 of EPA Draft Method 1633. 3) The lower limit of inhouse acceptance criteria cannot be < 20%. | <p>Repeat the analysis using a fresh aliquot of the extract. If failure does not confirm, report the second analysis. If the failure confirms, follow the requirements listed in EPA Draft Method 1633, Section 15.3.2. If EIS recoveries still fall outside of the acceptance range, the client must be contacted for additional measures to be taken.</p> | <p>Document and discuss the failure in the Case Narrative.</p> <p>Apply Q-flag to the result associated with the failure.</p> | |
|---|--|--|---|---|--|



Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by LC/MS/MS (EPA Draft Method 1633)

| | | | | | |
|--|---|--|--|--|--|
| Non-extracted Internal Standard (NIS) Compounds | Every field sample, standard, blank, and QC sample. | In addition to the requirements of EPA Draft Method 1633, the following must be met: 1) NIS areas must be greater than 30% of the average area of the calibration standards in undiluted sample extracts and sample extracts that required additional NIS to be added. 2) NIS areas corrected for the dilution factor must be greater than 30% of the average area of the calibration standards in diluted samples when additional NIS was not added post dilution of the extract. | Repeat the analysis using a fresh aliquot of the extract. If failure does not confirm, report the second analysis. If the failure confirms, examine the project-specific requirements. Contact the client as to additional measures to be taken. | Document and discuss the failure in the Case Narrative. Apply Q-flag to the result associated with the failure. | |
|--|---|--|--|--|--|



Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by LC/MS/MS (EPA Draft Method 1633)

| | | | | | |
|-------------------------------------|--|---|---|--|---|
| <p>Method Blank (MB)</p> | <p>One per preparatory batch</p> | <p>In addition to the requirements of EPA Draft Method 1633, the following must be met:</p> <p>No analytes detected > ½ LOQ or > 1/10th the amount measured in any associated sample or 1/10th the regulatory limit, whichever is greater</p> | <p>Correct the problem. If required, re-extract and reanalyze MB and all QC samples and field samples processed with the contaminated blank. Samples may be reextracted and analyzed outside of holding times, as necessary for corrective action associated with QC failure.</p> <p>Examine the project specific requirements. Contact the client as to additional measures to be taken.</p> | <p>If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative.</p> <p>Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.</p> | |
| <p>Matrix Duplicate (MD)</p> | <p>Each AFFF sample prepared using an aliquot of the field sample must be prepared in duplicate.</p> | <p>In addition to the requirements of EPA Draft Method 1633, the following must be met:</p> <p>RPD ≤ 30% (between sample and MD)</p> | <p>Examine the project specific requirements. Contact the client as to additional measures to be taken. If the analyte(s) are not listed, use inhouse LCS limits if project limits are not specified.</p> | <p>For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.</p> | <p>The data shall be evaluated to determine the source of difference.</p> <p>For Sample/MD: RPD criteria only applies to analytes whose concentration in the sample is ≥ LOQ.</p> |



Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by LC/MS/MS (EPA Draft Method 1633)

| | | | | | |
|----------------------------|--|---|-----|-----|--|
| Bile Salt Standards | Daily, prior to analysis of all matrix types (aqueous, solid, tissue, and AFFF). | All EPA Draft Method 1633 requirements for evaluation of the relationship of the retention time of the bile salt peak(s) to the retention time window of PFOS must be met for all matrix types. The retention time window of PFOS applies to the retention time of all isomers of PFOS. The retention time of the bile salt(s) peak must fall out of the retention time window of PFOS by at least one minute. | NA. | NA. | No samples shall be analyzed until acceptance criteria for the bile salt standard(s) has been met. |
|----------------------------|--|---|-----|-----|--|



Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by LC/MS/MS (EPA Draft Method 1633)

| | | | | | |
|---|---------------------------------------|---|---|--|---|
| <p>Laboratory Control Sample (LCS) and Low-Level Laboratory Control Sample (LLLCS)</p> | <p>One set per preparatory batch.</p> | <p>In addition to the requirements of EPA Draft Method 1633 the following must be met:</p> <p>1) Analyte recoveries must be within in-house limits if project limits are not provided; otherwise, project limits must be met. Preliminary inhouse acceptance criteria of 40-150% must be used until inhouse limits are generated in accordance with Section 14.5.4 of EPA Draft Method 1633.</p> <p>2) The lower limit of inhouse acceptance criteria cannot be < 40%.</p> | <p>In addition to the requirements of EPA Draft Method 1633, the following must be met:</p> <p>Samples may be reextracted and analyzed outside of holding times, as necessary for corrective action associated with QC failure.</p> <p>Examine the project specific requirements. Contact the client as to additional measures to be taken.</p> | <p>If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative.</p> <p>Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.</p> | <p>EPA Draft Method 1633 equivalent to the LCS is the Ongoing Precision and Recovery Standard (OPR).</p> <p>EPA Draft Method 1633 equivalent to the LLLCS is Low-Level Ongoing Precision and Recovery Standard (LLOPR).</p> |
|---|---------------------------------------|---|---|--|---|

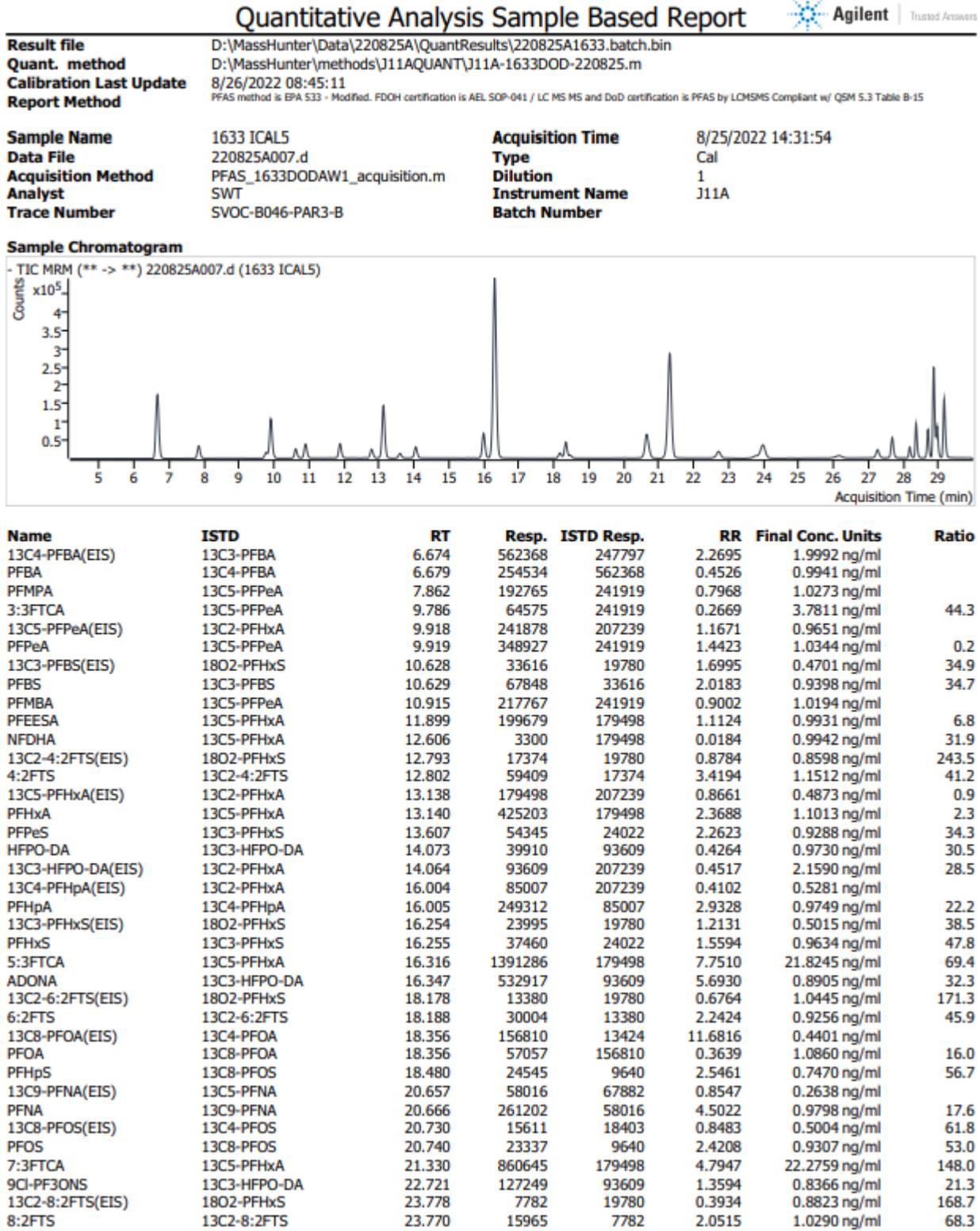


Table B-24. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by LC/MS/MS (EPA Draft Method 1633)

| | | | | | |
|---|--|---|--|---|--|
| Matrix Spike (MS) and Matrix Spike Duplicate (MSD) | One MS/MSD pair per preparatory batch. | In addition to the requirements of EPA Draft Method 1633, the following must be met: Analyte recoveries must be within in-house LCS limits if project limits are not provided; otherwise, project limits must be met. RPD \leq 30% (between MS and MSD) | Examine the project-specific requirements. Contact the client as to additional measures to be taken. If the analyte(s) are not listed, use inhouse LCS limits if project limits are not specified. | For the specific analyte(s) in the parent sample apply J-flag if acceptance criteria are not met and explain in the Case Narrative. | The data shall be evaluated to determine the source of difference. |
|---|--|---|--|---|--|



24.12 Figure 1 – Example Chromatogram for Reagent Water Fortified with Method Analytes (Chromatogram Plot Report)



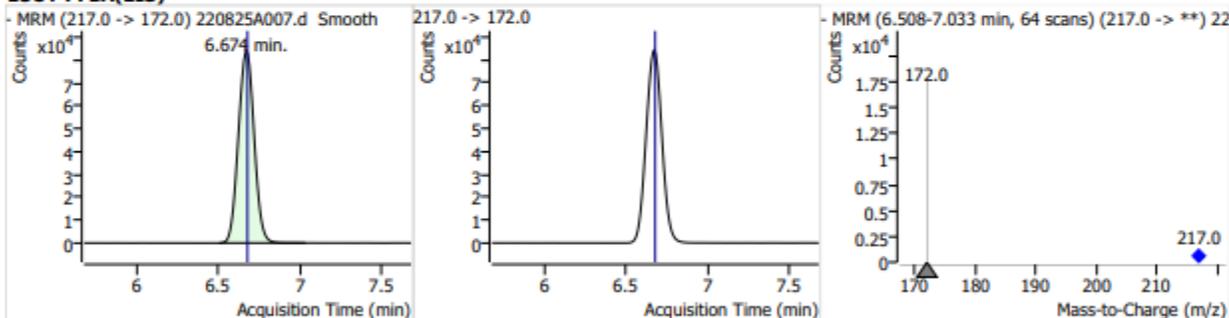


Quantitative Analysis Sample Based Report

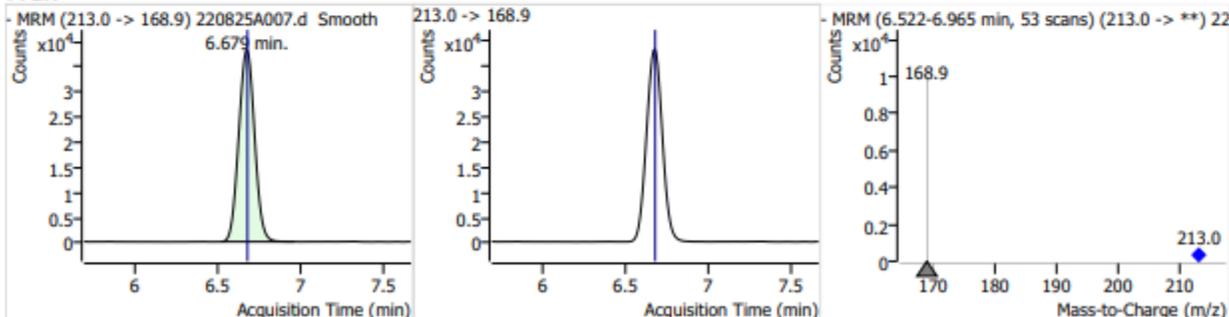


| Name | ISTD | RT | Resp. | ISTD Resp. | RR | Final Conc. Units | Ratio |
|-------------------|--------------|--------|--------|------------|---------|-------------------|-------|
| 13C6-PFDA(EIS) | 13C2-PFDA | 23.981 | 43499 | 49768 | 0.8740 | 0.2398 ng/ml | |
| PFDA | 13C6-PFDA | 23.990 | 210759 | 43497 | 4.8453 | 1.0423 ng/ml | 13.4 |
| PFNS | 13C8-PFOS | 24.027 | 28709 | 9640 | 2.9780 | 0.8297 ng/ml | 51.1 |
| N-Me-FOSAA | d3-N-MeFOSAA | 26.196 | 32564 | 37323 | 0.8725 | 1.0124 ng/ml | 39.0 |
| d3-N-MeFOSAA(EIS) | 13C4-PFOS | 26.119 | 37323 | 18403 | 2.0281 | 1.1080 ng/ml | |
| FOSA | M8FOSA | 27.273 | 89660 | 50818 | 1.7643 | 0.9551 ng/ml | 0.7 |
| M8FOSA(EIS) | 13C4-PFOS | 27.272 | 50819 | 18403 | 2.7615 | 0.5363 ng/ml | |
| PFDS | 13C8-PFOS | 27.671 | 31398 | 9640 | 3.2570 | 0.7785 ng/ml | 36.3 |
| 13C7-PFUnA(EIS) | 13C2-PFDA | 27.689 | 58539 | 49768 | 1.1762 | 0.2433 ng/ml | |
| PFUnA | 13C7-PFUnA | 27.681 | 78294 | 58539 | 1.3375 | 0.9584 ng/ml | 11.5 |
| N-Et-FOSAA | d5-N-EtFOSAA | 27.714 | 35762 | 34881 | 1.0253 | 1.0886 ng/ml | 69.1 |
| d5-N-EtFOSAA(EIS) | 13C4-PFOS | 27.688 | 34881 | 18403 | 1.8954 | 0.9580 ng/ml | |
| 11Cl-PF3OUdS | 13C3-HFPO-DA | 28.189 | 100608 | 93609 | 1.0748 | 0.8528 ng/ml | 13.6 |
| 13C2-PFDoA(EIS) | 13C2-PFDA | 28.366 | 52148 | 49768 | 1.0478 | 0.2202 ng/ml | |
| PFDoA | 13C2-PFDoA | 28.366 | 265680 | 52148 | 5.0947 | 0.9940 ng/ml | 14.0 |
| PFDoS | 13C8-PFOS | 28.674 | 18158 | 9640 | 1.8836 | 0.8794 ng/ml | 41.4 |
| PFTrDA | EIS Average | 28.708 | 252749 | 53822 | 4.6960 | 1.0347 ng/ml | 9.2 |
| N-Me-FOSE | d7-N-Me-FOSE | 28.878 | 681323 | 244053 | 2.7917 | 10.7578 ng/ml | |
| d7-N-Me-FOSE(EIS) | 13C4-PFOS | 28.852 | 244053 | 18403 | 13.2618 | 4.9259 ng/ml | |
| N-Me-FOSA | d3-N-Me-FOSA | 28.907 | 17126 | 9721 | 1.7618 | 0.8409 ng/ml | 175.6 |
| d3-N-Me-FOSA(EIS) | 13C4-PFOS | 28.898 | 9721 | 18403 | 0.5282 | 0.5318 ng/ml | |
| PFTeDA | M2PFTeDA | 28.967 | 227841 | 55497 | 4.1055 | 0.9028 ng/ml | 11.8 |
| M2PFTeDA(EIS) | 13C2-PFDA | 28.967 | 55496 | 49768 | 1.1151 | 0.2404 ng/ml | |
| N-Et-FOSE | d9-N-Et-FOSE | 29.170 | 391926 | 219223 | 1.7878 | 9.7107 ng/ml | |
| d9-N-Et-FOSE(EIS) | 13C4-PFOS | 29.153 | 219223 | 18403 | 11.9125 | 4.9663 ng/ml | |
| N-Et-FOSA | d5-N-Et-FOSA | 29.208 | 25852 | 9026 | 2.8642 | 1.0571 ng/ml | 125.9 |
| d5-N-Et-FOSA(EIS) | 13C4-PFOS | 29.199 | 9026 | 18403 | 0.4905 | 0.4836 ng/ml | |

13C4-PFBA(EIS)



PFBA





STANDARD OPERATING PROCEDURE

For
Method 9060A

Determination of Total Organic Carbon in Solid and Chemical Materials





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1.0 IDENTIFICATION OF TEST METHOD

- 1.1 This method is for the analysis of Total Organic Carbon (TOC) by method 9060A
- 1.2 Method modification for single analysis of calibration and QC rather than quadruplicate for the sample analysis.
- 1.3 Method deviates from the base method for preservation and hold time requirements. EPA 9060 states in instances where analysis cannot be performed within 2 hr from time of sampling, the sample is acidified (pH # 2) with HCl or H₂SO₄. The FDEP tables do not require preservation for Inorganic nonmetallic, except where noted and has a holding time range of 28 days for soils.

2.0 APPLICABLE MATRIX OR MATRICES

- 2.1 This method includes the measurement of organic carbon in solids, soils, and chemical materials.
- 2.2 The method is most applicable to measurement of organic carbon above 0.1 mg/g.

3.0 DETECTION LIMITS

- 3.1 For a list of the most current working MDLs , see the electronic version specific for the instrument and matrix MDLs in the "AEL-qa" folder on the lab server.
- 3.2 The detection limit is accomplished using the procedures listed in SOP Admin -012, which references 40CFR136, App. B.

4.0 SCOPE AND APPLICATION, INCLUDING COMPONENTS TO BE ANALYZED

- 4.1 Methods 9060A are used for the determination of total organic carbon (TOC) in solids, soils, and chemical materials.

5.0 SUMMARY OF THE TEST METHOD

- 5.1 Organic carbon in a sample is converted to carbon dioxide (CO₂) by combustion at 9000°C. The CO₂ formed can be measured directly by an infrared detector or converted to methane (CH₄) and measured by a flame ionization detector. The amount of CO₂ or CH₄ is directly proportional to the concentration of carbonaceous material in the sample.

6.0 DEFINITIONS

- 6.1 Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents within a 24hr period.
- 6.2 Standard: A solution prepared by diluting stock standard solutions used to calibrate the instrument response with respect to analyte concentrations.



- 6.3 Stock Standard: A concentrated solution containing method analytes that is purchased from a commercial source having Certificates of Analysis.
- 6.4 Primary Standard: A solution prepared in the laboratory from stock standards and diluted as needed to prepare calibration solutions and other needed analyte solutions.
- 6.5 Secondary Source Standard: A solution prepared in the laboratory from a second source standard and diluted as needed to prepare calibration verification solutions and other needed analyte solutions.
- 6.6 Calibration Standard (CAL): A solution prepared from the primary standard. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 6.7 Calibration Blank (BLK): A volume of reagent water or the same matrix as the calibration standards, analyte free. Analyzed as part of the calibration.
- 6.8 Initial Calibration Verification (ICV): Second Source standard analyzed after the initial calibration of an instrument in order to verify the initial calibration. If the response (or calculated concentration) is within $\pm 10\%$ then the ICV is considered valid.
- 6.9 Initial Calibration Blank (ICB): A volume of reagent water or the same matrix as the calibration standards, analyte free. Analyzed directly after the initial calibration verification.
- 6.10 Continuing Calibration Verification (CCV): A solution of the analyte used to evaluate the performance of the instrument system with respect to a defined set of criteria. The CCV is prepared with the primary source or a mid-level of the initial calibration.
- 6.11 Continuing Calibration Blank (CCB), also referred to as Instrument Blank (IB): A volume of water, analyte free, to verify instrument is free of contamination. Analyzed after every CCV.
- 6.12 Method Blank (MB): Treated exactly as a sample, including exposure to all equipment, acids, and internal standards. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, reagents or other materials.
- 6.13 Laboratory Control Sample (LCS): Known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate measurements.
- 6.14 Matrix Spike / Matrix Spike Duplicate (MS/MSD): Aliquots of an environmental sample to which known quantities of the method analyte is added in the laboratory. The MS/MSD are analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of the analyte in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD corrected for background concentrations.



- 6.15 Sample Duplicate (DUP): Separate aliquot of the sample carried through the complete preparation and analytical procedure used to evaluate precision. MSD is analyzed evaluated for precision.
- 6.16 Safety Data Sheets (SDS): Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire and reactivity data, including storage, spill and handling precautions.
- 6.17 Limit of Detection (LOD): LOD is **not** synonymous with MDL. LOD is an estimate of the minimum amount of a substance that an analytical process can reliably detect with a high level of confidence (99% Confidence, that is a false negative rate of 1%). An LOD is analyte, prep method, cleanup method, analysis method, and matrix specific and is laboratory dependent. The LOD is at the level of the MDL verifications. The LOD when used as the MDL verification must go through all the same processes that a sample will go through and be detected above instrument noise level.
- 6.18 Linear Dynamic Range (LDR) or linear calibration range (LCR): The concentration range over which the instrument response is linear.
- 6.19 Limits of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g. target analyte) that can be reported with a specific degree of confidence. It is also the lowest concentration that produces a quantitative result within specified limits of precision and bias. The LOQ can equal the PQL, but is not synonymous with the PQL.
- 6.20 Limits of Quantitation Verification (LOQV): LOQ Verifications are a spiked clean matrix sample that must go through all the same processes that regular samples will go through and be within the precision and bias acceptance criteria of the method. The LOQ verifications are spiked at the concentrations of the LOQ.
- 6.21 Method Detection Limit (MDL): An estimate of the minimum amount of a substance that an analytical process can readily detect. The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units.
- 6.22 Practical Quantitation Level (PQL): The lowest calibration standard or lowest quantitation level for the method and matrix. The concentration below which data is to be qualified as having less certainty. PQLs are at a concentration greater than that of the MDL.
- 6.23 Non-Conformity Form (NCF): Form which will be completed and processed for each QC failure or deviation from normal protocol that occurs outside the scope of normal operation as defined by the AEL Quality Manual, AEL SOP Admin-016, and Method SOP.



- 6.24 Case Narrative (CN): A case narrative is simply a means of describing exactly what transpired with the samples during the analytical process. Case narratives are required for variances that occur within a project.
- 6.25 Qualifier Codes
- 6.25.1 A - Value reported is the mean (average) of two or more determinations. This code shall be used if the results of two or more discrete and separate samples are averaged. These samples shall have been processed and analyzed (e.g. laboratory replicate samples, field duplicates, etc.) independently. Do not use this code if the data are the result of replicate analyses on the same sample aliquot, extract or digestate. Under most conditions, replicate values shall be reported as individual analyses.
- 6.25.2 I - The reported Value is between the laboratory method detection limit (MDL) and the laboratory practical quantitation limit (PQL).
- 6.25.3 K- Off scale low.
- 6.25.4 L- Off scale high. Use if reporting above the acceptable level of quantitation.
- 6.25.5 U- Indicates that a compound was analyzed for but not detected. The value associated with the qualifier will be the MDL.
- 6.25.6 V- Indicates that the analyte was detected in both the sample and the associated method blank. NOTE: The method blank value cannot be subtracted from the associated sample to give a result. The sample result will be reported as is with the "V" qualifier.
- 6.25.7 H - Value based on field kit determination; results may not be accurate. This code shall be used if a field-screening test (i.e. field gas chromatograph data, immunoassay, vendor-supplied field kit, etc.) was used to generate the value and the field kit or method has not been recognized by the Department as equivalent to laboratory methods.
- 6.25.8 O - Sampled, but analysis lost or not performed. Note: if reporting data to STORET, a numerical value must be entered. Such values are not meaningful and shall not be used.
- 6.25.9 Q - Sample held beyond the accepted holding time. This code shall be used if the value is derived from a sample that was prepared and/or analyzed AFTER the approved holding time restrictions for sample preparation and analysis.
- 6.25.10 Y - The laboratory analysis was from an unpreserved or improperly preserved sample. The data may not be accurate.
- 6.25.11 REJ - Data is rejected and should not be used. Some or all of the quality control data for the analyte were outside criteria, and the presence or absence of the analyte cannot be determined from the data.
- 6.25.12 NAI - Not analyzed due to interference
- 6.25.13 J - Estimated value; value not accurate. This code shall be used in the following instances:
- 6.25.13.1 "1" Surrogate recovery limits have been exceeded;
- 6.25.13.2 "2" No known quality control criteria exists for the component;
- 6.25.13.3 "3" The reported value failed to meet the established quality control criteria for either precision or accuracy;
- 6.25.13.4 "4" The sample matrix interfered with the ability to make any accurate determination; or



- 6.25.13.5 "5" The data is questionable because of improper laboratory or field protocols (e.g. composite sample was collected instead of a grab sample).
- 6.25.13.5.1 A "J" value shall be accompanied by justification for its use.
- 6.25.13.5.2 A "J" value shall not be used if another code applies (ex. K, L, M, T, V, Y, PQL)
- 6.25.13.5.3 If more than one code applies, and the data is to be entered into STORET, only one code shall be reported. The code shall be selected based on the following hierarchy: REJ, NAI, O, Y, V, H, J, B, K, L, M, PQL, T, Z, A.

7.0 INTERFERENCES

- 7.1 Carbonate and bicarbonate represent interferences under the terms of this test and must be removed or accounted for in the final concentration.

8.0 SAFETY

- 6.26 Refer to Standard Methods section 1090, 22nd Edition – Laboratory Occupational Health and Safety
- 6.27 Refer to the AEL Chemical Hygiene Plan and Safety Manual for safety precautions and for the Hygiene Plan and Emergency Response Plan.

9.0 EQUIPMENT AND SUPPLIES

Note: SOPs are updated on a set schedule and as a result may not reflect new additions or updates to laboratory equipment inventory at each site as the year progresses. For real-time equipment tracking, please reference the ADMIN-049a AEL QM Section 7.0 - Contemporary Equipment List (most recent revision) link on the intranet SOP system.

- 9.1 Shimadzu Total Organic Carbon Analyzer TOC-Vcsh
- 9.2 Shimadzu Solid Sample Module SSM-5000A
- 9.3 O.I. Aurora 1030 and soil sample Module
- 9.4 Oxygen gas- Zero Grade
- 9.5 Pipette and tips, 2-10 mL and 20-200 µL
- 9.6 Class A volumetric flasks: 1000 mL, 100 mL
- 9.7 Spatula
- 9.8 Sample boats
- 9.9 Ceramic Fiber Wool, purchased commercially from Shimadzu (part no. 638-60074)



9.10 Ottawa Sand

9.11 Oven or Furnace

10.0 REAGENTS AND STANDARDS

Reagents are ordered and tracked in accordance with SOP's ADMIN-013 and ADMIN-031

10.1 Reagent water, laboratory DeIonized (DI) water

10.2 D-Glucose stock solution, 100,000 mg carbon/L: Dissolve 2.5g anhydrous D-Glucose into DI water and dilute to 100 mL. Expires after 6 months.

10.3 Potassium hydrogen phthalate, stock solution, 10,000 mg carbon/L

10.4 Potassium hydrogen phthalate (secondary source standard): Dissolve 2.1254 g of potassium hydrogen phthalate stock solution in DI water and dilute to 100 mL. Expires after 6 months.

10.5 Phosphoric acid, concentrated, ACS Grade or equivalent.

10.6 Phosphoric Acid (5% vol/vol): Prepare a 5% by volume solution of phosphoric acid (H₃PO₄) by adding 59 mL of ACS Reagent Grade 85% H₃PO₄ to reagent water (1 L total volume).

10.7 Hydrochloric Acid, concentrated, ACS Grade or equivalent.

10.8 Hydrochloric Acid (5% vol/vol): Prepare a 5% by volume solution of hydrochloric acid (HCl) by adding 13.89 mL of ACS Reagent Grade 36% HCl to reagent water (100 mL total volume).

11.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND HANDLING

11.1 See AEL QM for sample acceptance policy.

11.2 See FDEP SOP FS1000 for preservation requirements, shipping conditions and holding time requirements.

11.2.1 Samples should be stored >0 - ≤6°C, with no frozen samples.

11.2.2 Samples have a holding time of 28 days from time of collection.

12.0 QUALITY CONTROL

12.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of capability (DOC), and the periodic analysis of laboratory reagent blanks, fortified blanks, and other laboratory solutions as a continuing check on performance. The



laboratory is required to maintain performance records that define the quality of the data that is generated.

- 12.1.1 Each analyst must perform and pass their iDOCs prior to being able to perform this method. A statement must be signed prior to analysis, which includes that the most current SOP has been read and will be adhered to.
- 12.1.2 The initial demonstration of capability will consist of 4 LCS replicates using a spike from a different source than used for normal daily operation.
 - 12.1.2.1 All 4 replicates must be within acceptance criteria for the iDOCs to be valid.
- 12.1.3 Continuing demonstration of capability (cDOCs) may consist of 4 single consecutive batch LCS, a passing PT, or another iDOC study.
- 12.2 The Linear Dynamic Range of the curve must be determined initially, every six months, or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use at least three standards to insure that the resulting curve is linear or quadratic and have a correlation value of at least 0.995. A quadratic curve does require at least six standard points to be analyzed and included for use. The verification of linearity must use a minimum of a blank and mid level standard. If any verification data differs from the initial values by 10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.
- 12.3 When beginning the use of this method, on a per run basis to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a calibration verification (ICV / CCV) and verification blank (ICB / CCB). The verification blank must be below the MDL. The verification must be 80-120% of the target. If the determined concentrations do not meet the requirements listed above, the analysis should be stopped and the source of the problem corrected. If the analyst cannot correct or determine the source of the problem, contact the lab manager or QA Officer for further instructions.
 - 12.3.1 ICV (2000 mg/Kg) – Add a small amount of ceramic wool to a clean boat. Pipette 0.20 mL secondary source standard (Potassium Phthalate) onto the wool.
- 12.4 Each batch of 20 samples should begin and end with the analysis of 1 CCV and 1 CCB. The CCV must be 90-110% of the target in order to continue the run. If the CCV fails to meet this criterion, stop the run, determine the source of the problem, correct the problem if possible and continue the run. If the problem is not immediately correctable, contact the lab manager or QA Officer for further instructions.
 - 12.4.1 CCV (2000 mg/Kg) – Add a small amount of ceramic wool to a clean boat. Pipette 0.20 mL secondary source standard (D-Glucose) onto the wool.
- 12.5 Method Blank (MB): Each batch of samples will contain a MB. The result must be <MDL. If acceptance criteria is not met, corrective action must take place; this can



include: reanalysis, re-preparation, narration or qualification. Use ceramic wool as the matrix.

12.6 Laboratory Control Sample (LCS): Each batch of samples will contain an LCS. The spike must recover at 80-120%. If this condition is not met, a corrective action must take place; this can include: reanalysis, re-preparation, narration, or qualification of the batch.

12.6.1 LCS (2000 mg/Kg) – Add a small amount of ceramic wool to a clean boat. Pipette 0.20 mL secondary source standard (Potassium Phthalate) onto the wool.

12.7 Matrix spike and matrix duplicate (MS/MSD) - Each batch of 20 samples should have 1 randomly selected sample ran with a spike and spike duplicate per matrix per batch of 10 samples.

12.7.1 MS/MSD – (2000 mg/Kg) – Weigh between 0.1 – 1.0 g of soil sample into eight clean boats (four for MS; four for MSD). **IMPORTANT: Directly before** sample is placed in TOC furnace, pipette 0.20 mL secondary source standard (Potassium Phthalate) into each of the four boats.

12.8 Method Detection Limit (MDL) - MDLs must be established for all analytes, following the procedures outlined in ADMIN SOP-012, which conforms to EPA CFR 40 part 136.6 appendix B, updated October 2017. Below is a summary of the steps for performing MDLs. Please refer to the ADMIN SOP-012 for the full procedures.

12.8.1 New MDL Study: Most MDL determinations are for already established analyte, matrix, and instrument combinations. In those cases where a new analyte is to be introduced, an initial MDL study will have to be implemented. Select a spiking level, typically 2 to 10 times the estimated MDL. Process a minimum of seven spiked samples and seven method blank samples through all steps of the method. The samples used for the MDL must be prepared in at least three batches on three separate calendar dates and analyzed on three separate calendar dates. (Preparation and analysis may be on the same day.)

12.8.2 New Instrument: To bring on a new instrument for an already established method a full set of seven low-level replicates is not needed for non-potable water. Only two spiked samples and two method blank samples prepared and analyzed on different calendar dates are required for the new instrument. The resulting values shall be compared against existing MDLs for validity. If both method blank results are below the existing MDL, then the existing MDL_b is validated. Combine the new spiked sample results to the existing spiked sample results and recalculate the MDLs. If the recalculated MDLs is within 0.5 to 2.0 times the existing MDL and fewer than 3% of the MB have results above the existing MDL, the existing MDL can be left unchanged and the new instrument is validated. Drinking water analysis requires a full new study be completed for new instruments.

12.8.3 Existing Instrument, Major Maintenance: Follow the procedures for bringing on a new instrument.



12.8.4 Ongoing Data Collection: During any quarter in which samples are being analyzed, prepare, and analyze a minimum of two spiked samples on each instrument, in separate batches (separate prep batches and separate analytical batches), using the same spiking concentration used with established MDLs. If any analytes are repeatedly not detected in the quarterly spiked sample analyses, or do not meet the qualitative identification criteria of the method then this is an indication that the spiking level is not high enough and should be adjusted upward. Note that it is not necessary to analyze additional method blanks together with the spiked samples; the method blank population should include all of the routine method blanks analyzed with each batch during the course of sample analysis.

12.8.4.1 At least once per year, re-evaluate the spiking level. If the spiked samples do not return positive numerical results that meet all method qualitative identification criteria, then the spiking level must be increased, and the initial MDL re-determined following the procedure for establishing a new MDL.

12.8.5 Ongoing Annual Verification: At least once every thirteen months, recalculate MDLs and MDL_b from the collected spiked samples and method blank results using the equations in the ADMIN SOP-012. These calculations shall be performed by the QA department along with updating and maintaining all chart and LIMs entry of any MDL changes.

12.9 Refer to the AEL Quality Manual for additional information regarding laboratory QAQC.

12.10 See the AEL Quality Manual for any other concerns.

12.11 All quality control data should be maintained and available for easy reference or inspection.

13.0 CALIBRATION AND STANDARDIZATION

13.1 Standard Curve: Prepare fresh standards and new calibration curve every 6 months.

13.2 Tampa prepare standard curve as follows:

| Standards for Curve (mg/Kg) | 20,000 mg/L Glucose Stock Solution |
|-----------------------------|------------------------------------|
| 0.5 | 0.025 mL |
| 1.0 | 0.05 mL |
| 5.0 | 0.25 mL |
| 10 | 0.5 mL |
| 20 | 1.0 mL |
| 30 | 1.5 mL |



13.3 Jacksonville prepare standard curve as follows:

Soil Curve - 100,000mg/L Stock Solution. Made by adding 2.5g D-Glucose stock up to 100mL FV with DiWater.

| | Curve Level (mg) | Volume stock solution added to approx. 0.1g |
|---|-------------------------|--|
| 1 | 0.5 | 5uL |
| 2 | 1 | 10uL |
| 3 | 2.5 | 25uL |
| 4 | 5 | 50uL |
| 5 | 10 | 100uL |
| 6 | 20 | 200uL |

13.4 The TOC analyzer has a calibration mode in the main menu. Follow the onscreen instructions to perform calibration.

13.4.1 A new curve must be established every six months.

13.4.2 Calibration is stored and accessible within the instrument.

13.5 Use a linear curve with a correlation coefficient of 0.995 or greater. Report results within the range of the calibration.

13.6 The lowest point on the curve, outside of the blank, shall be the establish PQL level, which is to be 1 to 10 times the level of the MDL. This point must be included in the curve. If this point does not work in the curve, then maintenance shall be performed on the instrument to correct for the failure and a new curve should be analyzed.

13.7 No result quantitated higher than the high point of the curve shall be reported, unless circumstances dictate such (such as with use of a valid LDR study), at which point those results must be qualified as having less certainty. Samples with results above the high point of the curve shall be re-analyzed at a reduced sample weight until results fall within the range of the curve.

13.8 Any internal point of the calibration curve cannot be dropped to meet the curve acceptance criteria. (In a case of a mis-spike, poor injection, or error in making the standard that is clearly evident, and can be documented as such, then dropping that point, upon the supervisor's approval, will be allowed.) The use of data used with a dropped point requires documentation of the reason and supervisor initials/date. The low point of the curve cannot be dropped.

13.9 Curve points dropped, as mentioned for reasons in section 13.5, can only be replaced with an additional analysis of the affected points upon the supervisor's approval, requires documentation of the reason and supervisor initials/date.

13.10 Relative Error. From the 2016 TNI Standards, the laboratory is required to use and document a measure of relative error in the calibration. By employing relative error acceptance criteria, concentrations calculated from the low end of the curve shall have the



same confidence in their accuracy as those taken from any other point on the curve. Acceptance criteria must be met.

13.10.1 For calibrations evaluated using an average response factor, the determination of the relative standard deviation (RSD) is the measure of the relative error. If the average response factor acceptance criteria have been met, then acceptance is also met for the relative error.

13.10.2 For calibrations evaluated using correlation coefficient or coefficient of determination, the laboratory shall evaluate relative error by either the measurement of the Relative Error (%RE) or the measurement of the Relative Standard Error (%RSE)

13.10.2.1 Relative error is calculated using the following equation:

$$\% \text{ Relative Error} = \frac{x'_i - x_i}{x_i} \times 100$$

x_i = True value for the calibration standard

x'_i = Measured concentration of the calibration standard

13.10.2.1.1 This calculation shall be performed for two (2) calibration levels: the standard at or near the mid-point of the initial calibration and the standard at the lowest level. The mid level of the calibration curve shall pass the method specified or CCV criteria. The low level of the calibration curve shall pass the method specified criteria (otherwise the default assigned limits will be +/-50% difference).

13.10.2.2 Relative Standard Error (%RSE) is calculated using the following equation:

Relative Standard Error is calculated using the following equation:

$$\% RSE = 100 \times \sqrt{\frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}}$$

x_i = True value of the calibration level i

x'_i = Measured concentration of calibration level i

p = Number of terms in the fitting equation
(average = 1, linear = 2, quadratic = 3)

n = Number of calibration points

13.10.2.2.1 The RSE shall meet the criterion specified in the method. If no criterion is specified in the method, the maximum allowable RSE shall be numerically identical to the requirement for RSD in the method. If there is no



specification for RSE or RSD in the method, then the RSE shall be default 20%.

14.0 PROCEDURE

14.1 Tampa Sample Pretreatment to Remove Inorganic Carbon

14.1.1 Tampa: Weigh 0.1-1.0 g of homogenized sample directly into four sample boats. Ensure all four boats are within ± 0.05 g. Record the sample weight in logbook.

14.1.2 Jacksonville: Weigh 0.1-1.0g amount into a crucible per sample.

14.1.1.1 Ottawa Sand is used for the batch QC

14.1.3 Tampa: Add enough concentrated phosphoric acid (drop-by-drop) to the sample to make a slurry. If after a few drops being added there is no inorganic carbon seen being bubbled off then stop adding. If after a few drops being added there is still bubbling off then continue adding until the inorganic carbon reaction dissipates. Do not add too much acid. Too much acid makes it sometimes impossible to clean the sample off the weigh boats after burning at 900 degrees.

14.1.4 A blanket of wool may be placed over the sample to prevent sample loss while transferring to oven. If wool is used, keep it on during the rest of the process.

14.1.5 Put the acidified sample in a 180°C-200°C oven or furnace and dry for about 2 hours.

14.1.6 Remove the dried sample from the oven and let cool. The sample is now ready for analysis.

14.1.7 If the wool blanket was not used prior to placing in oven, add a wool blanket prior to entering TOC instrument in order to prevent sample spillage.

14.2 Jacksonville Sample Pretreatment to Remove Inorganic Carbon

14.2.1 Jacksonville: Weigh 0.1-1.0g amount into a crucible per sample.

14.1.1.1 Ottawa Sand is used for the batch QC

14.2.2 Add enough diluted HCl (drop-by-drop) to the sample to make a slurry. If after a few drops being added there is no inorganic carbon seen being bubbled off then stop adding. If after a few drops being added there is still bubbling off then continue adding until the inorganic carbon reaction dissipates. Do not add too much acid. Too much acid makes it sometimes impossible to clean the sample off the weigh boats after burning at 900 degrees.

14.2.3 Put the acidified sample in a 180°C-200°C oven or furnace and dry for approximately 45min for dry matrices (such as sand), or up to 2 hours for wet matrices.



14.2.4 Remove the dried sample from the oven and let cool. Weigh approx. 0.1g of the QC and samples into a conditioned quartz cup w/glasswool at the base.

14.3 Batch Run Log Example:

With Calibration (every 6 months)

| |
|------------------------------|
| Calibration (Primary Source) |
| ICV (Secondary Source) |
| ICB/MB |
| LDR Study |
| CCV (Primary Source) |
| CCB |

Without Calibration

| |
|---------------------------|
| CCV (Primary Source) |
| CCB/MB |
| LCS (Secondary Source) |
| 10 samples |
| MS/MSD (Secondary Source) |
| CCV (Primary Source) |
| CCB |

14.4 Tampa Procedure:

14.4.1 Turn on both the oxygen gas tank (set at 44 psi) and the air gas tank (set at 70 psi).

14.4.2 Turn on both the power for the SSM (furnace) and the TOC Analyzer.

14.4.3 Open the front door of the SSM-5000A and adjust the carrier gas supply pressure to 200kPa, and turn the carrier gas flow adjustment knob to set the flow gauge to 500 mL/min. The gas pressure for the TOC-Vcsh is set to 200kPa and the flow gauge is set to 150 mL/min.

14.4.4 Open the TOC software on the desktop of the computer and turn on the TC furnace (set at 900°C) and the IC furnace (set at 200°C) using the SSM control window. Connect the TOC/SSM module. Wait till the furnaces temperature rise to the target temperatures and the baseline stabilizes.

14.4.5 Pull down tab “instrument;” select “background monitor” (baseline and oven temps). Once all green checked (about 1.5 hours) ready to begin.

14.4.6 Create a new sample table and insert samples using Auto Generate, choose the correct calibration curve and enter the total numbers of sample including quality controls. Edit the sample table and type in each sample ID in sequence.

14.4.7 Click “Connect” and “Start” icon on the top of the sample table window. Open the TC sample port cover and load the sample boat with pretreated sample or quality control standard into the sample holding position. Close the sample port



cover. Make sure both the TC and the IC sample ports are closed tight. After loading the sample boat and closing the TC sample port cover, wait about 0.5-1.0 minutes before pushing the sample boat into the measuring position.

14.4.8 If organic carbon is below 3 mg, the interference from the CO₂ in the atmosphere can affect the measurement accuracy. To avoid this problem, allow carrier gas to flow for 1.5-3.0 minutes after closing the sample port cover for each sample boat.

14.4.9 Follow the on screen directions from the software and analyze each sample.

14.4.9.1 For liquid sample/standard, lay some ceramic fiber in a clean sample boat first, then add the liquid solution.

14.4.9.2 For crystalline samples such as dry glucose powder, weigh the sample/standard in the sample boat first, then add some ceramic fiber to cover the opening of the crystals to prevent the sample from scattering.

14.4.10 After the samples have been analyzed, set the instrument to shut down in the software and wait at least 45 minutes before shutting down the SSM module. The TOC-V module will shut down on its own after 30 minutes. Very important to **NEVER turn off TOC-V immediately after shutdown or the acrylic TC slider will melt and air tight seal will be ruined.** Wait for the temperature of the TC furnace to drop to 400-450°C then turn off the AC power.

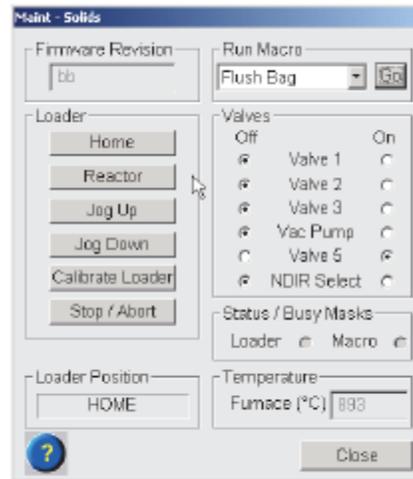
14.4.11 Turn off the oxygen gas cylinder and the air gas cylinder.

14.4.12 Each sample must be analyzed in quadruplicate. The average and the range and the low and high values are to be reported.

14.5 Jacksonville Procedure:

14.5.1 Conditioning Sample Cups: Sources of carbon contamination are common in the laboratory environment. To ensure accurate analytical results, it is necessary to remove as much residual carbon from sample-containing equipment and labware as possible. The 1030S TOC Solids Module includes a macro to condition sample cups. This procedure removes most carbon that might be present on the cup and possibly skew test results. The following procedure outlines how to condition sample cups for the 1030S TOC Solids Module.

14.5.1.1 Press Maint -> Solids to display the Maintenance Solids dialog box, as depicted below



14.5.1.2 Select Condition Cup on the Run Macro drop-down menu.

14.5.1.3 Using forceps, place a sample cup onto the loader arm.

14.5.1.4 Press Go.

14.5.1.5 The loader arm raises the cup into the combustion furnace where the residual carbon is burned off.

14.5.1.6 When the macro is complete and the loader arm lowers, allow the sample cup to cool slightly.

14.5.1.7 Use forceps to remove the sample cup from the loader arm and place it on a crucible rack.

14.5.1.8 Repeat steps 2–6 for all remaining sample cups

14.5.2 The 1030S TOC Solids Module analyzes solids, sludges, slurries, or suspensions. The solids should be ground to a powder or fine grit and homogenized as much as practical for accurate analysis. Mixing the powder with an inert heat mass such as quartz or zirconia pellets improves performance. Quartz wool or a quartz wool disk (PN 325159) should always be used as a liner in the sample cups. The quartz wool retains and suspends the sample, allowing oxygen to flow freely around the sample, and increases the life expectancy of the sample cups.

14.5.2.1 Use quartz wool to line the bottom of the cup. Fill the cup half full of quartz wool, condition the cup, and then add approximately 0.1g of the pre-treated, dried solid sample on top of the quartz wool. Record the weight of the sample added.

14.5.2.2 To limit the potential contamination of a sample cup, use forceps to handle the sample cups as much as possible, particularly after conditioning the cups.



14.5.2.3 Sequencing: Press Editor and select the Sequence tab to display the Editor Sequence screen

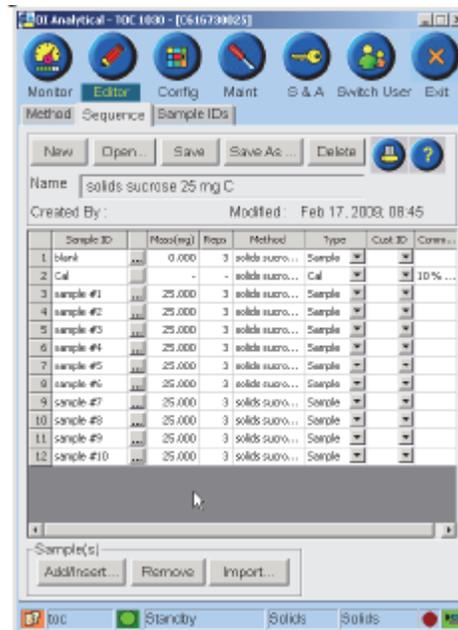
14.5.2.3.1 Press New. The Editor Sequence screen is cleared.

14.5.2.3.2 Enter a Name for the sequence.

14.5.2.3.3 Add samples to the sequence.

14.5.2.3.3.1 Press Add/Insert to display the Add/Insert dialog box.

14.5.2.3.3.2 From the Sample Type drop-down menu, select the appropriate sample type. (Std, QC, Sample).



14.5.2.3.3.3 Set the value for # Reps equivalent to your methodology. QC samples require 1 replicate. All field samples require 4 replicates.

14.5.2.3.3.4 Verify the value for # of Samples is 1.

14.5.2.3.3.5 Enter a Sample ID.

14.5.2.3.3.6 Enter the Sample Mass.

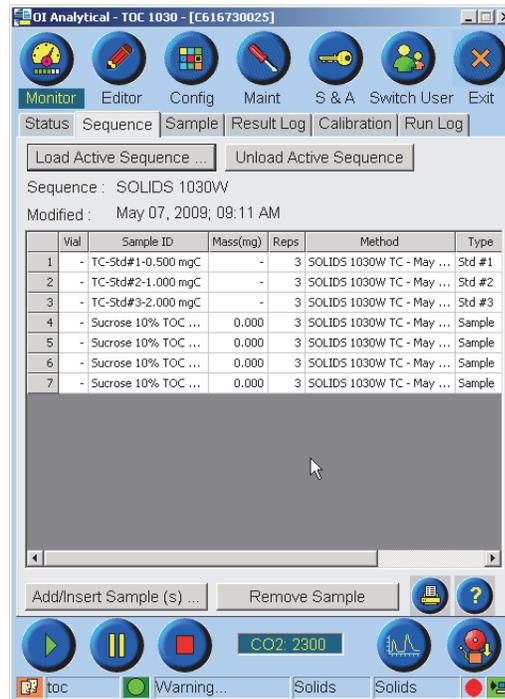
14.5.2.3.3.7 Select a Method from the Primary drop-down menu.



14.5.2.3.3.8 Press OK to exit the Add/Insert Samples dialog box.

14.5.2.3.3.9 Repeat steps above for each additional sample.

14.5.2.3.4 Starting the Sequence: Press Monitor -> Sequence tab to display the Monitor Sequence screen



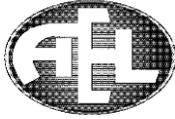
14.5.2.3.4.1 Press Load Active Sequence to display a list of all active sequences in the Aurora.

14.5.2.3.4.2 Select the desired **sequence**.

14.5.2.3.4.3 Press **Load**.

14.5.2.3.4.4 Press **Run** to begin the sequence.

14.5.2.3.4.5 The Aurora begins by priming itself as well as the 1030S TOC Solids Module and purging the sample bag in the 1030S TOC Solids Module. While running the sequence, the 1030S TOC Solids Module pauses between samples so that a new prepared sample can be loaded. After loading a new prepared sample, press **Run** to continue with the sequence when system goes to a “Waiting” state.



14.1 Tampa Sample Boat Cleaning

14.1.1 Remove as much of the sample remaining in the used sample boat as possible.

14.1.2 Use the tip of a metal spatula to scrape off pieces adhering to the bottom of the sample boat.

14.1.3 Clean with DI water.

14.1.4 Soak the boat in approximately 2 M sulfuric acid overnight.

14.1.5 Rinse the sample boat very thoroughly with DI water.

14.1.6 Bake the boats in the furnace at 500 degrees for 30-60 minutes.

15.0 CALCULATIONS

$$15.1 \quad \text{LCS \% Recovery} = \frac{(\text{Obtained Value})}{\text{True Value}} * 100$$

$$15.2 \quad \% \text{ RPD} = \frac{(\text{Difference b/w Dups})}{\text{Average of Dups}} * 100$$

$$15.3 \quad \% \text{ Spike Recovery} = \frac{(\text{Spiked Sample Value} - \text{Sample Value})}{\text{True Value of Spike}} * 100$$

15.4

Standard Make-up = (Desired Concentration)(Final Volume)/(Original Concentration) = mL needed

16.0 METHOD PERFORMANCE

16.1 MDLs are determined in accordance with the Quality Manual and Admin-012.

16.2 PQLs will be at a level greater than that of the MDL. PQLs will be the lowest level of quantitation, the low point of the calibration curve.

16.3 iDOCs are completed in accordance with Admin-030 and this SOP. A passing Demonstration of Capability must be completed by the analyst prior to analyzing any client samples.



16.4 Method control limits are as follows:

Tampa

| | Frequency | Acceptance Criteria |
|--|--|---------------------|
| Calibration Standards | Every 6 months or as needed | ≥0.995 |
| Initial Calibration Verification (ICV) | After Calibration | 80-120% |
| Initial Calibration Blank (ICB) | After ICV | <MDL |
| Continuing Calibration Verification (CCV) | Beginning of analysis, after every 10 samples & at end of analysis | 80-120% |
| Continuing Calibration Blank (CCB) | After CCV | <MDL |
| Method Blank (MB) | 1 per of 20 samples or fewer | <MDL |
| Laboratory Control Sample (LCS) | 1 per batch of 20 samples or fewer | 80-120% |
| Matrix Spike (MS) / Matrix Spike Duplicate (MSD) | 1 per batch of 10 samples or fewer | 80-120% RPD <20% |

Jacksonville

| | Frequency | Acceptance Criteria |
|--|--|---------------------|
| Calibration Standards | Every 6 months or as needed | ≥0.995 |
| Initial Calibration Verification (ICV) | After Calibration | 90-110% |
| Initial Calibration Blank (ICB) | After ICV | <MDL |
| Continuing Calibration Verification (CCV) | Beginning of analysis, after every 10 samples & at end of analysis | 90-110% |
| Continuing Calibration Blank (CCB) | After CCV | <MDL |
| Method Blank (MB) | 1 per of 20 samples or fewer | <MDL |
| Laboratory Control Sample (LCS) | 1 per batch of 20 samples or fewer | 90-110% |
| Matrix Spike (MS) / Matrix Spike Duplicate (MSD) | 1 per batch of 10 samples or fewer | 75-125% RPD <25% |

16.5 Control charts are generated and reviewed for trend analysis at least annually.

17.0 POLLUTION PREVENTION

17.1 See Standard Methods section 1100, 22nd Edition – Waste Minimization and Disposal.

17.2 See SOP Admin-018 and the AEL Safety Manual.

18.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

18.1 Also refer to Section 16.0.

18.2 The CCV must meet method acceptance criteria to report unqualified data. If the CCV is outside acceptance criteria, samples must be reanalyzed since the last passing CCV.

18.3 Method Blank (MB) must pass acceptance limits and be clean of analyte of interest. If there are hits on the method blank, these hits must be below the method reporting level. To report a hit in the MB, the batch needs to be qualified or narrated.



18.3.1 If above the MDL and the samples themselves are clean, sample results are fine to report out with a qualified MB.

18.3.2 If the hit in the sample is ten times the hit in the MB, the sample results are fine to report out with a case narrative.

18.4 Laboratory Control Spikes (LCS) must fall within control chart limits for percent recoveries for valid data reporting. If it fails, check instrument first for possible problems, document, then reanalyze LCS. If it still fails, all samples in the batch must be re-prepared or reanalyzed.

18.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)-Failure to meet control limits for analyte and surrogate recoveries does not in itself require data to be rejected. Data will be flagged (J4) for matrix interference.

18.6 Any and all QC failures must be reported to a supervisor.

18.7 At any point in the analytical batch, an analyst may use discretion to fail or reject data to be suspicious or in error. At this point, the analyst will seek guidance from a supervisor or the QC officer.

18.8 The analyst is responsible for reviewing the QC samples to ensure that they pass criteria.

19.0 CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

19.1 Refer to Sections 12.0 and 18.0.

20.0 CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

20.1 Refer to Sections 12.0 and 18.0.

20.2 Refer to the SOP for Non-Conformities (ADMIN-016) for instructions on NCF use.

21.0 WASTE MANAGEMENT

21.1 See Standards Method section 1100, 22nd Edition – Waste Minimization and Disposal

21.2 Refer to AEL SOP ADMIN-018, Waste Disposal

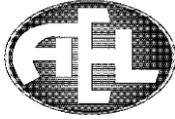
22.0 MAINTENANCE

22.1 Tampa: TOC Combustion Tube Cleaning for reuse.

22.1.1 You will need two types of brushes. A 6 inch and an 18 inch, both soft bristle.



- 22.1.2 Initially, just rinse the tube out well and clean the end of the tube out as it will probably be partially clogged.
 - 22.1.3 Next, sit the tube in H₂SO₄ with a normality around 12. Concentrated Sulfuric is about 36 Normal, so add 1 part Sulfuric to two parts DI water. Let the tube sit overnight in the acid mix.
 - 22.1.4 The next day, rinse the tube well and scrub with liquinox using the brushes.
 - 22.1.5 Then, submerge the tube in 1N NaOH overnight and then again, rinse the tube well and scrub with liquinox using the brushes.
 - 22.1.6 Finally, rinse the tube well and dip back in the sulfuric acid used from the first day and immediately rinse with DI and let it dry.
 - 22.1.7 The tube will not look pretty, but it will be ready to go.
 - 22.1.8 When you go to use the cleaned combustion tubes, run with 10 to 15 blanks before you start again. This should be done anyways, since you repacked the tube.
 - 22.2 Jacksonville: See Attachment 24.2 for manufacturer's instructions for maintenance and replacement parts.
- 23.0 REFERENCES
- 23.1 EPA Method 9060A
 - 23.2 AEL Chemical Hygiene Plan and Safety Manual
 - 23.3 AEL Quality Manual
 - 23.4 2016 TNI standards
 - 23.5 Admin SOPs
 - 23.5.1 ADMIN-016: Non-Conformities
 - 23.5.2 ADMIN-018: Waste Management
 - 23.5.3 ADMIN-008: Data Qualifiers
 - 23.5.4 ADMIN-011: Significant Figures
 - 23.5.5 ADMIN-013: Ordering Reagents
 - 23.5.6 ADMIN-030: DOC
 - 23.5.7 ADMIN-031: Receipt of Consumables
 - 23.5.8 ADMIN-032: DI Water Maintenance
 - 23.5.9 ADMIN-033: Acceptable Ranges and Control Charts
 - 23.5.10 ADMIN-049a AEL QM Section 7.0 - Contemporary Equipment List (most recent revision)
 - 23.6 1030S TOC Solids Module Operator's Manual, OI Analytical Rev 2.0, April 2014



24.0 ATTACHMENTS

- 24.1 Validation Data. See the employee file for the following individuals for an acceptable initial demonstration of capability, which serves as validation date for this method in AEL.
- 24.2 Excerpts from 1030S TOC Solids Module Operator's Manual, OI Analytical Rev 2.0, April 2014 (see below):



Chapter 5 Maintenance

This chapter discusses the routine and non-scheduled maintenance of the 1030S TOC Solids Module, starting with some general information and a maintenance schedule.

Exterior Maintenance

Wipe exterior surfaces with a lint-free, nonabrasive cloth to remove loose dust. Do not use abrasive cleaners. Wipe up any spills immediately with a soft cloth dampened with water. Wipe dry with a soft cloth. Neutralize corrosive spills immediately with an appropriate compound.

Routine Maintenance Schedule

OI Analytical recommends maintaining an instrument log book to record instrument operation time and document periodic maintenance. Use this log book to record inspection results and component replacements necessary for proper maintenance of the 1030S TOC Solids Module.

For the most reliable performance of the 1030S TOC Solids Module and as a condition of the warranty, use the following schedule for routine maintenance. Scheduled hours refer to number of hours of operation.

Table 5.1. Maintenance Schedule

| Maintenance Item | Schedule |
|---------------------|------------------------|
| Sample cup | As needed |
| Combustion tube | 9–12 months |
| Combustion tube cap | As needed |
| Particulate filter | Empty daily |
| Riser tube | As needed |
| O-rings | Grease every two weeks |
| Gas Service | As needed |
| Sample Bag | Replace every 6 months |



| | |
|-------------------------|---------------------------|
| Quartz wool/filter | As needed |
| Fittings combustion cap | Check tightness as needed |

Checking the Crucible

Solid Samples to be analyzed are manually transferred and weighed in quartz crucibles/ sample cups. Two different volume crucibles are available (1 mL and 2.5 mL) to address differences in mass, bulk density, and anticipated carbon content of sample. Refer to chapter 7 for crucible part numbers.

Replacing the Crucible

The following procedure outlines how to replace damaged or worn crucibles.

WARNING:

Ensure that the sample cup is properly cooled to room temperature to avoid risk of fire or personal injury.

carbon from sample cup surface.

Checking the Combustion Tube

Carbon is oxidized to carbon dioxide inside the 1030S TOC Solids Module's Combustion Tube. The CO₂ is transferred through the tube via oxygen carrier gas to a collection bag.

Replacing the Combustion Tube

The following procedure outlines how to replace the combustion tube.

1. Remove sample cup from riser and place in proper glass waste receptacle.
 2. Retrieve fresh sample cup.
 3. Place glass wool in bottom of sample cup.
 4. Place sample cup in riser.
 5. Maint-> Solids --
- From drop-down menu, Select "Condition Cup" to remove residual
 8. Replace with packed combustion tube. (Follow procedure xxx on packing combustion tube.)
 9. Reconnect gas collection tube to the side port of the combustion tube cap.
 10. Reconnect inlet gas tube from top of combustion tube cap.
 11. Replace the cover on the Solids module.
 12. Turn on 1030S Solids Module.
1. Turn off the 1030S Solids Module.
 2. Allow for the Solids Module to cool down before removing the tube.
 3. Remove the top cover of the Solids Module.
 4. Remove inlet gas line from the top of combustion tube cap. Be careful not to damage the counterflow needle.
 5. Remove gas collection tube from the side port of the combustion tube cap.
 6. Pull combustion tube from ring. (Pull firmly but gently as fit is tight to prevent leakage.)
 7. Add Krytox® to bottom of the tube to properly slide tube into ring.



Checking the Combustion Tube Cap

The combustion tube cap allows for oxygen carrier gas to enter into the combustion tube, as well as, the CO₂ produced during combustion to flow through to a sampling. **Replacing the Combustion Tube Cap**

The following procedure outlines how to replace the combustion tube cap:

1. Turn off the 1030S Solids Module.
2. Allow for the Solids Module to cool down before removing the tube.
3. Remove the top cover of the Solids Module.
4. Remove inlet gas line from top of combustion tube cap.
5. Remove gas collection tube from the side port of the combustion tube cap.
6. Pull combustion tube from ring. (Pull firmly but gently as fit is tight to prevent leakage.)
7. Use 1 1/8" wrench on stainless nut and 1" wrench on combustion tube cap to loosen.
8. Place needle in new combustion cap.
9. Remove ceramic needle.
10. Place cap on combustion tube and tighten with wrenches
11. Add Krytox® to bottom of the tube to properly slide tube into ring.
12. Reconnect gas collection tube to the side port of the combustion tube cap.
13. Reconnect inlet gas tube from top of combustion tube cap.
14. Replace the cover of the Solids module.
15. Turn on 1030S Solids Module.

Checking the Particulate Filter

The 1030S TOC Solids Module's particulate filter traps excess water and filters out any particulate matter in the flow path. Check the filter to ensure it is not clogged.

CAUTION:



The particulate filter may contain sulfuric acid, depending on the sample materials combusted in the furnace. Sulfuric acid is a known corrosive compound. Always wear appropriate chemical eye and skin protection while handling the filter insert.

1. Locate the **particulate filter** (PN 319855) on the rear side of the 1030S TOC Solids Module, as depicted in Figure 5.1.



Figure 5.1. Particulate Filter

2. Visually inspect the **polyethylene filter element** for discoloration.
3. Replace the **filter** if it is **dirty**.
4. Drain accumulated liquid, if present.

Replacing the Particulate Filter

The following procedure outlines how to replace the particulate filter (PN 319854).

CAUTION

the left side of the 1030S TOC Solids Module.

Note the orientation of the flow filter by the indicator arrow in the middle.

2. Remove the fitting on the right side (line from combustion tube) and attach to new particulate filter (PN 319855).

NOTE: Flow indicator arrow on filter should be pointing to the left.

1. Locate the **particulate filter** on

3. Remove the fitting on the left side (line going to the sample bag).

4. Remove old particulate filter from clip.

6. Attach fitting to left side of particulate filter (sample bag side).

5. Attach new particulate filter (PN 319855) to clip.

- a. Pull combustion tube straight up from the reactor base and allow the tube to come to rest as you completely unsew the reactor base from the furnace stage and remove it. Rest the combustion tube assembly on the furnace and stage while you replace the o-ring.



- b. Remove the old reactor base o-ring from the reactor base and discard properly. Wipe down the inside of the reactor base with clean Kimwipes™ or paper towels until they come away clean.
- c. Obtain a new reactor base o-ring and, while wearing gloves, work some Krytox™ into the surface of the o-ring. Then, insert the o-ring into the reactor base and give it a light coating of Krytox™ on the inner bearing surface of the o-ring.
- d. Screw the reactor base back into the furnace stage fully and re-insert the combustion tube.

Checking the Riser Tube

The 1030S Solids Module's Riser Tube allows for the crucible to be raised into the combustion tube and furnace as well as assist in flow of oxygen through the combustion tube to move CO₂ to the sampling bag. **Replacing the Riser Tube**

The following procedure outlines how to replace the riser tube.

1. Turn off Solids module.
2. Gently, but firmly, slide riser tube out of holder.
3. Slide new riser tube into holder and seat it firmly.

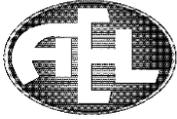
Checking the O-rings

The 1030S Solids Module's O-Rings provide solid support of glassware in the furnace for the combustion tube.

Replacing the O-rings

The following procedure outlines how to replace the O-rings.

1. Turn off the 1030S Solids Module.
2. Allow for the Solids Module to cool down before removing the tube.
3. Remove the top cover of the Solids Module.
4. Remove inlet gas line from top of combustion tube cap.
5. Remove gas collection tube from the side port of the combustion tube cap.
6. Pull combustion tube from ring. (Pull firmly but gently as fit is tight to prevent leakage.)
 - a. If O-ring is attached to tube, remove o-ring and place new ring on tube.
 - b. If O-ring resides in combustion furnace assembly,



7. Reconnect gas collection tube to the side port of the combustion tube cap.
8. Reconnect inlet gas tube from top of combustion tube cap.
9. Replace the cover to the Solids module.
10. Turn on 1030S Solids Module.

Gas Service

Gas consumption is listed in “Gas Requirements” on page 3 of Chapter 1, “Introduction”. A standard 232-cubic foot cylinder is pressurized to approximately 2,200 psi. There are 28.32 liters per cubic foot. Thus, a standard 232-cubic foot cylinder should last at least 260 hours. Monitor cylinder gas pressure after each 100 hours of operation with gas flow to confirm sufficient gas for planned operation.

Checking the Sample Bag

To check the sample bag for leaks, ensure the bag properly inflates and deflates by activating the "Flush Bag" macro in Maintenance, Solids. The sample bag should deflate first to avoid double-filling, and then go through a series of backfill and purge cycles. The number and duration of which are set in Configuration, Sample Intro, Solids.

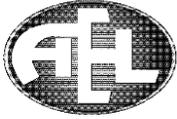
The sample bag should hang vertically in the compartment and completely deflate during the purge cycles. The pump cadence will change to reflect that it is pulling a vacuum on an empty bag. Also, the mixing straw present in the bag should hang straight down into the center of the bag, which can be seen during the purge cycle. The straw should originate at the entrance fitting of the bag and hang straight down in a 6 o'clock position, terminating in the middle of the sample bag. If not, the straw can be manipulated from outside the bag by hand.

The easiest way to do this is to toggle on Valve 2 in Maintenance, Solids for 5 seconds and then toggle it off again. This should partially inflate the bag so the mixing straw can be freely moved into position. Re-purge the bag and check the mixing straw position. Repeat as necessary.

Replacing the Sample Bag

The following procedure outlines how to replace the sample bag.

1. With the instrument in a Standby state, open the bag compartment door and lift the sample bag from its cradle in the center bulkhead.
2. Unscrew the yellow 1/4-28 fitting from the bag and remove it. Dispose of the bag properly.
3. Install a new bag by tightening the yellow 1/4-28 fitting into the fitting at the mouth of the bag and sliding it into place in the cradle in the center bulkhead.
 - a. Make sure that the bag hangs vertically in the compartment by reseating the fitting as necessary.



Checking the Quartz Wool/Filter

The quartz wool or filter pad will become contaminated over time with salts and uncombusted material, in addition to any uncombustible sample matrices. It will also become brittle over time. If the wool or filter pad becomes noticeably stained or contaminated, or brittle, it will need to be replaced. It may also be replaced more frequently if you prefer.

Replacing the Quartz Wool/Filter

The following procedure outlines how to replace the quartz/wool filter.

1. Remove the old wool or filter with forceps and dispose of properly.
 - a. For wool, use forceps to gather an amount of wool sufficient to fill half the crucible and press it into shape in the crucible.
 - b. For a filter pad, use forceps to place the pad inside the crucible so it is not under spring tension, which can eject your sample if the pad dislodges from the crucible.

Checking the Fittings to the Combustion Cap

Make sure the fittings attached to the top and sides of the combustion cap are finger tight. If not, remove the fittings and check the compression of the ferrule fitting to be sure it is still usable (not crushed or elliptical).

Also, check for charring of the fitting and ferrule. If the ferrule and fitting appear to be mechanically stable and not charred, re-tighten the fittings in the correct ports (blue on top; red on the side) and use the supplied black plastic torque wrench to tighten the fittings to the recommended level.

Replacing the Fittings to the Combustion Cap

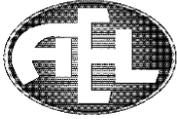
If the ferrule appears deformed or the fitting or ferrule appear charred, they will need to be replaced. The following procedure outlines how to replace the fittings to the combustion cap.

1. Remove the damaged ferrule by pulling it from the end of the tubing and slide the 1/4-28 fitting away from the end of the tubing.
 - a. If the tubing is pinched, cut the tubing just behind the pinch.
 - b. If the 1/4-28 fitting is charred, remove it and dispose of it properly.
2. Slide the 1/4-28 fitting (either original or replacement) toward the end of the tubing and slide a 1/8" ferrule onto the end of the tubing with the flat side toward the end of the tubing.
3. Replace the end of the tube in the port from which it was removed and tighten it using the supplied black torque wrench until it clicks and releases.

Non-Scheduled Mechanical Maintenance

This section describes procedures for setting and testing certain mechanical components for proper operation if they are replaced during nonscheduled maintenance (troubleshooting).

Table 5.2. Non-Scheduled Mechanical Maintenance



| Maintenance Item | Schedule |
|--------------------------------------|-----------|
| Conditioning Sample Cups | As needed |
| Leak Check | As needed |
| Combustion Tube to Sample Bag Tubing | As needed |
| Sample Bag to Aurora Tubing | As needed |
| Aurora Syringe Valve | As needed |

WARNING

Sample cups can be VERY hot. ALWAYS use forceps to remove cups from the 1030S TOC Solids Module. Touching a hot cup can cause severe burns.

Conditioning Sample Cups

Sample cups should be cleaned and conditioned prior to use. Properly cleaned and conditioned sample cups prevent carbon contamination of the sample for accurate measurements.

1. If necessary, **wash** the sample cups with a solution of **10% nitric acid** and **reagent water**.

WARNING:

Nitric acid is a corrosive substance. Always wear appropriate chemical eye and skin protection when handling this material.

2. Place **quartz wool** in the sample cup.
There are two options: quartz wool fiber (PN 144501) or 25–mm quartz wool disks (PN 325159). Place the wool as depicted in Figure 5.2.



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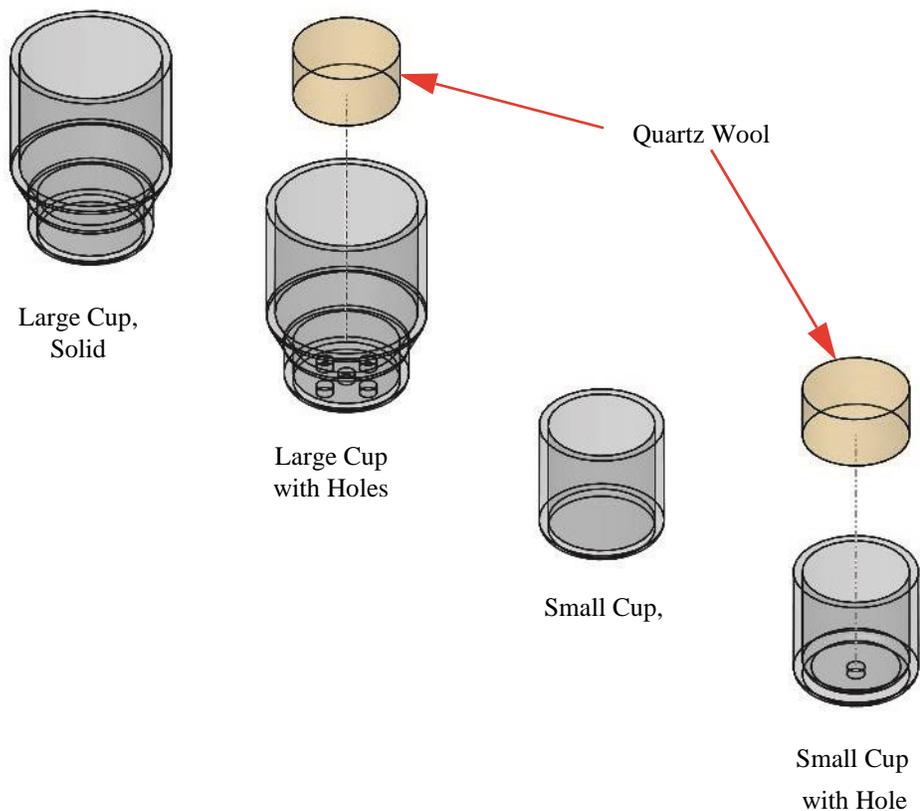


Figure 5.2. Packing the Sample Cup with Quartz Wool or Disk

3. Place the **sample cup** on the **quartz riser tube**.
4. Press **Maint** **Solids**.

The Solids Maintenance dialog box is displayed, as depicted in Figure 5.3.

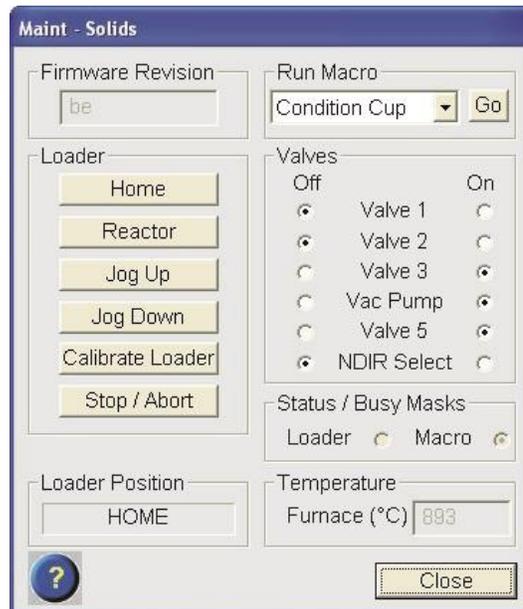


Figure 5.3. Solids Maintenance Dialog Box

5. Select **Condition Cup** from the Run Macro drop-down menu.

NOTE: Ensure furnace is at 900 °C to properly burn off all carbon residue.

6. Press **Go**.

The loader arm lifts the cup into the furnace automatically and lowers it when the cup is conditioned.

WARNING

Sample cups will be VERY hot after conditioning routine. ALWAYS use forceps to remove cups from the 1030S TOC Solids Module. Touching a hot cup can cause severe burns.

The Aurora briefly displays a dialog box indicating it is busy. The Solids Maintenance dialog box stays on screen. This allows for quickly conditioning more than one cup by loading a new cup and pressing Go again. Once the final cup is conditioned, press **Close** to dismiss the dialog box.

At the end of the conditioning routine, the loader arm lowers to allow removal of the conditioned cup.

WARNING

Place sample cups on a non-flammable surface such as the sample rack.

- Remove the **cup** using forceps.

Leak Check

Before performing any leak check procedures, verify the gases are turned on and connected to the instrument and that all shipping plugs have been removed from the exit vents. Tools needed to check for leaks include a flowmeter with a range of 0–1,000 mL/min. or greater and a pressure gauge with a range of 0–30 psi.

NOTE: Gauges with a higher range are more difficult to read at lower pressures.

A calibrated flowmeter is available from OI Analytical as PN 320438. A calibrated pressure gauge is available from OI Analytical as part of a kit, PN 323690.

There are three potential areas for leakage: from the combustion tube to the sample bag, from the sample bag to the Aurora, and the syringe valve in the Aurora. The former requires the flowmeter, while the latter two require the pressure gauge.

Combustion Tube to Sample Bag

This check measures the flow of gases through the module up to the sample bag but does not include the sample bag. To check for a leak in the sample bag, see “Sample Bag to Aurora” on page 65.

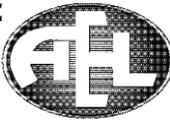
The following procedure outlines how to check for leaks between the combustion tube and the sample bag.

- Detach the **tube** between the **combustion tube** and the **particulate filter**, as depicted in Figure 5.4.



Figure 5.4. Tube Between Particulate Filter and Combustion Tube

- Raise the **loader arm** into the furnace.
- Connect a **calibrated flowmeter** (PN 320438) to the tube from the combustion furnace for a minimum of **30 seconds**.

| | | |
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|--|--|---|

The flowmeter should read a steady 130 mL/min., ± 10 mL/min.

If the flowmeter does not read 130 mL/min., check the O-ring seal at the base of the combustion tube and the O-ring seal on the loader arm. Applying a light coating of Krytox® may help ensure a good seal.

If the flowmeter reading continues to be too low or too high, check the pressure to ensure it is still 20 psi. The flow is regulated by a frit and the pressure directly influences the flow.

Ensure that there is approximately 1 mm of free movement in the position of the riser tube holder to allow for the tube to properly seat itself.



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Aurora Syringe Valve

This check measures the flow at the Aurora syringe valve. The following procedure outlines how to measure this flow.

1. Locate the **tube** from the 1030S TOC Solids Module at **Port F** of the **syringe valve** on the Aurora, as depicted in Figure 5.5.

Syringe
Valve
Port F

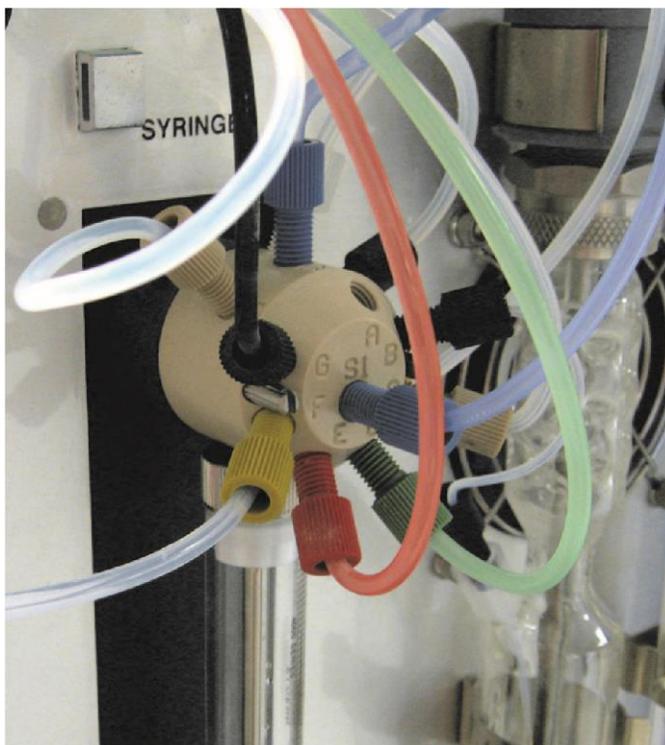


Figure 5.5. Aurora Syringe Valve with Tubing from Solids TOC Analyzer Module

2. Disconnect the **tube** from the syringe valve
3. Attach the **pressure gauge** to the **tube**.
4. On the Aurora, select **Maint** **Solids**.

The Solids dialog box appears, as depicted in Figure 5.6.



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Figure 5.6. Solids Maintenance Dialog Box

5. Change **Valve 2** to **On**.
6. Allow **sample bag** to **fill** for approximately **5 seconds**.
7. Change **Valve 2** to **Off**.
8. Measure **pressure** on the pressure gauge.

Pressure should hold steady at **20 psi**.

If pressure does not hold steady, check all connections.

If pressure still does not hold steady, then call technical support at OI Analytical. The syringe valve may need replacing.

9. Press **Close** to dismiss the dialog box and return to the Maintenance screen.

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Chapter 6 Troubleshooting

The following is a list of 1030S TOC Solids Module symptoms (using keyboard or printer), their probable causes and suggested corrective actions. Before using this section, become thoroughly familiar with the operation and maintenance information contained in previous chapters.

System Performance Symptoms

Table 6.1 lists the symptoms affecting system performance and lists probable causes and corrective actions.

Table 6.1. System Performance Symptoms

| Symptom | Probable Cause | Corrective Action |
|----------------------------------|--|--|
| No response | No gas flow. | Check gas source. |
| | Loose communication cable. | Check cable connections. |
| | No sample. | Introduce sample. |
| Non-reproducible response for TC | System leak. | Perform leak check. |
| | Insufficient detect time for high samples. | Extend detect times. |
| | Variable amounts of quartz wool used. | Fill sample cups consistently. |
| | Sample bag has leak. | Perform leak check. |
| Non-linear response for TC | Furnace temperature set too low. | Set correct furnace temperature. |
| | Carbon mass exceeds detector's linear range. | Reduce sample weight introduced. |
| | Flow rate incorrect. | Set flow rate. See Chapter 5, "Flow Adjustment for Dryer/Actuator Gas" on page 48. |
| | Open cup used instead of a closed cup. | Use a closed cup. |

| | | |
|--|---|---------------------------------|
| | Calibration range does not match samples. | Bracket samples with standards. |
|--|---|---------------------------------|

1030S TOC Solids Module Operator's Manual: Chapter 6

Table 6.1. System Performance Symptoms (Continued)

| Symptom | Probable Cause | Corrective Action |
|--------------------------------------|--|---|
| Negative values displayed or printed | Bad calibration. | Recalibrate 1030S TOC Solids Module. See Chapter 4, "Calibration Procedures" on page 28. |
| Low response for TC | Incorrect calibration. | Recalibrate with 1030S TOC Solids Module. See Chapter 4, "Calibration Procedures" on page 28. |
| | Open cup used instead of a closed cup. | Use a closed cup. |
| | Furnace temperature set too low. | Set correct furnace temperature. |
| | Incorrect sample weight entered. | Enter proper sample weight. |
| | Carrier system leak. | Perform leak check. |
| | TC mass exceeds detector's linear range. | Reduce sample size or dilute sample. |
| High response for TC | Calibration incorrect. | Recalibrate 1030S TOC Solids Module. See Chapter 4, "Calibration Procedures" on page 28. |
| | Incorrect carrier flow rate. | Set carrier flow rate. |
| | System contamination. | Check crucible, combustion tube, combustion tube cap, particulate filter, riser tube, and O-rings |

Table 6.1. System Performance Symptoms (Continued)

| Symptom | Probable Cause | Corrective Action |
|---------|----------------|-------------------|
|---------|----------------|-------------------|

| | | |
|--|--|--|
| 1030S TOC Solids Module will not power up | 1030S TOC Solids Module not plugged into appropriate line voltage. | Check power cord connection. Check power breaker to plug outlet. Reset if tripped. |
| | Blown fuse. | Check A/C power receptacle fuses and replace if blown. Caution: Turn off the main power switch before attempting to change fuses. Operator shall not replace fuses that are hazardous live. |
| Lift mechanism does not move smoothly | Leadscrew not clean. | Clean leadscrew and lubricate. |
| | Leadscrew needs lubrication. | Clean leadscrew and lubricate. |
| Lift mechanism does not move. | Motor dead. | Check connections for loose/broken wires. Replace motor. |
| Peak Detection results of sample stair-step. | Sample not fully combusted. | Increase combustion time. Samples generally require at least 3 minutes to fully combust. |
| Flow from combustion furnace outside acceptable limits | Bad seal at base of combustion tube or loader arm. | Reset the combustion tube, applying a light coating of Krytox® grease. Replace O-ring |

*1030S TOC Solids Module Operator's Manual:
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Chapter 7 Replacement Parts

This chapter provides a list of replacement parts and support items for the 1030S TOC Solids Module and its associated options. An asterisk indicates replacement parts that are considered expendable (XPN). Replace expendable parts regularly, since they may become deformed or broken. Keep a supply of expendable parts in stock.

NOTE: All replacement parts specified below are to be supplied only by OI Analytical or one of our authorized distributors.

Replacement Parts

Table 7.1 lists the replacement parts for the TOC Solids Module

Table 7.1. Replacement Parts for the 1030S by section

| Replacement Parts for the TOC Solids Module | | | | |
|---|----------|------|--------|-----|
| 1030S-GDM Structural Components | | | | |
| Product | Size | Unit | PN | XPN |
| Lexan Front Window | n/a | Each | 326495 | |
| Lexan Side Window | n/a | Each | 326496 | |
| Handle for Side Window | n/a | Each | 326497 | |
| Handle for Front Window | n/a | Each | 326498 | |
| Water and Particle Trap Assembly | n/a | Each | 326633 | |
| Elevator Sealing Brush Assembly | n/a | Each | 326640 | |
| 1030S Electronic Components | | | | |
| 115-V Power Cable | 4 meters | Each | 116038 | |
| 6.3 A Slow Blow Fuse | n/a | Each | 249177 | * |
| Green Power Indicator LED | n/a | Each | 321130 | |
| Red Furnace Status LED | n/a | Each | 321131 | |
| 7' RS485 Cable | 7 feet | Each | 322313 | |
| Cooling Fan Assembly | n/a | Each | 324918 | |
| Valve 1-4 Wiring Harness | n/a | Each | 325051 | |



| | | | | |
|--------------------------|-----|------|--------|--|
| Valve 5-6 Wiring Harness | n/a | Each | 325117 | |
|--------------------------|-----|------|--------|--|

1030S TOC Solids Module Operator's Manual: Chapter 7

| Replacement Parts for the TOC Solids Module | | | | |
|--|-----|------|--------|--|
| Vacuum Pump | n/a | Each | 325306 | |
| Thermocouple Assembly | n/a | Each | 325309 | |
| Power Entry Switch | n/a | Each | 325311 | |
| Power Supply Grounding Cable | n/a | Each | 325608 | |
| DC Power Cable | n/a | Each | 325609 | |
| AC Power Cable for PCF | n/a | Each | 325610 | |
| AC Power Cable for Power Supply | n/a | Each | 325611 | |
| Line Filter Grounding Cable | n/a | Each | 326192 | |
| Magnetic Sensor | n/a | Each | 326199 | |
| Main Circuit Board | n/a | Each | 326402 | |
| Gas Manifold Assembly | n/a | Each | 326408 | |
| Replacement 3-Way Gas Valve | n/a | Each | 326480 | |
| Replacement Metric Valve Screws | n/a | Each | 326482 | |
| Thermocouple Extension Cable | n/a | Each | 326726 | |
| Furnace Power Extension Cable | n/a | Each | 326727 | |
| 115 V Furnace Assembly | n/a | Each | 326729 | |
| 230 V Furnace Assembly | n/a | Each | 326733 | |
| 1030S Fittings and Plumbing | | | | |



| | | | | |
|---|-------------------------|------|--------|--|
| In-line Membrane Moisture Filter | n/a | Each | 192120 | |
| 3-way Pushlock Splitter | n/a | Each | 319861 | |
| 1/16" 1/4-28 Fittings and Ferrules | n/a | 5/pk | 322247 | |
| PTFE washer | .210" D by 0.070" ID | Each | 323689 | |
| 1/8" 1/4-28 Ferrules | n/a | 5/pk | 323903 | |
| Tan 1/4-28 1/8" Fittings | n/a | 5/pk | 323907 | |
| 1/4-28 Restrictor Housing Fitting | n/a | Each | 326147 | |
| Restrictor, 30 ccm @ 20 psi O ₂ | n/a | Each | 326642 | |
| Restrictor, 750 ccm @ 20 psi O ₂ | n/a | Each | 326644 | |
| 1/4-28 to 1/8" Barb Fitting | n/a | Each | 326731 | |

| Replacement Parts for the TOC Solids Module | | | | |
|--|-----|------|--------|---|
| Pressure Relief Valve Assembly | n/a | Each | 327034 | |
| Restrictor, 100 ccm @ 20 psi O ₂ | n/a | Each | 327409 | |
| 1030S Combustor-Related Parts | | | | |
| Combustor Ferrule | n/a | Each | 224204 | |
| Combustor Knurled Nut | n/a | Each | 224675 | |
| Quartz Combustion Tube | n/a | Each | 323972 | * |
| Quartz Riser Tube | n/a | Each | 324728 | * |
| Riser Tube Holder Assembly | n/a | Each | 324912 | |
| Counterflow Needle | n/a | Each | 326406 | * |

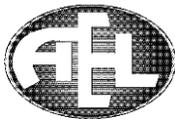


| | | | | |
|--|-----------|------|--------|---|
| Combustion Tube Cap | n/a | Each | 326477 | |
| Combustion Tube Lower Manifold Assembly | n/a | Each | 327334 | |
| 1030S Startup and Preventive Maintenance Kits | | | | |
| Combustion Tube Kit | n/a | Each | 325155 | * |
| Startup Kit | n/a | Each | 325152 | * |
| 1030S Software, Tools, and Accessories | | | | |
| 100- μ L Gas Tight syringe | n/a | Each | 110221 | * |
| Angled-Head Forceps | n/a | Each | 325157 | * |
| Stainless Steel Sample Rack | n/a | Each | 325160 | * |
| Crucible Tongs | n/a | Each | 325161 | * |
| 1030S Consumables and Calibration Standards | | | | |
| Quartz Wool Fiber | 3.5 grams | Each | 144501 | * |
| Conditioned Pt Catalyst | 26 grams | Each | 303032 | * |
| Water and Particle Filter Cartridge | n/a | Each | 319854 | * |
| Zirconia Catalyst | 30 grams | Each | 319856 | * |
| Large, Closed Bottom Crucible | 2.5 mL | Each | 324729 | * |
| Large, Perforated Bottom Crucible | 2.5 mL | Each | 324730 | * |
| Riser Tube Holder Inner O-Ring | n/a | Each | 324742 | * |

| Replacement Parts for the TOC Solids Module | | | | |
|--|------------------------|------|--------|---|
| Riser Tube Holder Outer O-Ring | 0.162 X 0.103 X S70 | Each | 324743 | * |
| Small, Perforated Bottom Crucible | 1 mL | Each | 324899 | * |



| | | | | |
|-------------------------------|------------|-------|--------|---|
| Small, Closed Bottom Crucible | 1 mL | Each | 324900 | * |
| Riser Tube Holder Spacer | n/a | Each | 324910 | * |
| Combustion Tube Lower O-Ring | n/a | Each | 325195 | * |
| Quartz Filter Disk | 25 mm Dia. | 25/pk | 325310 | * |
| 1% Carbon Sucrose Kit | n/a | Each | 326483 | * |
| Krytox High Temp Lubricant | n/a | Each | 326484 | * |
| 10% Carbon Sucrose Kit | n/a | Each | 325621 | * |
| Two Pack of Sample Bags | 1 Liter | 2/pk | 326919 | * |

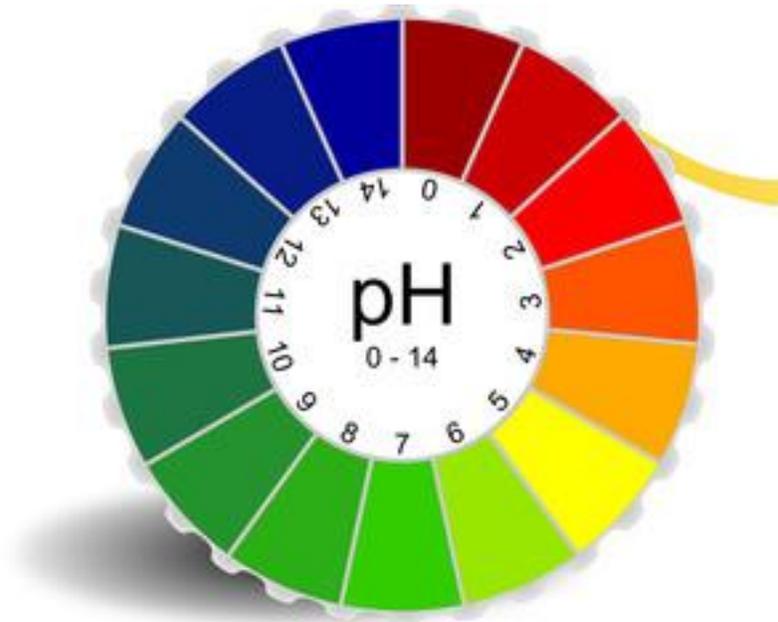


STANDARD OPERATING PROCEDURE

For

Method 9045D

Determination of pH Electrometrically
In Soil and Waste



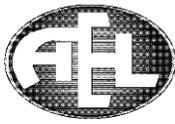


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1. IDENTIFICATION OF TEST METHOD

- 1.1 Determination of pH in sediment samples electrometrically using method EPA 9045D. Also, for the determination of corrosivity.

2. APPLICABLE MATRIX OR MATRICES

- 2.1 Method 9045D is applicable to soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample.

3. DETECTION LIMITS

- 3.1 Not Applicable.

4. SCOPE AND APPLICATION, INCLUDING COMPONENTS TO BE ANALYZED

- 4.1 Method 9045D is used for the electrometric determination of pH in soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. **If water is present, it must constitute less than 20% of the total volume of the sample, otherwise the sample must be analyzed by EPA 150.1/9040C.**

5. SUMMARY OF THE TEST METHOD

- 5.1 9045D: The sample is mixed with reagent water, and the pH of the resulting aqueous solution is measured as described above.

6. DEFINITIONS

See also AEL ADMIN SOP-039 Laboratory Definitions

- 6.1 pH: The term pH was derived from the manner in which the hydrogen ion concentration is calculated, it is the negative logarithm of the hydrogen ion (H⁺) concentration:

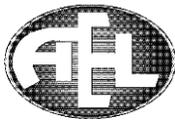
$$pH = \log_{10}(a_{H^+})$$

where log is a base-10 logarithm and a_{H⁺} is the activity (related to concentration) of hydrogen ions. According to the Compact Oxford English Dictionary, the "p" stands for the German word for "power", potenz, so pH is an abbreviation for "power of hydrogen". A higher pH means there are fewer free hydrogen ions, and that a change of one pH unit reflects a tenfold change in the concentrations of the hydrogen ion. For



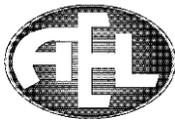
example, there are 10 times as many hydrogen ions available at a pH of 7 than at a pH of 8. The pH scale ranges from 0 to 14. A pH of 7 is considered to be neutral. Substances with pH of less than 7 are acidic and substances with pH greater than 7 are considered to be basic.

- 6.2 Corrosivity: Corrosives are defined in terms of pH by the EPA and have a $\text{pH} \leq 2$ or ≥ 12.5 . The DOT defines Corrosivity in terms of the substance's ability to cause visible destruction or changes in skin tissue at the site of contact or a liquid that has a severe corrosion rate on steel or aluminum. AEL and this SOP shall define Corrosivity as a measure synonymous with a reading of pH. pH units are used to report Corrosivity under this SOP. Other forms of Corrosivity such as microbiological, wear by fluid flow, and high temperature are not discussed or measured under this SOP.
- 6.3 Conductivity: also can be referred to as specific conductance, is a measurement of the electrical conductance per unit distance in an electrolytic or aqueous solution.
- 6.4 CCV: Continuing Calibration Verification - evaluated at the beginning of a 12- hour analytical shift, after every 10 samples, and end of an analytical batch to confirm calibration throughout the run. CCV's bracket each analytical run of up to 10 samples. Same source as the calibration.
- 6.5 ICV: Initial Calibration Verification – evaluated after the initial calibration of an instrument in order to verify the initial calibration. Second source standard to verify a calibration.
- 6.6 Hygroscopic – A sediment or solid material that absorbs liquid, reducing the amount of free liquid in the mixture.
- 6.7 Case Narrative (CN) -- A case narrative is simply a means of describing exactly what transpired with the samples during the analytical process. Case narratives are required for variances that occur within a project.
- 6.8 Non-Conformity Form (NCF) -- Form which will be completed and processed for each QC failure or deviation from normal protocol that occurs outside the scope of normal operation as defined by the AEL QM Section 10, AEL SOP Admin-016 and Method SOP.
- 6.9 Standard -- A solution prepared by diluting stock standard solutions used to calibrate the instrument response with respect to analyte concentrations. Also referred to as calibration standards (CALs) as in section 10.10.
- 6.10 Stock Standard -- A concentrated solution containing method analytes that is purchased from a commercial source having Certificates of Analysis.
- 6.11 Calibration Standard (CAL) -- A solution prepared from the primary dilution standard



solution and stock standard solutions of the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

- 6.12 Quality Control Sample (QCS) -- A sample matrix containing method analytes or a solution of method analytes in a water miscible solvent which is used to fortify reagent water or environmental samples. The QCS is obtained from a source external to the laboratory and is used to check laboratory performance with externally prepared test materials.
- 6.13 Safety Data Sheets (SDS): Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire and reactivity data, including storage, spill and handling precautions.
- 6.14 Analytical Batch: The set of samples started through the analytical process to a maximum of 20 samples.
- 6.15 Qualifier Codes (For Florida and FDEP work)
- 6.15.1. A - Value reported is the mean (average) of two or more determinations. This code shall be used if the results of two or more discrete and separate samples are averaged. These samples shall have been processed and analyzed (e.g., laboratory replicate samples, field duplicates, etc.) independently. Do not use this code if the data are the result of replicate analyses on the same sample aliquot, extract or digestate. Under most conditions, replicate values shall be reported as individual analyses.
- 6.15.2. I - The reported Value is between the laboratory method detection limit (MDL) and the laboratory practical quantitation limit (PQL).
- 6.15.3. K- Off scale low.
- 6.15.4. L- Off scale high. Use if reporting above the acceptable level of quantitation.
- 6.15.5. H - Value based on field kit determination; results may not be accurate. This code shall be used if a field-screening test (i.e. field gas chromatograph data, immunoassay, vendor-supplied field kit, etc.) was used to generate the value and the field kit or method has not been recognized by the Department as equivalent to laboratory methods.
- 6.15.6. O - Sampled, but analysis lost or not performed. Note: if reporting data to STORET, a numerical value must be entered. Such values are not meaningful and shall not be used.
- 6.15.7. Q - Sample held beyond the accepted holding time. This code shall be used if the



value is derived from a sample that was prepared and/or analyzed AFTER the approved holding time restrictions for sample preparation and analysis.

6.15.8. Y - The laboratory analysis was from an unpreserved or improperly preserved sample. The data may not be accurate.

6.15.9. REJ - Data is rejected and should not be used. Some or all of the quality control data for the analyte were outside criteria, and the presence or absence of the analyte cannot be determined from the data.

6.15.10. NAI - Not analyzed due to interference

6.15.11. J - Estimated value; value not accurate. This code shall be used in the following instances:

6.15.11.1. "1" Surrogate recovery limits have been exceeded.

6.15.11.2. "2" No known quality control criteria exists for the component.

6.15.11.3. "3" The reported value failed to meet the established quality control criteria for either precision or accuracy.

6.15.11.4. "4" The sample matrix interfered with the ability to make any accurate determination.

6.15.11.5. "5" The data is questionable because of improper laboratory or field protocols (e.g. composite sample was collected instead of a grab sample).

6.15.11.6. "J" value shall be accompanied by justification for its use.

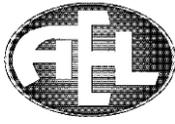
6.15.11.7. "J" value shall not be used if another code applies (ex. K, L, M, T, V, Y, PQL)

6.15.12. If more than one code applies, and the data is to be entered into STORET, only one code shall be reported. The code shall be selected based on the following hierarchy: REJ, NAI, O, Y, V, H, J, B, K, L, M, PQL, T, Z, A

7. INTERFERENCES

7.1 Coatings of oily material or particulate matter can impair electrode response.

7.2 The interference of temperature on the electrometric measurement of pH is overcome by calibrating the electrode –instrument system at the temperature of the samples.



8. SAFETY

- 8.1 Refer to the AEL Chemical Hygiene Plan and Safety Manual for safety precautions and for the Hygiene Plan and Emergency Response Plan.
- 8.2 See Standard Methods section 1090, 22nd edition-Laboratory Occupational Health and Safety.

9. EQUIPMENT AND SUPPLIES

Note: SOPs are updated on a set schedule and as a result may not reflect new additions or updates to laboratory equipment inventory at each site as the year progresses. For real-time equipment tracking, please reference the ADMIN-049a AEL QM Section 7.0 - Contemporary Equipment List (most recent revision) link on the intranet SOP system for the current listing by room location and letter designation for each piece of major equipment (by make, model, and serial number) and a full inventory of all major pieces of equipment in each lab.

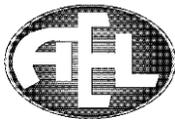
- 9.1 Laboratory pH meter with temperature compensation and accuracy to 0.05 pH units.
(In Jacksonville - Thermo Orion Model 115, S/N 003782)
- 9.2 Beakers or plastic cups.
- 9.3 Stir plate.
- 9.4 Stir bars.
- 9.5 Calibrated balance (For solids).
- 9.6 Quantitative filter paper, 18.5 cm (For solids).

10. REAGENTS

- 10.1 Primary calibration standards: 4.00, 7.00, 10.00 pH units. Also have available 2 and 12 pH standards when readings go below 4 or above 10 pH units.
- 10.2 Second source standard: 7.00 pH units.

11. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND HANDLING

- 11.1 See AEL QM Section 6.0 for sample acceptance policy.
- 11.2 See AEL Admin-005 and Admin-023
- 11.3 See FDEP SOP FS1000 for preservation requirements, shipping conditions and holding time requirements.



11.4 Samples should be analyzed as soon as possible, preferably in the field at the time of sampling.

11.5 Sample must be collected in a polyethylene or glass container, then cooled to $<6^{\circ}\text{C}$, if not analyzed immediately.

11.6 Sample Hold Time: 15 minutes/immediately.

12. QUALITY CONTROL

12.1 Each analyst must perform and pass their initial demonstration of capability (iDOC) prior to being able to perform this method. A statement must be signed prior to analysis that the most current SOP has been read and will be adhered to.

12.2 The iDOC consists of the analysis of four replicates of a laboratory control sample. The sample should be analyzed in accordance with the method procedure (Section 14.0) with the recovery of the four replicates having an RSD $<10\%$ and no result more than 0.1 pH units from the target.

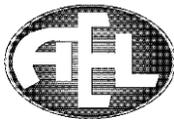
12.2.1 A sample can be classified as a solid when brought into the lab listed as a solid (such as a biosolid) with a % moisture content less than 50% and greater than 20% and can be analyzed under 9040.

12.2.2 An IDOC is required for this solids testing as well as for the aqueous matrix if solids testing is to be performed by the lab.

12.2.3 The IDOC for solids shall consist of 4 replicates of a 20gram matrix (such as Teflon chips or Ottawa sand) combined with 20mls of a pH buffer (such as a 4). In this example, the sample aliquot would have a 0% moisture content and an expected pH of 4. Each of these 4 replicates as they do not fall in the range of less than or equal to a pH of 2, or greater than or equal to a pH of 12.5, would be classified as non-corrosive. Note in the IDOC documentation that tested for corrosivity as well as pH and designate whether corrosive or non-corrosive. The average of the replicate study shall be within ± 0.20 pH units of true value to be considered passing.

12.3 When beginning the use of this method, on a per run basis to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of an initial calibration verification (ICV). If the determined concentrations do not meet the requirements listed above, the analysis should be stopped and the source of the problem corrected. If the analyst cannot correct or determine the source of the problem, contact the lab manager or QA Officer for further instructions.

ICV (7.00 pH units)



- 12.4 Each batch of 10 or fewer samples should begin and end with the analysis of a CCV. The CCV must be within 0.05 of true value. If the CCV fails to meet this criterion, stop the run, determine the source of the problem, correct the problem if possible and continue the run. If the problem is not immediately correctable, contact the lab manager or QA Officer for further instructions.

CCV (7.00 pH units)

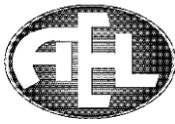
- 12.5 Each sample is first analyzed then a fresh aliquot of that sample is to be re-run until the readings are 0.1 pH units or less apart.

13. CALIBRATION AND STANDARDIZATION

- 13.1 Always keep pH probe wet. When not in use, keep probe immersed in a 7 buffer solution. When ready to use, rinse with DI water and pat with a kimwipe just prior to immersing into next solution to measure.
- 13.2 Calibration is required upon initial method set-up, and every 6 months or when the calibration verification checks read out of compliance, whichever occurs sooner. The meter calibration is verified daily with Buffers 4.0, 7.0, and 10.0 (per manufacturer instruction). The slope (if applicable) is recorded on the benchsheet.

13.3 Accumet Model 50:

- 13.3.1. Make sure electrode is plugged into the corresponding input channel being displayed on the screen. If they do not correspond, press CHANNEL until they do.
- 13.3.2. Press STANDARDIZE. This will display 2 choices. Press 2-CLEAR EXISTING STANDARDS.
- 13.3.3. Place the 4.00 buffer on the stir plate, insert electrode, and begin moderate stirring.
- 13.3.3.1.1. **NOTE:** Care must be taken that the stirring speed remains constant throughout the run. Leave until there is a stable reading.
- 13.3.3.1.2. **NOTE:** Care should be taken to make sure electrode is not struck by the stir bar.
- 13.3.4. Press STANDARDIZE. This will display 2 choices. Press 1-UPDATE OR ADD A STANDARD. This will display a new screen. Enter the value of the first buffer solution (4.00) using the number keys. Press ENTER and a new screen will be displayed. Press ENTER again. This will calibrate the meter at that value.
- 13.3.5. Repeat steps 13.1.3 – 13.1.4 for 7.00 and 10.00. When a complete calibration is done there will be three values on the left side of the screen.



13.3.5.1.1.1.1. **NOTE:** For corrosivity characterization, the calibration of the pH meter should include a buffer of pH 2.00 for acidic wastes and a pH 12.45 buffer for caustic wastes.

13.3.6. Read all three standards again to make sure that they are within 0.05 pH units of their true value. If all do, analysis of samples may begin. If not, meter must be recalibrated.

13.4 **Accumet Basic:**

13.4.1. Place the 4.00 buffer on the stir plate, insert electrode, and begin moderate stirring.

13.4.1.1.1. **NOTE:** Care must be taken that the stirring speed remains constant throughout the run. Leave until there is a stable reading.

13.4.1.1.2. **NOTE:** Care should be taken to make sure electrode is not struck by the stir bar.

13.4.2. Press STANDARDIZE. The '4' on the screen will begin to blink and then the screen will either display 'Good Electrode' or 'Electrode Error'. If the display is 'Good Electrode', continue the standardization process with the 7.00 and 10.00 buffers. If the display is 'Electrode Error', press ENTER and repeat the previous step until 'Good Electrode' is displayed on the screen.

13.4.3. Read all three standards again to make sure that they are within 0.05 pH units of their true value. If all do, analysis of samples may begin. If not, meter must be recalibrated.

13.5 **Corning 215**

13.5.1. Place the 4.00 buffer on the stir plate, immerse the electrode into the solution, and begin moderate stirring.

13.5.1.1.1. **NOTE:** Care must be taken that the stirring speed remains constant throughout the run. Leave until there is a stable reading.

13.5.1.1.2. **NOTE:** Care should be taken to make sure electrode is not struck by the stir bar.

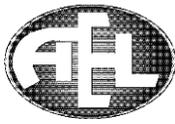
13.5.2. Record the pH of the buffer solution.

13.5.3. Repeat procedure 13.3.1 – 13.3.2 for the 7.00 and 10.00 buffer solutions.

13.5.3.1. All readings must be within 0.05 pH units of their true value. If they are, begin analysis of the samples. If not the meter must be recalibrated.

13.5.3.1.1. To recalibrate this meter, immerse the electrode into the 7.00 buffer solution and set the pH meter equal to 7.00 using the Cal 1 dial.

13.5.3.1.2. Immerse the electrode into the 4.00 buffer solution and set the pH meter equal to 4.00 using the Cal 2 dial.



13.5.3.1.3. Repeat steps 13.3.1 – 13.3.2 with all buffer solutions. If still not within 0.05 pH units of the target, contact the lab manager for further instructions.

13.6 Thermo Orion Aplus 920A+

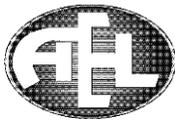
- 13.6.1. Press any key to take instrument off of standby mode
- 13.6.2. Press the calibrate key (#2 key).
- 13.6.3. Press 3 for number of buffers.
- 13.6.4. Place pH & temperature probes into the 4.00 buffer and wait for it to stabilize. (It will display RDY at the bottom of the screen
- 13.6.5. Enter 4.00, then press the 'Yes' key
- 13.6.6. Repeat for 7.00 & 10.00 buffers
- 13.6.7. Read all three standards again to make sure that they are within 0.05 pH units of their true value. If all do, analysis of samples may begin. If not, the meter must be recalibrated

13.7 VWR Symphony

- 13.7.1. In measurement mode, press the selection button until pH is selected
- 13.7.2. Press the calibration button. Rinse the probe with DI water and place it in the pH 4.00 buffer solution
- 13.7.3. Wait until the ph stops flashing. If the meter has automatically assigned the correct value to the calibration point, press the calibration button. Otherwise, use the arrow buttons to select the correct value and then press the calibration button
- 13.7.4. Rinse the probe with DI and repeat 13.5.3 with the pH 7.00 and 10.00 buffer solutions. Press the probe button to save the points and end the calibration
- 13.7.5. **Note:** For all curves, a second source standard must be run to verify the initial curve, every twenty samples, and at the end of the analytical batch. The independent calibration verification (ICV) must vary no greater than ± 0.1 pH units from expected true value.

13.8 Thermo Orion 115

- 13.8.1. Hit the "Cal" button and then fill measuring cup and measure the first buffer std of 4.0.
- 13.8.2. Wait until it stabilizes then remove the probe from the cup and rinse with di water.
- 13.8.3. Load up the 7 pH buffer and hit "Cal" again. Stabilize. Rinse repast with the 10 buffer.
- 13.8.4. Once the 10 buffer is stable, press "End" and "Save".



13.8.5. Verify curve with an ICV (7.0 pH) by hitting “Read”.

13.8.5.1.1. **Note:** For all curves a second source standard must be run to verify the initial curve, every ten samples, and at the end of the analytical batch. The independent calibration verification (ICV) must vary no greater than ± 0.1 pH units from expected true value.

13.9 From Relative Error. From the 2016 TNI Standards, the laboratory is required to use and document a measure of relative error in the calibration. By employing relative error acceptance criteria, concentrations calculated from the low end of the curve shall have the same confidence in their accuracy as those taken from any other point on the curve. Acceptance criteria must be met.

13.9.1. For calibrations evaluated using an average response factor, the determination of the relative standard deviation (RSD) is the measure of the relative error. If the average response factor acceptance criteria have been met, then acceptance is also met for the relative error.

13.9.2. For calibrations evaluated using correlation coefficient or coefficient of determination, the laboratory shall evaluate relative error by either the measurement of the Relative Error (%RE) or the measurement of the Relative Standard Error (%RSE)

13.9.3. Relative error is calculated using the following equation:

$$\% \text{ Relative Error} = \frac{x'_i - x_i}{x_i} \times 100$$

x_i = True value for the calibration standard

x'_i = Measured concentration of the calibration standard

13.9.3.1. This calculation shall be performed for two (2) calibration levels: the standard at or near the mid-point of the initial calibration and the standard at the lowest level. The mid level of the calibration curve shall pass the method specified or CCV criteria. The low level of the calibration curve shall pass the method specified criteria (+/-0.05SU).

13.9.4. Relative Standard Error (%RSE) is calculated using the following equation:

Relative Standard Error is calculated using the following equation:

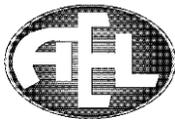
$$\% \text{ RSE} = 100 \times \sqrt{\frac{\sum_{i=1}^n \left[\frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}}$$

x_i = True value of the calibration level i

x'_i = Measured concentration of calibration level i

p = Number of terms in the fitting equation
(average = 1, linear = 2, quadratic = 3)

n = Number of calibration points

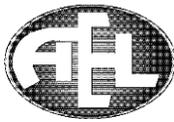


- 13.9.4.1. The RSE shall meet the criterion specified in the method. If no criterion is specified in the method, the maximum allowable RSE shall be numerically identical to the requirement for RSD in the method. If there is no specification for RSE or RSD in the method, then the RSE shall be default 20%.

14. PROCEDURE

14.1 Solid and Waste Sample Analysis:

- 14.1.1. Always keep pH probe wet. When not in use, keep probe immersed in a 7 buffer solution. When ready to use, rinse with DI water and pat with a kimwipe just prior to immersing into next solution to measure.
- 14.1.2. Make sure meter is calibrated (as per section 13) prior to analyzing samples and calibration is verified with an ICV.
- 14.1.3. Set meter to take readings. Follow manufacturer's instructions for taking pH measurements. Begin with QC analysis.
- 14.1.4. To 20g of soil or waste sample, add 20 mL of DI water and stir continuously for 5 minutes.
- 14.1.5. Allow the mixture to sit for one hour to allow most of the suspended material to settle out or filter or centrifuge off the aqueous phase for pH measurement.
- 14.1.6. Immerse the electrode into the mixture being careful not to allow the solid material to remix into the liquid.
- 14.1.7. After the reading has stabilized, record the pH of the liquid. This is defined as the pH of the sediment. Record the temperature also.
- 14.1.7.1. If the waste is hygroscopic, add an additional 20 mL of DI water to the mixture. Continue adding water until there is adequate free liquid to measure the pH. Record the volume of DI water and the mass of solid material used.
- 14.1.7.2. If the supernatant is multiphase, decant the oily phase and measure the pH of the aqueous phase.
- 14.1.8. Between every Buffer and sample, rinse the probe with DI water. Lightly pat with a Kimwipe to dry the probe prior to next standard or sample.
- 14.1.9. Run the Buffer 7.00 CCV at a frequency of every 10 samples and always close the batch with a passing CCV.
- 14.1.10. If testing for Corrositivity, the reported pH value will also include a statement indicating either "corrosive" or "non-corrosive" based upon the sample pH observed.



If the sample has a pH that is < 2 or > 12.5 then the sample will be noted as corrosive.

15. CALCULATIONS

$$15.1 \%RPD = \{|Value 1 - Value 2| \div [(Value 1 + Value 2)/2]\} \times 100\%$$

$$15.2\%RSD = \text{Standard Deviation (all results)} \div \text{Average (all results)} \times 100\%$$

15.2.1. The standard deviation and average may be determined by Excel spreadsheet, handheld calculator or by arithmetic calculations. Refer to the appropriate manual or textbook for determining the standard deviation and average of a set of values.

16. METHOD PERFORMANCE

16.1 The control limits for the recovery of the CCV are ± 0.05 pH Units.

16.2 The control limits for the recovery of the ICV are + 0.1 pH Units

16.3 Duplicates must agree within 0.1 pH units.

16.4 The Initial Demonstration of Capability (IDOC) must be successfully performed by each analyst, in accordance with ADMIN-030.

16.4.1. The IDOC consists of the analysis of four replicates of a solid sample. The sample should be analyzed in accordance with the method procedure (Section 14.0) with the recovery of the four replicates having an RSD <10%. The average of the replicate study shall be within +/- 0.20 pH units of true value to be considered passing.

17. POLLUTION PREVENTION

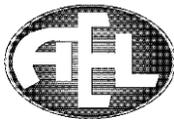
17.1 See Standards Methods section 1100, 22nd Edition-Waste Minimization and Disposal.

17.2 See SOP Admin-018 and the AEL Safety Manual.

18. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

18.1 The CCV and ICV must be within the acceptable limits.

18.2 The analyst is responsible for reviewing the results of the QC data to ensure it passes criteria.



19. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

19.1 If a CCV does not meet criteria, the CCV and samples associated with that CCV must be rerun. If after one additional reanalysis, the CCV still fails, contact the lab supervisor for further instruction.

19.2 If the ICV does not meet criteria, recalibrate the meter. If the ICV again fails, the reason for the failure must be determined. Seek out a supervisor for further instruction.

20. CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

20.1 For QC failures on samples that cannot be rerun under passing criteria, a non-conformity form (NCF) must be completed. Refer to the SOP for Non-Conformities (ADMIN-016).

21. WASTE MANAGEMENT

21.1 Refer to the SOP for waste management (ADMIN-018).

21.2 See Standards Methods section 1100, 22nd Edition—Waste Minimization and Disposal.

22. MAINTENANCE SCHEDULE

22.1 A regular schedule of maintenance is dictated more by the instrument checks than by any set schedule. Most maintenance is done in response to a failure of one of the QC checks done during the course of normal operation. These checks ensure that the instrument is working at the top of its performance and is proof that the instrument is in good working order.

22.2 The instrument calibration is checked daily as outlined in section 13.0. Failure of one of the calibration checks will result in a standardization and recalibration of the instrument. If continued failure occurs, the probe shall be thoroughly cleaned by rinsing multiple times with di water. If this does not resolve the problem, the probe shall be replaced.

22.3 The meter should exhibit a stable reading once equilibrium has been reached. If the readings are erratic or unstable, the instrument shall not be used until the condition has been corrected. Cleaning of the probe, replacement of the probe, or replacement of the meter may be necessary.

22.4 The GEL probes require no maintenance other than to keep them in aqueous solution. If a gel probe fails to perform correctly, no other course of action can be taken other than replacement.

22.5 Visual inspection of the meter, the wires connecting the meter to the probe, and the probe itself shall be done during normal use. If any condition such as a frayed or



corroded wire is seen, the condition shall be corrected as soon as possible or prior to use if the condition may affect the quality of the results.

23. REFERENCES

23.1 EPA Method 9045D rev 4, effective 8/2002

23.2 AEL Chemical Hygiene Plan and Safety Manual

23.3 AEL Quality Manual, most current revision.

23.4 AEL ADMIN SOPs

23.5 ADMIN-049a AEL QM Section 7.0 - Contemporary Equipment List (most recent revision)

23.6 Standard Methods 23rd edition.

23.7 TNI Standards 2016

23.8 ISO 17025: 2005 & 2017 Standards

23.9 DoD ELAP QSM, most current revision.

24. APPENDICES (Tables, Figures, Flowcharts, etc.)

24.1 Validation Data: See the employee files (stored electronically on the network or hardcopy in the laboratory's QA office) for the individuals for an acceptable initial demonstration of capability, which serves as validation data for this method in AEL.

Appendix B
Project Planning Session Documents

**Project Kick-off Teleconference – Final Meeting Minutes
Per- and Polyfluoroalkyl Substances (PFAS) Remedial Investigation (RI)
Yakima Training Center (YTC), WA**

**ECC Prime Contract No. W9124J-18-D-0004
Task Order No. W9124J-22-F-0144
Friday, 14 October 2022
0900-1000 HRS CST/1000-1100 HRS EST**

Meeting Attendees:

U.S. Army Environmental Command (USAEC)

Mr. Roger Walton, PE – Contracting Officer's Representative (COR)
Mr. Mike Brown, Environmental Support Manager (ESM)

Environmental Chemical Corporation (ECC)

Mr. Rob Wasserman, PG – Deputy Program Manager (PgM)
Mr. Tim Woods, PMP – Technical Subject Matter Expert (SME)
Ms. Audra Balson, PG – Project Manager (PM)
Ms. Grace Carmichael, PG – Assistant PM

Arcadis

Ms. Rhonda Stone, PMP – PgM, Tech SME
Mr. Alex Villhauer, PG – Project Geologist, Technical Lead
Ms. Jesse Hemmen, RG – Technical Team

Project Kick-off Teleconference Agenda is attached for reference.

Introductions

- Ms. Balson began with introductions.
- Mr. Walton inquired if Mission and Installation Contracting Command (MICC) representatives Mr. Cedric Hargrove and Mr. Daniel Nascimento accepted the invitation; Ms. Balson confirmed they did not.
- Mr. Walton introduced Mr. Brown as ESM for YTC and Joint Base Lewis-McCord (JBLM) and described their respective project roles. Mr. Walton will act as COR for a period and then Mr. Brown will step into the role at some point in the future. Mr. Brown will serve as primary point of contact (POC) for technical issues and day-to-day operations.
- Ms. Balson introduced ECC and Arcadis Teams.

Administrative Items

- Ms. Balson confirmed receipt of Certificate of Insurance (delivered through MICC); Rob provided a copy of the certificate to Mr. Walton via email for completeness.
- Ms. Balson reviewed Key Personnel; no changes are expected from ECC or Arcadis. If changes in key personnel become necessary, ECC will present the proposed replacement(s) to the MICC, the Contracting Officer (KO), and the COR for review and approval.
- Ms. Balson reviewed Project Stakeholders.
 - For YTC – Mr. Mark Mettler identified as Installation Restoration Program (IRP) Manager for both Installations; but not official POC of YTC.

- Mr. Walton stated that an Environmental Manager may be hired at YTC, but for now, the Department of Public Works (DPW) Director will be notified of field work and presence of crews on-site and will be invited to attend the Site Orientation Meeting.
- ECC will notify JBLM DPW representatives of the YTC Site Orientation Meeting.
- Mr. Brown stated that obtaining access to YTC for a single day visit is a simple process.
- Mr. Walton clarified the site is within U.S. Environmental Protection Agency (USEPA) Region 10 and stated that because the site is not listed as National Priority List (NPL), no involvement from USEPA is required at this time.
- Mr. Walton stated that for Washington State Department of Ecology (DOE), the representative is Mr. Greg Caron. Mr. Walton stated that the relationship between USAEC and DOE is complex, as the site operated in a RCRA-like manner for an extended period; in 2020, the project began operating under CERCLA.
- Mr. Walton stated that the USAEC will handle Public Affairs at this stage of the project, and questions/issues will be brought to Ms. Lalita (Lally) Laksbergs, designated USAEC PAO representative.

Project Management

- Ms. Balson stated that ECC will hold monthly status calls and increase frequency if needed, especially during active field work.
- ECC offered to host additional meetings with DOE if necessary.
- On-site Orientation meeting to be scheduled; Mr. Brown and Ms. Balson will coordinate off-line.
- YTC trainings reviewed; ECC to provide documentation for completed trainings.
- Ms. Balson reviewed deliverables; highest priority is Tech Memo Work Plan for Boundary Well Installation task.
- Mr. Walton stated his preference for deliverables is electronic format. Any hard copies should be reserved for large figures for in-person meetings. When applicable, Ms. Balson will inquire of DOE's preferred format for deliverables.
- Ms. Balson stated that a draft MPS and Project Schedule will be provided to Mr. Walton for review.
- Ms. Balson reviewed the invoice process.

Data Management

- Ms. Balson confirmed that data will be provided to USAEC as needed for upload to Headquarters Army Environmental System (HQAES). Mr. Walton stated that he prefers to use DoD SAFE-Link for receipt of documents with a file size exceeding the capacity of email transfer. ECC offered a SharePoint site for rapid file sharing to the Army and Arcadis.
- Ms. Balson inquired of the process for obtaining GIS files; Mr. Walton suggested contacting JBLM but to note the GIS technician is leaving very soon. Ms. Stone offered to share GIS files obtained during Arcadis' work on the Preliminary Assessment (PA)/ Site Investigation (SI).
- Mr. Walton stated that ECC should be prepared for submission of data packages outside of formal reports to comply with public affairs; ECC acknowledged the request.

Contract Objectives

- Ms. Balson reviewed the awarded tasks in the Performance Work Statement (PWS).
- Ms. Balson acknowledged that some areas of the site may require additional coordination, to ensure field work does not interfere with Installation missions (i.e., Fire Chief, Air Traffic Control).
- Mr. Walton stated that a project under contract with SIA may provide insight into optimal depths/screened intervals for Boundary Wells.
- Mr. Walton stated that he and Mr. Brown are in favor of collaborating as much as possible on the planning side to streamline project plans and shorten the review process.

- Mr. Walton stated that he will invite Mr. Caron of DOE into the planning process, especially with Boundary Well construction and placement.
- Ms. Balson reviewed Option tasks.
- Mr. Walton noted that the first bullet in Option Task Contract Line Item Number (CLIN) 1002 should have been removed from the PWS, which is associated with evaluating alternative water supplies. ECC and SIA should plan to participate in any monthly/bimonthly status calls that are scheduled by USAEC. Mr. Walton stated that the point-of-entry treatment (POET) approach would not be sufficient for a number of off-post wells, as PFAS concentrations were too high.
- Ms. Balson stated that the Dig Permit is a high priority.
- Mr. Brown stated that Installation security will add names of contractors to a roster, for entrance into YTC over an extended period.
- Ms. Balson confirmed that restricted areas will be reviewed during the site visit with Directorate of Plans, Training, Mobilization, and Security (DPTMS) and Range personnel.
- Ms. Balson inquired about the protocols for collecting photos during field work, on- and off-post. Mr. Brown stated that approval for photographs is obtained by submitting a memorandum through Security.
- Ms. Balson reviewed project deliverable target dates.
- Ms. Balson reviewed proposed field tasks as data driven activities that will be performed in phases.

General Project Sensitivities

- Ms. Balson stated that field teams will review PFAS sampling protocols and be aware of the high potential for cross-contamination from personal care products and equipment/supplies.
- Ms. Balson reiterated that off-post work and right-of-entry [ROE(s)] will likely be necessary but require elevated sensitivity and confidentiality from field crews and subcontractors.
- Mr. Walton stated that although it is not ideal to rely on private wells for generating definitive data sets, there is a robust network of existing wells, and to the extent possible, off-post work will be limited to what is absolutely necessary.
- Ms. Balson inquired of protocols for interactions with public, in the event crews are approached with sensitive questions; ECC offered to prepare an Information Sheet, with contact information for Ms. Laksbergs.
- Mr. Walton stated that USAEC has invested substantial effort in notifying the public that the Contract was awarded, that work is expected, and that it is a positive step toward resolution.
- Ms. Stone noted a lesson learned during previous sampling efforts – it was beneficial to be accompanied by a Government representative to handle inquiries from public during field work and prevent schedule delays to the sampling crew.

Analytical/Laboratory

- Ms. Balson stated that primary and secondary labs are in place to prevent delays due to capacity issues and/or equipment breakdowns. Mr. Walton inquired if the backup labs will be included in the Quality Assurance Project Plan (QAPP); Ms. Balson confirmed.
- Mr. Walton confirmed that PFAS analysis will be exclusively by Method 1633; Ms. Balson and Mr. Woods confirmed.

Wrap Up and Open Discussion

- No additional comments presented.

Action Items

1. Ms. Balson and Mr. Brown to schedule site visit/orientation meeting.
2. ECC and Arcadis to participate in preliminary technical discussion.
3. Ms. Balson to prepare and distribute meeting minutes.

Attachment: Project Kick-off Teleconference Agenda

Technical Approach Discussion – Final Meeting Minutes
Per- and Polyfluoroalkyl Substances (PFAS) Remedial Investigation (RI)
Yakima Training Center (YTC), WA

ECC Prime Contract No. W9124J-18-D-0004
Task Order No. W9124J-22-F-0144
Tuesday, 18 October 2022
1430-1530 HRS CST/1530-1630 HRS EST

Meeting Attendees:

U.S. Army Environmental Command (USAEC)

Mr. Roger Walton, PE – Contracting Officer’s Representative (COR)
Mr. Mike Brown, Environmental Support Manager (ESM)

Environmental Chemical Corporation (ECC)

Mr. Rob Wasserman, PG – Deputy Program Manager (PgM)
Mr. Tim Woods, PMP – Technical Subject Matter Expert (SME)
Ms. Audra Balson, PG – Project Manager (PM)
Ms. Grace Carmichael, PG – Assistant PM

Arcadis

Ms. Rhonda Stone, PMP – PgM, Tech SME
Mr. Joseph Quinnan, PE, PG – Senior Engineer
Mr. Alex Villhauer, PG – Project Geologist, Technical Lead
Ms. Jesse Hemmen, RG – Technical Team

Purpose

- To align USAEC and ECC/Arcadis expectations in the approach and schedule for the installation of boundary wells.
- To review the schedule content of the Technical Memorandum (Tech Memo) Work Plan deliverable.

Introductions

- Ms. Balson began with introductions, stated the purpose of the call, and reviewed/reiterated the objectives of the accelerated schedule.
- Ms. Hemmen stated high-level expectations of Arcadis with respect to data quality objectives (DQOs) of boundary wells and proposed schedule.
- Ms. Stone opened the discussion to USAEC to share zones and/or properties of elevated concern along the western site boundary.

Boundary Wells and Surface Geophysics

- Mr. Walton stated that public perception is the driving factor for the accelerated schedule and acknowledged that the timeframe for the Tech Memo Work Plan is limited but manageable. He stated that it is important that work begins soon, and he does not want to back off the acceleration of the schedule.

- Mr. Villhauer shared a figure showing proposed boundary well locations, which were tentatively placed to capture upgradient source areas and intercept groundwater flowing toward downgradient private wells.
- Mr. Villhauer stated that the boundary wells are strategically designed to generate a robust monitoring network, as well as provide lithologic and structural data that will be used for correlating with surface geophysical data sets.
- Mr. Villhauer and Ms. Hemmen discussed their proposed approach for the surface geophysical transects (seismic and resistivity methods).
- Mr. Quinnan followed up with a high-level discussion of the DQOs and stated that Arcadis will leverage the success of various technologies implemented at similar sites to optimize the approach for geophysics at YTC.
- Mr. Walton stated that the proposed wells along the north-south boundary and east-west boundary in the southern end of the site are not positioned to capture elevated concentrations in groundwater leaving the site to the southwest.
- Mr. Villhauer concurred that the Phase III off-Post sampling data demonstrate that the southwest corner is not fully captured, and that the conceptual model will be adjusted accordingly – both in spacing and location. If key transport zones are identified, locations can be adjusted accordingly.
- Mr. Wasserman reiterated that well locations should serve a specific purpose, with the intent to answer any questions that we pose. He acknowledged Mr. Walton’s experience and familiarity with the site and how it will be instrumental in formulating our approach around that foundational knowledge of existing conditions.
- Mr. Walton stated there is transport originating from north of Firing Center Road; two monitoring wells along the fence line with TCE concentrations were the trigger for off-post action. He stated that the predominance of impacts follow Schlagel Road and turn southwest, and outflow may be occurring in multiple zones, but depth data from SIA are still pending. Concentrations of 10 to 20 parts per trillion (ppt) were observed in this area, at much greater depths than what was observed during the Phase I residential well sampling. Subsequently, the PWS assumptions were generally based on what was observed in residential wells here.
- Mr. Walton stated that the proposed transect does not capture the southern property line, particularly the southwest corner of the site, where elevated concentrations are observed. He stated that the boundary wells are not married to the fence line and can be shifted inward as necessary to best represent groundwater quality flowing off-site to the southwest.
- Mr. Quinnan stated that Arcadis is currently working through the Phase III off-Post data and suggested that they reassess the placement of geophysical transects and proposed well locations based on those concentrations.
- Mr. Quinnan stated that the application of surface geophysics will be used to identify anomalies in the basalt and optimize well placement through the integration of multiple data sets.
- Mr. Walton advised not to wait too long on Phase III results, as chemical data were released. Generally speaking, exceedances were identified during Phase II sampling. With the exception of descriptive well logs, ECC/Arcadis should have everything they need.
- Ms. Stone suggested that some of the data sets may not have been available when the tech approach was drafted.
- Mr. Walton stated that the pending depth information is important and should be incorporated into well construction design.
- Ms. Balson confirmed the number of boundary wells with Mr. Walton – in the event that geophysics suggest fewer pathways, is there a benefit to reserving some capacity for wells inside the Installation? Mr. Walton confirmed that 15 wells will be installed eventually, but there is flexibility in execution and the number of mobilizations. He concurred with a phased, data-driven approach that will potentially allow more time for an iterative process.

- Mr. Quinnan stated that Arcadis will stage work to achieve the best value and review data continuously to address data gaps. Boundary wells can be prioritized, with groups of wells targeting highest concentrations be installed first.
- Mr. Wasserman stated that our approach will be data driven, with schedule being the main obstacle. In response, Mr. Walton stated that the Period of Performance (PoP) is listed in the PWS, but it may be pushed, provided the team has demonstrated some progress of field work and it will lead to more meaningful and usable data points. Mr. Walton will handle any no-cost PoP extensions with the MICC.
- Mr. Quinnan stated that a single surface geophysical transect is the simplest approach and suggested wrapping the proposed transect around the southeastern corridor to capture the subsurface structure in the southwestern portion of the site.
- Mr. Walton agreed that the transects may be oriented to cut the corner (perpendicular to groundwater flow toward the southwest); to date, that plane is currently most unknown and has been the most difficult to quantify.
- Mr. Walton stated that it is possible that an Area of Potential Interest (AOPI) was missed in southwest corner, and that accelerated transport from the fire training area was an unlikely source for the off-Post impacts observed in this area.
- Ms. Balson confirmed that southwest corner of site is free of restrictions and could accommodate an accelerated geophysics field effort. Mr. Walton stated that in the north, we may have some range schedules that will require coordination.
- Ms. Balson confirmed that proposed geophysical transects are positioned along the roadway and within the right-of-way, and shared concerns of interferences potentially compromising the quality of data.
- Mr. Villhauer discussed potential problems with vegetation and interferences.
- Mr. Brown is unsure of extent of vegetation along the roadway.
- Mr. Walton stated that we should not proceed further south of the east-west oriented property line, and that there are orchards to be aware of in the southern end of the site.
- Ms. Balson confirmed with Arcadis that they plan to have a field team member completing borehole geophysics in sequence with well installations.
- Mr. Walton advised ECC/Arcadis to stay inside the fence line and on Army property as much as possible. For unintrusive work, it is easier to obtain access; intrusive off-post work may take up to 6 months to obtain access.
- Mr. Wasserman stated that the off-Post right-of-entry (ROE) process may present a good reason to stagger drilling mobilizations if off-Post drilling becomes necessary, and the Army needs time to obtain ROE(s). However, Mr. Walton would prefer to hold a well in reserve before proceeding off-Post (there must be a very good reason to drill off-Post).
- Mr. Quinnan stated that reserving wells will be implemented to optimize well placement.
- Mr. Walton reminded ECC/Arcadis that the PWS Option tasks allow for additional wells to fill data gaps.
- Mr. Wasserman stated that ECC/Arcadis' approach also offer the flexibility for additional wells.
- Mr. Walton requested that the phased approach to field work align with the Milestone Payment Schedule (MPS).

Tech Memo Work Plan

- Ms. Stone acknowledged the 150-day PoP for the well installations and suggested that the 30 October 2022 submission date for the draft Tech Memo Work Plan may be too tight. In response, Mr. Walton stated that the target date is manageable and expected.
- Mr. Walton stated that the drilling component of the Tech Memo Work Plan is as simple as describing a Standard Operating Procedure (SOP), and that proposed well locations will be considered draft, with the expectation that some of the locations will need to be shifted due to access, utilities, and/or more recent data developments.
- Mr. Walton stated that the level of specificity required in the Tech Memo Work Plan is minimal, and he expects the concurrence process to proceed quickly.
- Ms. Balson reviewed high-level plan for completing the Tech Memo Work Plan, with flexibility and governing health and safety programs covered in other documents that will be referenced.
- Ms. Balson proposed a draft submission date to ECC by 24-25 October 2022.
- Ms. Hemmen stated that Arcadis' current strategy is to submit for internal/Senior review by 24 October 2022.
- Ms. Stone reiterated the PWS schedule and offered to expedite the review process by submitting to Mr. Quinnan concurrently with ECC. Ms. Balson concurred.
- Ms. Stone confirmed that the APP/HASP can be submitted after the Tech Memo Work Plan but must be completed with USAEC concurrence prior to mobilization.
- Ms. Balson stated that ECC is in the process of selecting a drilling contractor and discussed flexibility in well construction. There may be a benefit to leaving some of the wells open, with respect to fracture connectivity; these items were reserved for a separate call.
- Ms. Balson concluded the call.

Action Items

1. Arcadis to review the Phase III off-Post sampling data.
2. Arcadis to accelerate Tech Memo Work Plan Draft and submit to ECC for review by 25 October 2022.
3. Ms. Balson to prepare and distribute meeting minutes.

Site Orientation Visit – Final Meeting Minutes
Per- and Polyfluoroalkyl Substances (PFAS) Remedial Investigation (RI)
Yakima Training Center, WA
Building 248

ECC Prime Contract No. W9124J-18-D-0004
Task Order No. W9124J-22-F-0144
Wednesday, 16 November 2022
0830-1530 HRS PST

Meeting Attendees:

U.S. Army Environmental Command (USAEC)

Mr. Mike Brown, Environmental Support Manager (ESM)
Mr. Mark Mettler – JBLM/YTC IRP Program Manager

Yakima Training Center (YTC)

Mr. Vance Penn, Deputy Garrison Commander
Ms. Margaret Taaffe – Environmental Division Chief
Mr. Brian Lawrence – Compliance Program Manager

Washington State Department of Ecology (DOE)

Mr. Greg Caron, Hazardous Waste Manager (Afternoon session only)
Mr. Kurt Walker, Hydrogeologist, Hazardous Waste Division (Afternoon session only)

Environmental Chemical Corporation (ECC)

Ms. Audra Balson, PG – Project Manager (PM)
Mr. Tim Woods, PMP – Technical Subject Matter Expert (SME)

Arcadis

Ms. Jesse Hemmen, RG – Project Manager
Mr. Alex Villhauer, PG – Project Geologist, Technical Lead

Purpose

- To view the site, impacted properties, and surrounding topography.
- To discuss the approach, components, and timeline of the RI.
- To establish a path forward for involvement of stakeholders.
- To select dates for upcoming project planning meetings.

Introductions

- Mr. Brown began with introductions, stated the goals of the site orientation meeting, and reiterated that we are one team with a single mission.
- Individual attendees introduced themselves and provided a brief background of PFAS experience.

General Discussion

- Mr. Brown initiated the meeting as an informal discussion and project introduction. ECC reviewed the PWS and Contract for clarification on Option Task 1001 – Response Actions, to include point

of entry treatment (POET) system installations only. It is anticipated that the U.S. Environmental Protection Agency (USEPA) will issue the draft Maximum Contaminant Level (MCL) for PFOS/PFOA before the end of the year, which will lower the MCL from 70 parts per trillion (ppt) to less than 10 ppt for each contaminant.

- Ms. Taaffe stated that YTC is attempting to fill Environmental Division positions to support the PFAS RI.
- Mr. Penn stated that relations with the public are contentious, and the media has been involved. The Government has been actively moving the project forward, while trying to convey that site activities are a positive step in the investigation.
- Mr. Woods inquired about the breakdown of positive, negative, and neutral positions of the impacted property owners. Mr. Mettler stated that the breakdown of positive and negative feedback is not 50/50, but relations have improved as residents see the process starting. Roughly 60% of property owners are amicable; 20% are contentious; 10% not concerned at all and have declined bottled water service.
- Mr. Brown stated that the public is mainly concerned with hearing about USAEC's efforts to develop a plan, and when and how it will be implemented. One of the goals of today's meeting is to set some dates for collaborative planning meetings and timelines for public outreach. He stated that the homes that tested at 70 ppt or higher were contacted; however, there is more work to be done:
 - the new construction observed along Leininger Drive, with a well not previously sampled
 - the undetermined downgradient reach of the off-post plume, and
 - the potential for the revised USEPA MCL going into effect, and associated need to view homes to determine the feasibility of installing POET systems.
- Mr. Brown stated the intended timeline is to reach out to properties before 20 December 2022. He suggested attaching a flyer to the bottled water service deliveries and stressed the need for clear communication with the Garrison.
- Ms. Balson provided a high-level summary of current geophysical field activities, and the ECC/Arcadis Team's phased approach to subsequent RI activities.
- Mr. Brown reiterated the purpose of the accelerated geophysical schedule.
- Mr. Penn stated that the Garrison recently issued a contract for the Public Affairs Officer (PAO) position.
- Mr. Lawrence inquired about the Team's intent to notify residents of the upcoming drilling activities. Ms. Balson discussed preparing an information sheet for field teams to distribute if approached by residents.
- Mr. Brown stated that a local presence is important to demonstrate our concern for progress and understands there are challenges with communication. He is limited to what he can do and say in response to community concerns. Ms. Lally Laksbergs has compiled an email list to inform residents periodically, and the list can be expanded if necessary.

- Mr. Penn inquired how to best approach the occurrence of multi-well impacts and if there is a prescribed approach (i.e., playbook) to follow for YTC. Mr. Woods stated that lessons learned at other Installations will be evaluated and applied to YTC.
- Mr. Brown reiterated that the complex geology creates additional challenges and there is no single solution for impacted wells due to variable levels of impact. The objective is to identify primary contaminant flow paths.
- Mr. Penn stated that the media has been involved and may return to the site to observe boundary well installation activities. Some media have been more friendly than others, so crews should be prepared for both situations.
- Ms. Balson inquired how interactions with Installation and military personnel should be handled. Mr. Brown responded with the same approach as property owners.
- Mr. Woods discussed community groups formed for the neighboring JBLM PFAS RI and fact sheets that were developed, which he will distribute to the Team.
- Mr. Lawrence explained there is a communication barrier with the Hispanic community. Mr. Penn reiterated that communications and receptiveness to action has been most challenging with this group of impacted residents.
- Mr. Woods inquired about the protocols for impacted agricultural land; Mr. Brown stated that this component is challenging and requires specific notifications and alternative water supplies. Mr. Mettler stated that they are not far into the solution process with respect to addressing this component of the PWS at JBLM.
- Mr. Penn stated that YTC is not well known throughout the city, and most residents do not realize the Installation is still active. There is a small base employment population.
- Mr. Brown stated he expects some public reaction to when the USEPA issues the draft MCL, which may lead to additional Options being exercised under ECC's Contract No. W9124J22F0144 for POET system installs. The team will discuss what options need to be leveraged to ensure everyone is working toward solution as quickly as possible.
- Mr. Brown stated that USAEC has received conflicting information regarding when the draft MCL will be issued. The 20 December 2022 date is consistent with USEPA's original roadmap that would issue the revised MCL before the end of calendar year 2022. There is an expectation for USAEC to react regarding the 106 Installations with PFAS impacts, and they are prepared for additional unrest from public.
- Mr. Lawrence referenced the WA State Advisory Levels for PFOS and PFOA are 10 and 15 ppt, respectively (combined 25 ppt). Mr. Brown mentioned these limits have not been made law.
- Mr. Brown stated that USAEPA does not regulate private wells.
- Ms. Hemmen provided a background of the RI components, including the baseline sampling effort and focus on Areas of Potential Interest (AOPIs) during later phases for delineation and characterization.
- Mr. Lawrence identified two subsurface irrigation ditches, one of which is oriented perpendicular to original Transect A. He stated there appears to be a spike in PFAS concentrations when the

drainage ditches are flowing. The ditches are activated annually during the first week of April and shut down during the first week of November.

- Mr. Villhauer explained the baseline sampling efforts on and off-post (e.g., sampling media, frequency).
- Ms. Hemmen inquired of the ROE process as work progresses off-post.
- Mr. Brown inquired about the amount of publicly owned off-post land (i.e., rights-of-way [ROWs]) available for well installations. Mr. Lawrence and Mr. Mettler stated that space is very limited. The waste-water treatment plant in the area is available but is not impacted with PFAS.
- Mr. Villhauer provided an estimated timeline of 18 months for off-post work to begin. Mr. Brown stated that it may require that length of time for ROEs to be established.
- Mr. Lawrence inquired if there is a possibility of placing wells along the Selah air strip, as this is the location of the potable Selah well, which is impacted with PFAS concentrations above 70 ppt. He will provide a well construction log for the Selah well to ECC/Arcadis. Mr. Villhauer stated it is possible during later phases of work. The group discussed natural overland flow in the area of the air strip.
- Mr. Villhauer inquired about the need for UXO clearance in advance of intrusive activities. Ms. Taaffe confirmed that it is required but is included in the Dig Permit.
- Mr. Brown stated that data management will be a challenge and inquired how to appropriately include partners who would like to be involved. To date, the primary method has been use of spreadsheets and it has not been successful.
- Mr. Brown stated that JBLM personnel should be coming to sample all sources at YTC during the fourth quarter 2022 and data are to be shared with the Team. The sampling event will be organized by Ms. Becky Kowalski.
- Mr. Lawrence stated that the Yakama Nation should be kept informed as they anticipate return of their lands. A request was submitted to the National Tribal Council for YTC. YTC Government leadership will turn over before this exchange occurs, therefore communication pathways must be established in advance.
- Ms. Hemmen stated that Arcadis' data management platform has recently been upgraded with dynamic options. At a minimum, the interactive map feature can display GIS data in real time and access is per user basis. The digital CSM (DCSM) offers options to manipulate, view, interact, and edit/propose well locations.
- The group briefly discussed involvement of stakeholders and developing a relationship with them. The goal is to solicit added value to the planning process. Mr. Brown acknowledged that the Drinking Water Standard (10/15 ppt) is a point of contention for WA State Department of Health (DOH), because the action level associated with YTC is currently 70 ppt.
- Mr. Brown acknowledged that the U.S. Geological Survey (USGS) may want to assist; however, he would like to avoid duplicating efforts.
- Mr. Mettler inquired about the level of involvement with DOE. Mr. Brown responded it will be for collaborative discussions and major decision points.

- Mr. Woods suggested the possibility of amending Arcadis' Preliminary Assessment (AS)/Site Inspection (SI) Uniform Federal Policy – Quality Assurance Project Plan (UFP-QAPP) to expedite portions of the field effort until the RI UFP-QAPP is finalized.
- Ms. Hemmen inquired of any photo restrictions that apply to YTC and surrounding areas. Ms. Taaffe will compile the Memo for Record (MFR) for ECC/Arcadis to collect photos.
- Mr. Brown stated that he intends to follow up with Mr. Greg Caron of the WA State Department of Ecology (DOE) to progress the agreement with DOE, within the information sharing guidelines.

Afternoon Session (1200 to 1440 HRS)

- WA State DOE personnel arrive (Mr. Greg Caron and Mr. Kurt Walker)
- Ms. Taaffe provided a status update on the Dig Permit for Arcadis.
- Mr. Brown repeated introductions and individuals provided summary of background, level of PFAS experience and role in the project.
- Ms. Balson provided a summary of work in progress and subsequent RI phases and provided a description of upcoming deliverables – Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP) and Public Involvement Plan (PIP).
- Mr. Brown invited DOE to attend the first of several Technical Project Planning (TPP) meetings to be conducted in early January 2023, in advance of the boundary well installation work.
- Mr. Brown solicited the assistance of DOE in establishing the ROEs.
- Mr. Brown opened the discussion to DOE to share any questions or concerns.
- Mr. Caron inquired about USAEC's limitations for data sharing. He was pleased with the receipt of the planning maps and is interested to review Work Plans and schedules. He understood that timing on these first phases is an issue and as the regulators, they do not want to stand in the way of progress. DOE has frequent contact with the community.
- Mr. Caron spoke about the Public Participation Grant (PPG) and is waiting to hear the community's concerns for ways to best utilize funds from the PPG.
- Mr. Brown stated that he wants to make sure communication is handled properly, with respect to sharing information across stakeholders. He is required to vet every piece of information before sharing.
- Mr. Caron stated that he is sensitive to data sharing issues and acknowledged the conflict associated with USAEC's CERCLA approach to the RI and the State's RCRA model.
- Mr. Woods requested a description of the PPG; Mr. Caron stated that the PPG is a WA State fund reserved for impacted communities from contaminated sites. Some Yakima residents have approached DOE for assistance through the program, which allots \$60K annually to residents for private facilitators, consultant fees, etc. The funds cannot be used for analytical testing or well treatment.
- Mr. Brown stated there are still residents that need to be contacted.
- Mr. Mettler stated that bottled water service is underway. Mr. Caron inquired of the service provider. Mr. Mettler answered Culligan® Water and that the contract is handled by JBLM.

- Mr. Walker inquired about the network of wells sampled and trends of analytical data. Mr. Brown informed Mr. Walker that he was not able to distribute data immediately but is working on solution to share data electronically in real time.
- Mr. Brown provided a map for DOE personnel to view during the meeting only, which depicted properties at concentrations above the current MCL of 70 ppt (red) and properties above the WA State Advisory Level 10/15 ppt (orange).
- Mr. Lawrence stated that properties without a red or orange dot are connected to the Pomona irrigation system. Some of the neighbors have shared connections to the system.
- Mr. Brown stated that the red dot properties are priority for assessment and POET system installation. USAEC's estimated timeline is roughly 30 days from Site Orientation visit. We need collaborative solutions before Christmas.
- The group set a goal to identify the homes that could potentially be connected to Pomona water system.
- Arcadis provided cross-sections for DOE to view during the meeting that were prepared using available driller logs. Arcadis acknowledged that the data quality represented in the cross-sections was limited. A discussion of preferential pathways followed.
- Mr. Brown stated that from a public health point of view, no additional wells installations are permitted in the area. Mr. Caron concurred with USAEC's "durable solutions" method, acknowledging that the public may not always favor that approach. He mentioned a *Challenges and Next Steps briefing* for DOE's executive leadership team to occur in December 2022.
- The group discussed the first TPP with DOE, and tentatively proposed a meeting during the first two weeks of January 2023.
- Mr. Brown established expectations for DOE review timeframes for deliverables, and the group concurred with 10 days.
- Mr. Caron offered to host the first TPP meeting, acknowledging that it may be easier than getting access to the base.
- Mr. Walker suggested that chip samples be collected during boundary well installation and analyzed via X-ray fluorescence spectroscopy (XRF) at the WA State University's lab, as a means of precisely identifying geologic units.
- Mr. Villhauer provided an overview of the baseline sampling approach and anticipated media.
- Mr. Caron inquired of the feasibility of extending the baseline surface water sampling to the Yakima River. He stated that the public most frequently asks if the Yakima River is impacted. Mr. Woods acknowledged that sampling the river is risky due to the extensive watershed and potential for non-DoD PFAS sources to be captured in sample sets. Mr. Caron stated that any off-post surface water sampling would go a long way to address some of the public's concerns.
- Mr. Woods stated that Arcadis' previous PA/SI UFP-QAPP will be leveraged to expedite the baseline sampling timeline.

- Mr. Caron discussed a recent water supply survey for PFAS impacts that was conducted by DOH, and most sources were below the current MCL. He mentioned that the media is still active, and he receives calls often for interviews and newspaper articles.

Action Items

1. Mr. Woods to distribute the JBLM Fact Sheet for reference.
2. ECC/Arcadis to expedite sampling of the new construction well observed along Leininger Drive during the site visit.
3. USAEC to work with the Garrison on distributing agricultural notifications.
4. ECC and USAEC to select date of first TPP meeting.
5. Arcadis to provide a rough estimate of the number of off-post properties that may require ROE.
6. Arcadis to complete geophysics data acquisition and processing in advance of first TPP meeting.
7. ECC to prepare and distribute meeting minutes.

**Boundary Investigation Field Work Discussions – Final Meeting Minutes
Per- and Polyfluoroalkyl Substances (PFAS) Remedial Investigation (RI)
Yakima Training Center, WA**

ECC Prime Contract No. W9124J-18-D-0004

Task Order No. W9124J-22-F-0144

Monday, 14 November 2022

1030-1050 HRS CST / 1130-1150 HRS EST and 1400-1430 HRS CST / 1500-1530 HRS EST

Purpose

A two-part discussion to alert ECC of deviations from the Technical Memorandum Work Plan by Arcadis field crew and issues with the start of work on Monday morning, 14 November 2022, and to align expectations and path forward for resuming work along geophysical transects.

Part 1 Meeting Attendees:

U.S. Army Environmental Command (USAEC)

Mr. Roger Walton, PE – Contracting Officer’s Representative (COR)

Mr. Mike Brown, Environmental Support Manager (ESM)

Yakima Training Center (YTC)

Mr. Eric Brouwer – Director of Public Works

Environmental Chemical Corporation (ECC)

Ms. Audra Balson, PG – Project Manager (PM)

Part 2 Meeting Attendees:

U.S. Army Environmental Command (USAEC)

Mr. Roger Walton, PE – Contracting Officer’s Representative (COR)

Mr. Mike Brown, Environmental Support Manager (ESM)

Yakima Training Center (YTC)

Mr. Eric Brouwer – Director of Public Works

Environmental Chemical Corporation (ECC)

Ms. Audra Balson, PG – Project Manager (PM)

Mr. Rob Wasserman, PG – Deputy Program Manager

Mr. Tim Woods, PMP – Technical Subject Matter Expert (SME)

Arcadis

Ms. Rhonda Stone, PMP – Program Manager, Tech SME

Mr. Gregory Byer, PG, LPG – Geophysical Support

Ms. Jesse Hemmen, RG – Technical Support

Part 1

- Mr. Brown alerted Ms. Balson to an issue that involved the Arcadis geophysics field crew and impacts to the YTC Garrison. Field team leader Mr. Gabriel Hebert of Arcadis walked the transects with YTC personnel and identified issues with feasibility in completing the transects (i.e., rough terrain and dense vegetation). After determining that the locations of Transects A and C were problematic, Mr. Hebert communicated directly to the Garrison that the lines will be shifted.
- Ms. Balson shared a figure of the geophysical transects, as presented in the Final Technical Memorandum Work Plan.
- Mr. Brouwer provided a summary of his concerns – Arcadis directly proposed to shift Transect A to an east-west orientation along the transmission line inside the fence, due to rough terrain and dense ground vegetation. Mr. Brouwer stated he felt we could have anticipated this type of issue if crews had observed the terrain in advance. He stated that Arcadis also suggested shifting Transect C approximately 30 feet to the west. Ms. Balson inquired of the rationale for this shift, to which Eric responded for ease of access and to avoid paved areas.
- Mr. Brown stated that western shift of Transect C is favorable, due to less interference with the Garrison. Mr. Brouwer did not object to the change in position but expressed frustration with having to repeat utility clearance efforts in this area. He stated that Mr. Hebert's tone was overly assumptive, and he was doing his best to accommodate Arcadis' field work. Mr. Brouwer was not expecting a site visit until Wednesday, 16 November 2022, and therefore he did not expect work to begin before the meeting took place.
- Mr. Walton requested further explanation for the proposed shifts in the transects. Mr. Brouwer stated their primary rationale was the terrain.
- Ms. Balson acknowledged the breach in lines of communication between Arcadis field crews, the YTC Garrison, and USAEC. Ms. Balson stated that a stop work order will be placed immediately after the current call, particularly for work along Transects A and C.
- Mr. Walton recalled the planning discussions and stated he was relying on Arcadis' technical advice for the original placement of the transects.
- Mr. Brown stated that repeated efforts is a point of contention among USAEC and the Garrison and understood the Garrison's point of view in having to repeat utility clearances along Transect C. He concurred that the western shift in Transect C is optimal, but Arcadis crews will have to wait for lines to be cleared again, as many as three days.
- Mr. Brown inquired about the concurrence status for Transect B. Mr. Brouwer expects the location to work but is waiting on Network Enterprise Center (NEC) to officially provide clearance. He stated that Transect B at 4th ½ Avenue and southward is feasible; however, the Parade field to the north of that point contains shallow fiber lines. Mr. Brouwer will request concurrence from the NEC but does not see any major red flags.
- Ms. Balson offered to schedule a follow up call later today to include additional ECC and Arcadis personnel. Mr. Brouwer accepted the invitation and set a call time of 1200 HRS PST / 1400 HRS CST / 1500 HRS EST.
- Ms. Balson reiterated that Arcadis will be instructed to stop work immediately following this conference call.

Part 2

- Ms. Balson requested that Mr. Brouwer describe the events of the morning and the tone of field crews. Mr. Brouwer repeated the issues as described above.
- Ms. Stone acknowledged the actions of the Arcadis field crew and provided some additional detail regarding the perception of the field crew and objective to stay on task. She offered the option to leave transects in their original locations but take additional time to alter the terrain and vegetation along Transect A to allow access (i.e., construct a road along the diagonal).
- Mr. Brouwer stated that moving Transect A is not a burden and the revised location for Transect C is more convenient for the Garrison and will not compromise the existing infrastructure. He stated that these items should have been considered during the planning process, not when the field team arrived on-post. Ms. Stone concurred and referenced the compressed schedule and upcoming holidays that are impacting the Boundary Investigation. She acknowledged that communication was not handled well by field crews.
- Mr. Brouwer requested that a YTC representative (Mr. Peter Nissen, or himself) be invited to all future planning meetings. Mr. Walton and Mr. Brown concurred.
- Mr. Brown resumed the discussion of the transects and inquired if the westward shift of Transect C was preferable. Mr. Walton followed by referencing the extensive discussion over the potential interference from the fence line. Mr. Byer stated that Mr. Hebert used his professional judgement to place the transect the minimum distance from the fence and acknowledged the conflict between using Google Earth to plan vs. field truthing methods.
- Mr. Brown confirmed with Mr. Brouwer that he preferred Transect C be shifted westward.
- Mr. Brown requested that Arcadis prepare and submit to ECC an updated KMZ file (or similar) to show the revised transect locations.
- Ms. Stone and Ms. Hemmen opened the discussion to the impact of revised Transect A on the data quality objectives (DQOs). Ms. Hemmen acknowledged that the angle would be an ideal orientation, but an east-west transect will still achieve the DQOs. Mr. Walton stated that the transect was not angled for optimal orientation, but to split the distance between the source areas and the impacted off-post properties. The wider distance will increase the margin of error. Ms. Hemmen referenced a possible north-south trending fault line that may be identified by the revised Transect A data sets. She acknowledged that distance is part of the equation, but equally important is geologic structure.
- Ms. Balson inquired of utility clearance along Transect A, to which Mr. Brouwer responded that he will check with local NEC personnel. He will not proceed with any further utility clearance activities until revised transects are finalized.
- Mr. Brouwer stated that Arcadis has permission to proceed with painting the electrode/geophone points along Transects B and C for utility clearance purposes.
- Mr. Wasserman clarified the status of each transect and acknowledged the Garrison's preference to be included in the planning process.
- Mr. Walton stated he is comfortable with crews remaining on site but requested that he reviews the revised transects before work along Transects A or C resumes. He expressed frustration over the events of the previous six days and stated that the Team has maximized the limits of the Garrison.

- Ms. Stone stated that Arcadis will expedite the placement of the revised transects and requested clarification on the amount of clearance necessary. Mr. Walton stated that all lines require at least some level of utility clearance.
- Mr. Byer provided a brief explanation of the limited penetration of seismic geophones.
- Mr. Walton stated there are other entities that need to be contacted besides the Garrison for proper utility clearance.
- Ms. Balson summarized the action items. Mr. Wasserman confirmed that only Transect B is approved for crews to proceed at this time.
- Mr. Brouwer stated that he appreciated the pause in work activities today.
- Ms. Stone expressed gratitude to the USAEC for accommodating the meeting on short notice, and for allowing the Arcadis team flexibility with repositioning the transects to ensure work would be completed.

Action Items

1. Arcadis to provide figure showing revised transects to ECC within 12 to 24 hours.
2. Ms. Balson to forward to USAEC and the YTC Garrison for review and concurrence. Revised geophysical transects submitted on the evening of 14 November 2022.
3. Ms. Balson to submit to USAEC the Technical Memorandum Work Plan, Revision 1 showing the revised transect locations.

Site Orientation Visit Debrief – Final Meeting Minutes
Per- and Polyfluoroalkyl Substances (PFAS) Remedial Investigation (RI)
Yakima Training Center, WA

ECC Prime Contract No. W9124J-18-D-0004
Task Order No. W9124J-22-F-0144
Monday, 21 November 2022
0930-1030 HRS CST / 1030-1130 HRS EST

Meeting Attendees:

U.S. Army Environmental Command (USAEC)

Mr. Roger Walton, PE – Contracting Officer’s Representative (COR)

Mr. Mike Brown, Environmental Support Manager (ESM)

Environmental Chemical Corporation (ECC)

Mr. Rob Wasserman, PG – Deputy Program Manager (PgM)

Ms. Audra Balson, PG – Project Manager (PM)

Mr. Tim Woods, PMP – Technical Subject Matter Expert (SME)

Arcadis

Ms. Rhonda Stone, PMP – PgM, Technical SME

Ms. Jesse Hemmen, RG – Project Support

Mr. Alex Villhauer, PG – Technical Lead

Purpose

- To review objectives and outcome of Site Orientation Visit.
- To discuss timing and content for the first Technical Project Planning (TPP) meeting.

General Discussion

- Ms. Balson provided a summary of the Site Orientation Visit.
- Mr. Walton clarified that Option 1001 task refers only to POET system installations and does not include bottled water service.
- Ms. Balson discussed the anticipated release of USEPA’s revised MCL for PFOS/PFOA, from the current 70 parts per trillion (ppt) to single digit levels for both contaminants.
- Ms. Balson discussed the map and content that was shared with WA State DOE during the meeting. Mr. Brown followed with an explanation of his limitations in data sharing at this phase of the project.
- Ms. Balson and Mr. Brown discussed the general findings of the windshield survey performed on 17 November 2022. Items of interest included but were not limited to:
 - Proximity of private wells to the Pomona system water mains
 - Feasibility of POET system installations.
- The group discussed the objectives of the geophysical survey, and how data sets will be used to shed light on the conceptual site model. According to maps prepared by USAEC, the impacted properties appear to be somewhat randomly distributed, owing in part to the depiction of properties

connected to the Pomona Water Supply interspersed with private wells at concentrations above the current MCL.

- Mr. Brown relayed information received by YTC Deputy Garrison Commander (DGC), Mr. Vance Penn that the Parade Field must not be disturbed during the investigation activities.
- Mr. Brown initiated a discussion of the irrigation canals that pass through the Installation, many of which are unlined. Mr. Kurt Walker of WA State DOE provided information regarding the channels during the Site Orientation meeting – there are two separate systems that discharge into the Yakima River.
- Mr. Villhauer discussed a verbal report from YTC personnel of perched water encountered during construction of the newer Fire Station, estimated at 4 ft bgs.
- Ms. Balson initiated a discussion of the new construction observed during the windshield survey, with a potable well not previously sampled. Mr. Brown stated there are other additional wells in the area that still need to be sampled.
- Mr. Walton stated that JBLM personnel are scheduled to perform a quarterly sampling event at YTC before year end.
- Ms. Hemmen provided an update of the geophysics program progress – data acquisition was completed, and crews demobilized on 19 November 2022. The freight was scheduled for FedEx pickup on 21 November 2022.
- The first TPP meeting was tentatively scheduled for Wednesday, 11 January 2023, to be hosted by WA State DOE.
- Mr. Walton inquired about the content of the TPP meeting. He would like to maximize the discussion points with the regulators. Topics to be discussed include but are not limited to:
 - Placement of boundary wells
 - Influence of on-post irrigation channels and drainage
 - Drilling and baseline sampling schedule
 - Areas of potential interest (AOPIs).
- Ms. Balson inquired about the schedule for distribution of agricultural notifications. Mr. Brown stated they are scheduled to go out this week (21 November 2022).
- Mr. Woods confirmed that Mr. Walton is still in concurrence with the data-driven, phased approach to boundary well installation, which may require multiple mobilizations.
- Ms. Balson inquired of the anticipated schedule for geophysics data processing. Mr. Villhauer responded two to three weeks (16 December 2022).
- A meeting was scheduled for Monday, 16 December 2022 to discuss the geophysical findings internally before presenting data to the regulators.

Action Items

1. USAEC to confirm the tentative 11 January 2023 date for the first TPP meeting with the YTC Garrison and WA State DOE.
2. ECC to draft an agenda for the upcoming TPP meeting.
3. Arcadis to process geophysical data sets.
4. ECC to prepare and distribute meeting minutes.

**Boundary Geophysical Survey Findings – Final Meeting Minutes
Per- and Polyfluoroalkyl Substances (PFAS) Remedial Investigation (RI)
Yakima Training Center (YTC), WA**

**ECC Prime Contract No. W9124J-18-D-0004
Task Order No. W9124J-22-F-0144
Monday, 19 December 2022
1000-1100 HRS CST / 1100-1200 HRS EST**

Meeting Attendees:

USAEC

Mr. Roger Walton, PE – Contracting Officer’s Representative (COR)
Mr. Mike Brown, Environmental Support Manager (ESM)

ECC

Ms. Audra Balson, PG – Project Manager (PM)
Mr. Tim Woods, PMP – PFAS Subject Matter Expert (SME)
Mr. Rob Wasserman – Deputy Program Manager

Arcadis

Ms. Rhonda Stone, PMP – Program Manager, Tech SME
Ms. Kimmie Schrupp, PMP – Technical Support
Ms. Jesse Hemmen, RG – Technical Support
Mr. Gregory Byer, PG, LPG – Geophysical Support
Mr. Alex Villhauer, PG – Project Geologist, Technical Lead
Mr. Joseph Quinnan, PE, PG – Senior Engineer

Discussion Points

- **Boundary Geophysical Survey Findings**
 - Arcadis presented an overview of the complementary survey methods performed (resistivity and seismic), and shared interpreted data plots for each transect, which depicted local geologic conditions and identified key features in the subsurface. Eight locations were recommended for well installation during the first mobilization. These locations will be presented to the WA State Dept. of Ecology during the first Technical Project Planning (TPP) meeting, scheduled for 11 January 2023.

Action Items

1. ECC to provide a copy of the presentation to USAEC via DoD SAFE Link.
2. Arcadis to prepare figures with off-post data layer removed (i.e., private well locations/depths, and PFAS concentrations). Additional edits may be incorporated after USAEC completes a follow up review before material is presented at the TPP meeting.

Pre-Technical Project Planning Meeting – Final Minutes
Per- and Polyfluoroalkyl Substances (PFAS) Remedial Investigation (RI)
Yakima Training Center (YTC), WA

ECC Prime Contract No. W9124J-18-D-0004
Delivery Order No. W9124J-22-F-0144
Tuesday, 10 January 2023
0900-1130 HRS PST

Purpose

At the request of the U.S. Army Environmental Command (USAEC), Environmental Chemical Corporation (ECC) and Arcadis presented the findings of the surface geophysical survey to the YTC Garrison and discussed logistics and planning for the upcoming Baseline sampling event and well installation tasks.

Meeting Attendees:

USAEC

Mr. Roger Walton, PE – Contracting Officer’s Representative (COR)

Mr. Mike Brown, Environmental Support Manager (ESM)

YTC

Mr. Vance Penn, Deputy Garrison Commander

Lt. Col. Tim Horn

Mr. Eric Brouwer – Director of Public Works

Ms. Margaret Taaffe – Environmental Division Chief

Mr. Mark Mettler – JBLM/YTC IRP Program Manager

ECC

Ms. Audra Balson, PG – Project Manager (PM)

Mr. Tim Woods, PMP – PFAS Subject Matter Expert (SME)

Arcadis

Ms. Jesse Hemmen – Technical Support

Mr. Alex Villhauer – Technical Support

Mr. Greg Byer – Geophysics SME

Discussion Points

- ECC/Arcadis Team presented the findings of the surface geophysical survey. Several anomalies were identified along the three transects, pertaining to both lithology and structure of the subsurface.
- Eight locations were selected for well installation during Mobilization #1 (tentatively scheduled for late February/early March 2023), based on review and correlation of existing well logs, stratigraphy, groundwater chemistry, and observations made during the field-truthing event. The locations were presented to the Garrison and staked/painted in the field for future utility clearance efforts. Geologic and borehole geophysical logs prepared during the first mobilization will be used to inform the selection of additional locations for remaining boundary and other monitoring wells.
- ECC/Arcadis provided a summary of the Baseline sampling event, well installation activities, and the anticipated schedule over the next several months.
- ECC will maintain close communication with USAEC and the YTC Garrison as mobilization dates near.
- ECC to initiate the necessary dig permits, and coordination with the YTC Airfield as needed.

- Before leaving the Installation, ECC/Arcadis met with the YTC Garrison regarding the staked locations of boundary wells along Transect C. Obstructions (i.e., overhead power lines, chain-link fence, storm water retention basin) are anticipated to require adjustments to well locations. ECC/Arcadis will coordinate with the Garrison on selection of final drilling locations.

**Technical Project Planning (TPP) Meeting – Final Minutes
Per- and Polyfluoroalkyl Substances (PFAS) Remedial Investigation (RI)
Yakima Training Center (YTC), WA**

**ECC Prime Contract No. W9124J-18-D-0004
Delivery Order No. W9124J-22-F-0144
Wednesday, 11 January 2023
0900-1100 HRS PST**

Purpose

U.S. Army Environmental Command (USAEC), Environmental Chemical Corporation (ECC) and Arcadis presented the findings of the surface geophysical survey to the Washington State Department of Ecology (Ecology) and discussed the upcoming Baseline sampling event, well installation activities, and a tentative project schedule.

Meeting Attendees:

USAEC

Mr. Roger Walton, PE – Contracting Officer’s Representative (COR)
Mr. Mike Brown, Environmental Support Manager (ESM)

Ecology

Mr. Greg Caron – Hazardous Waste Manager
Mr. Kurt Walker – Hydrogeologist/Site Manager

ECC

Ms. Audra Balson, PG – Project Manager (PM)
Mr. Tim Woods, PMP – PFAS Subject Matter Expert (SME)

Arcadis

Ms. Jesse Hemmen – Technical Support
Mr. Alex Villhauer – Technical Support
Mr. Greg Byer – Geophysics SME

Discussion Points

- Group acknowledged that no regulatory updates have been released yet regarding the current USEPA MCL for PFAS (70 ppt).
- ECC/Arcadis Team presented the findings of the surface geophysical survey. Several anomalies were identified along the three transects, pertaining to both lithology and structure of the subsurface. Mr. Walker provided insight into the interpretation of various structural anomalies.
- Eight locations were selected for well installation during Mobilization #1 (tentatively scheduled for late February/early March 2023), based on review and correlation of existing well logs, stratigraphy, groundwater chemistry, and observations made during the field-truthing event. Geologic and borehole geophysical logs prepared during the first mobilization will be used to inform the selection of additional locations for remaining boundary and/or other monitoring wells.
- ECC/Arcadis to submit draft of the Baseline Sampling Work Plan, for USAEC to review and share with Ecology.
- ECC/Arcadis provided a summary of the Baseline sampling event and the anticipated schedule over the next several months.
- Ecology will be kept informed of the schedule and other project developments, as available.

Baseline Sampling Event Kickoff Meeting – Final Minutes
Per- and Polyfluoroalkyl Substances (PFAS) Remedial Investigation (RI)
Yakima Training Center (YTC), WA

ECC Prime Contract No. W9124J-18-D-0004
Delivery Order No. W9124J-22-F-0144
Monday, 30 January 2023
0900-0930 PST / 1200-1230 HRS EST

Meeting Attendees:

USAEC

Mr. Mike Brown, Environmental Support Manager (ESM)

YTC

Mr. Eric Brouwer – Director of Public Works

Ms. Margaret Taaffe – Environmental Division Chief

Mr. Mark Mettler – JBLM/YTC IRP Program Manager

ECC

Ms. Audra Balson, PG – Project Manager (PM)

Ms. Grace Carmichael – Asst. PM

Ms. Courtney Bigelow – Technical Support

Ms. Bre Pollard – Technical Support

Arcadis

Ms. Jesse Hemmen – Technical Support

Mr. Alex Villhauer – Technical Support

Discussion Points

- ECC reviewed the schedule for Baseline Sampling, beginning on Monday, 06 Feb 2023. A copy of the Field Team Brief was provided to DPW/Environmental for reference.
- Group discussed long-term equipment storage options on-post; corner of Refractometer Testing lot selected for lay-down area for ECC (20-ft Conex storage box, secondary containment for temporary staging of IDW drums, etc.); 24-hour access to lot. Vendors must use care when off-loading equipment to prevent damage to asphalt. DPW will provide secondary containment for IDW drum(s).
- DPW provided a contact for Range Control and instructed ECC to check in prior to accessing Selah Airstrip. ECC checked in with Scott Blodget and Joe Richardson on Thursday, 02 Feb 2023 to arrange for sampling at Selah Airstrip on Wednesday morning, 08 Feb 2023.
- DPW provided a contact for the YTC Fire Department. ECC checked in with Chief Chris Dykstra on Monday, 30 Jan 2023 for assistance with hydrant sampling.

**Final Minutes: PFAS Sampling and Treatment Forum – Alignment and Path Forward
Joint Base Lewis-McChord (JBLM), WA (Day 2 only)
Yakima Training Center (YTC), WA**

**Wednesday, 19 April 2023 0900-1500 PST
Thursday, 20 April 2023 0800-1500 PST**

Meeting Attendees:

US Army Environmental Command

Mr. Roger Walton, PE, Contracting Officer's Representative (COR)
Mr. Mike Brown, Environmental Support Manager (ESM)
Captain Bud Bateman, U.S. Army, USAEC Intern

YTC Garrison

Ms. Guadalupe (Lupe) Lara, Department of Public Works (DPW) PFAS Engineer
Mr. Paul Noel, Public Affairs Officer (PAO)

JBLM Garrison

Mr. Mark Mettler, Installation Restoration Program (IRP) Manager
Ms. Tia Misuraca, Installation Readiness and Impact Analysis (AGEISS)

US Army Engineering and Support Center

Ms. Allyson Charbonnet, Chemist and Program Manager

US Army Corps of Engineers (USACE)

Mr. Jeff Weiss, Hydrogeologist
Mr. Jake Williams, Program Manager
Ms. Meseret Ghebreslassie, Remediation Project Manager

US Geological Survey (USGS)

Mr. Jackson Mitchell, Hydrogeologist
Ms. Wendy Welch, Supervisory Hydrologist

Environmental Chemical Corporation (ECC)

Ms. Audra Balson, PG – Project Manager (PM)

Arcadis

Ms. Rhonda Stone, PMP, Program Manager
Ms. Kimmie Schrupp, PMP, PFAS Subject Matter Expert (SME)
Ms. Jesse Hemmen, RG, PM
Mr. Eric Killenbeck, PFAS SME
Mr. Joe Quinnan, North American Director of Emerging Contaminants

Tanaq Environmental

Mr. Nick Alfino, PE, Emerging Contaminants Program Manager

Purpose – A multifold discussion on the challenges and implications associated with Point of Entry Treatment (POET) systems as a means of remediating PFAS-impacted drinking water; to collaborate on lessons learned through similar case studies and applying those lessons to the unique conditions associated with YTC; and to develop a solid path forward for on- and off-post sampling, keeping the community informed on Remedial Investigation (RI) progress, and the timing of the upcoming initial five POET system installations.

Discussion Points

1. General Concerns and Public Perception

- Homeowners feel they are not being kept informed of the investigation and have implied that action by the Army is too slow.
- With respect to public inquiries, Washington State Department of Ecology (Ecology) maintains that they cannot verify that garden produce, chicken eggs, etc. are safe for consumption.
- Some property owners with combined or individual PFOA and PFOS concentrations just below the 70 parts per trillion (ppt) threshold are concerned and feel they should be provided with bottled water.
- Some residents have installed insufficient granular activated carbon (GAC) treatment units independently and have requested reimbursement from the Government. Some do not want their existing treatment system replaced and there is a general lack of understanding that Government supplied POETs include operation and maintenance (O&M).

2. Potential Issues with POET Systems

- POETs are not a one-size fits all solution; each will require an individual design.
- Long-term challenges with O&M of GAC units, such as frequency of carbon change-outs, quantifying water use, and diminished water pressure.
- Proposed POETs are likely not equipped to handle agricultural or irrigation use.
- The frequency of carbon filter change-outs may increase if/when the proposed Maximum Contaminant Level (MCL) of 4 ppt is enforced.
- How will complaints associated with reduced flow be handled?
- Case studies show that some homeowners have installed water softeners post-treatment, which further reduces flow/pressure. Unapproved post-treatment modifications are a concern and may require system components to be secured.
- The installation of water softeners in conjunction with the POETs is not planned. Other agencies have included food chain pathways in remedial investigation, for example, by sampling eggs from local chickens.
- POETs are sensitive to geochemistry. The USGS is actively working on establish a geochemistry baseline.

3. Concerns associated with the Design, Installation, and Use of POETs

- Booster pumps may be required for each system, but these may not be effective for multiple POETs on a shared well.
- For properties that require the installation of a shed to house the POET, how will the shed be supplied with power/temperature control?
- How will seasonally occupied homes be handled with respect to carbon-filter change-outs, and power supplied to a shed (i.e., potential for freeze/thaw damage).
- Information from former Reese Air Force Base (AFB) – homeowners reported a strong sulfur odor associated with the treatment systems; likely caused by bacterial growth. How can we determine the source of the odor and prevent it in future POETs? Disinfection is a critical component.
- No studies to date to gauge individual water use for the design of individual POETs.
- Water use has not been quantified for agricultural, gardening, and or other irrigation activities.

- The typical two to three-cubic feet POET system would not support high volume agricultural use, shared water systems, and homes with PFOS/PFOA concentrations exceeding 1,000 ppt will require frequent monitoring.

4. Access Agreements

- USAEC and YTC Garrison will partner to update all required Rights of Entry (ROE). The anticipated timeframe is two to six months.
- There is a need for Spanish/English translators during the ROE process.

5. Off-post Sampling

- USAEC is anticipating Army policy to maintain a quarterly sampling schedule that has not been issued to date. Previous off-post sampling efforts included as many homes as possible, and USAEC has not turned down any sampling requests.
- A USEPA Federal attorney is leading the current off-post sampling effort and an EPA contractor will conduct the off-post sampling. Region 10 has no involvement to the Army's knowledge.
- Due to the extended laboratory analytical periods associated with PFAS, sampling periods may overlap and lead to confusion for homeowners with respect to the distribution of analytical results.
- Homeowners may experience fatigue from the intrusion of congruent sampling programs (USAEC Contractors, USGS, and USEPA).
- Bottled water service will remain available to those currently in the program, even if quarterly concentrations fluctuate below the threshold of 70 ppt.
- Sampling results from each event will be provided to the homeowner.
- For on-post water systems, aqueous samples must be reported as non-detect (ND) for eight consecutive quarterly events to be designated as 'clean'.
- Ecology applied for a grant to provide homes with concentrations below 70 ppt with a Brita® filter (or similar).
- A case study showed seasonal variability in groundwater flow, which resulted in 30 to 40 ppt fluctuation in PFOS/PFOA concentrations. It was suggested that the newly installed boundary wells be sampled quarterly to evaluate seasonal trends, as these are true monitoring wells and will more accurately represent undisturbed conditions. If boundary wells show seasonal variability, the quarterly monitoring schedule can be extended to select off-post private wells. The USAEC needs to provide justification for looking for seasonal trends.
- The criteria specified for bottled water service was a hard line. One exception was made for a medical case, where a family petitioned for bottled water service and the Army concurred.
- The former Army Public Health Center (PHC) analyzed Installation drinking water wells; if PFAS was detected, the wells were to be sampled quarterly.

6. General Concerns Associated with Treatment of Water Supplies

- Treatment of irrigation water has no regulatory or risk driver.
- Based on current conditions, the Pomona Artesian Irrigation Company (PAIC) system has the capacity to support one additional connection.
- Connection to Army's Pomona (supply) Well system will introduce chlorinated water into on-lot septic systems, which is anticipated to alter the natural chemistry.

- Connections to the Roza Irrigation System are no longer permitted.
- The Army’s off-post Wastewater Treatment Plant (WWTP) does not have capacity for additional connections. In addition, trained personnel (i.e., electricians, plumbers, and WWTP operators) are in short supply).
- The yield of the three Pomona public water supply wells is unknown, and these wells were likely not aquifer tested (i.e., stepped draw-down test and/or constant-rate pumping test).
- The closest public water supply outside of Pomona water system is the Selah public water system. Cost per mile of connection is estimated at \$2M.
- Is wellhead treatment an option?
- How will occurrences of breakthrough be handled efficiently?

7. Regulatory Updates

- USAEC is aware of Yakima County/City of Selah exploring the application of the Federal water infrastructure grant for connections to public water supply.
- HQDA officials met with Ecology regarding the implementation of a Cooperative Agreement in response to Ecology’s enforcement order.

8. Public Outreach

- Hosting community events is an effective way to demonstrate that the Army is taking steps to inform the public and be available to the community. The group acknowledged that the YTC community has not been addressed since October 2022 at the start of the RI; however, public events need to be intentional and organized to provide a clear message and path forward. The local press is notified of these events and are expected to attend.
- According to the PAO, there are approximately 100 new followers of the Army’s PFAS Information website.
- The Garrison aims to ‘connect the dots’ for the public – to correlate and decipher the components of the RI completed to date. There is value in presenting the work that has been accomplished, which is most conducive to poster-style presentations and/or Open Houses.
- A monthly news release was suggested; however, there have been perceived legal implications for content that is distributed to the public. The USAEC can assist with the approval of informative content.
- Off-post outreach does not need to include everyone; however, selective meetings (i.e., by appointment and/or those included in the pilot test) will likely not be well received by the public. In general, Open House events are an optimal approach. The Army had little success with previous panel-style meetings.
- To date, the Army has made two attempts to establish a Restoration Advisory Board (RAB); however, there has not been interest from the public.
- The reserved date of 07 June 2023 for a community event is considered premature at this time, because there is no new substantive information to distribute. USAEC believes efforts are better spent on updating ROE records. A date will be selected after validated groundwater analytical results are available.
- A second Technical Project Planning (TPP) meeting is currently scheduled for 06 June 2023; however, there may be value in postponing the TPP to coincide with a community event.

9. Installation of the Initial Five POET Systems

- Five locations were selected for the initial pilot study to demonstrate progress by the Army, based primarily on existing infrastructure and PFOA/PFOS concentrations. The intent is to evaluate the installation and operation of these systems and project on a larger scale and identify any potential points of failure to mitigate before additional POET installs.
- The proposed pilot study of five POET installs may not be well received by the public.
- Limited resources (i.e., supplies and manpower – electricians, plumbers) in the Yakima, WA area may necessitate the sequential installation of POETs, rather than simultaneous.

10. General Concerns Regarding the PFAS RI

- Homeowners continue to install wells intended for potable use in the impact area, and there is no mechanism in place currently to implement land use controls. The RI process must proceed in accordance with CERCLA.
- The general message circulating amongst impacted homeowners is to have their existing well deepened, which may compromise the deep, artesian water-bearing zone.

11. EDMS

- USAEC and the YTC Garrison will partner with Synectics, Inc. to develop the Electronic Document Management System (EDMS) for YTC and implement for all sampling events. The target date for release is June 2023.
- The EDMS is designed for internal use only and will not be accessible to the public.
- Section 345 reporting is mandated for every off-post sample.

12. Contract Structure

- Execution of RI Optional Tasks 1001 and 1002 is imminent.
- The POET installation, and the operation and maintenance tasks may be handled better if they were under a single contract. USAEC will explore reorganization of the Contracts offline with the KO.

13. Next Steps and Action Items

- Update community ROE records.
- Draft a brochure to detail the installation of POETs and what this means long-term for homeowners.
- Suggest that appropriate controls be established by Ecology to prevent the spread of PFAS contamination (i.e., prevent deepening of existing wells and the installation of any additional wells in the impacted area).
- Initiate the pilot test of five POET system installations and schedule an appointment with selected homeowners to discuss a path forward.
- Establish EDMS for YTC and implement for all sampling events.
- Arrange for Government presence during all off-post sampling events, for safety purposes, to field questions, and allow the sampling team to focus and work efficiently.
- Initiate Quarterly Monitoring Events, both on and off-post.
- Beginning in May 2023, USGS will initiate off-post sampling for general groundwater chemistry parameters, and USEPA will initiate off-post sampling for PFAS.
- USAEC to distribute sampling guidance.
- ECC to develop a timeline for the pilot test.

- ECC/Arcadis to finish a draft of the Uniform Federal Policy (UFP) Quality Assurance Project Plan (QAPP).

14. Schedule

- TPP #2 - 06 June 2023 (subject to change).
- Analytical review and validation of Boundary Well groundwater samples - 09 May to 21 July 2023.
- UFP QAPP - Period of Performance (POP) 27 September 2023.

Appendix C
UFP-QAPP Acknowledgement Form

UFP-QAPP Acknowledgement Form (Placeholder)

This Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP) was prepared in accordance with the Intergovernmental Data Quality Task Force - Optimized UFP-QAPP Worksheets guidance document, dated March 2012.

Name: _____

License Number: _____

Expiration Date: _____

Name

Date

Appendix D
Boundary Well Construction Logs

WELL ID: MW-01

CLIENT: Army Environmental Command
LOCATION: Yakima Training Center, WA

Project #: 30124009
Reviewed by: Jesse Hemmen, PG#2958

Logging Probes Used

- Natural Gamma
- Fluid Temperature/Resistivity
- Induction Conductivity
- Normal Resistivity
- 3-Arm Caliper
- Acoustic Televiwer
- Optical Televiwer
- Heat Pulse Flow Meter
- Spinner Flow Meter
- Spectral Gamma
- Full Waveform Sonic
- Nuclear Magnetic Resonance
- CDFM Flow Meter
- SPR/SP
- Other:

DATE DRILLING STARTED: 4/12/2023

DATE DRILLING FINISHED: 4/12/2023

DATE WELL COMPLETE: 4/13/2023

DRILLING COMPANY: Gregory Drilling

DRILLING RIG: Foremost DR-12

DRILLER'S NAME: Chris Gregory

DRILLER'S ASSISTANT(S): M. Grauberer, J. Davis

LOGGED BY: Larissa Sleeper

NORTHING: 492518.30 **EASTING:** 1651519.53

TOC ELEVATION: 1322.95 ft

GROUND SURFACE ELEVATION: 1320.60 ft

TOTAL BOREHOLE DEPTH: 142 feet bgs

BOREHOLE DIAMETER: 6.0 inches

DRILLING METHOD: Air Rotary

SAMPLING INTERVAL: Continuous

SAMPLING DEVICE: Cyclone

DRILLING FLUID USED: Water as Needed

WELL CONSTRUCTION

WELL CASING: Schedule 40 PVC

WELL DIAMETER: 2.0 inches

WELL SCREEN: Schedule 40 PVC

SCREEN DIAMETER: 2.0 inches

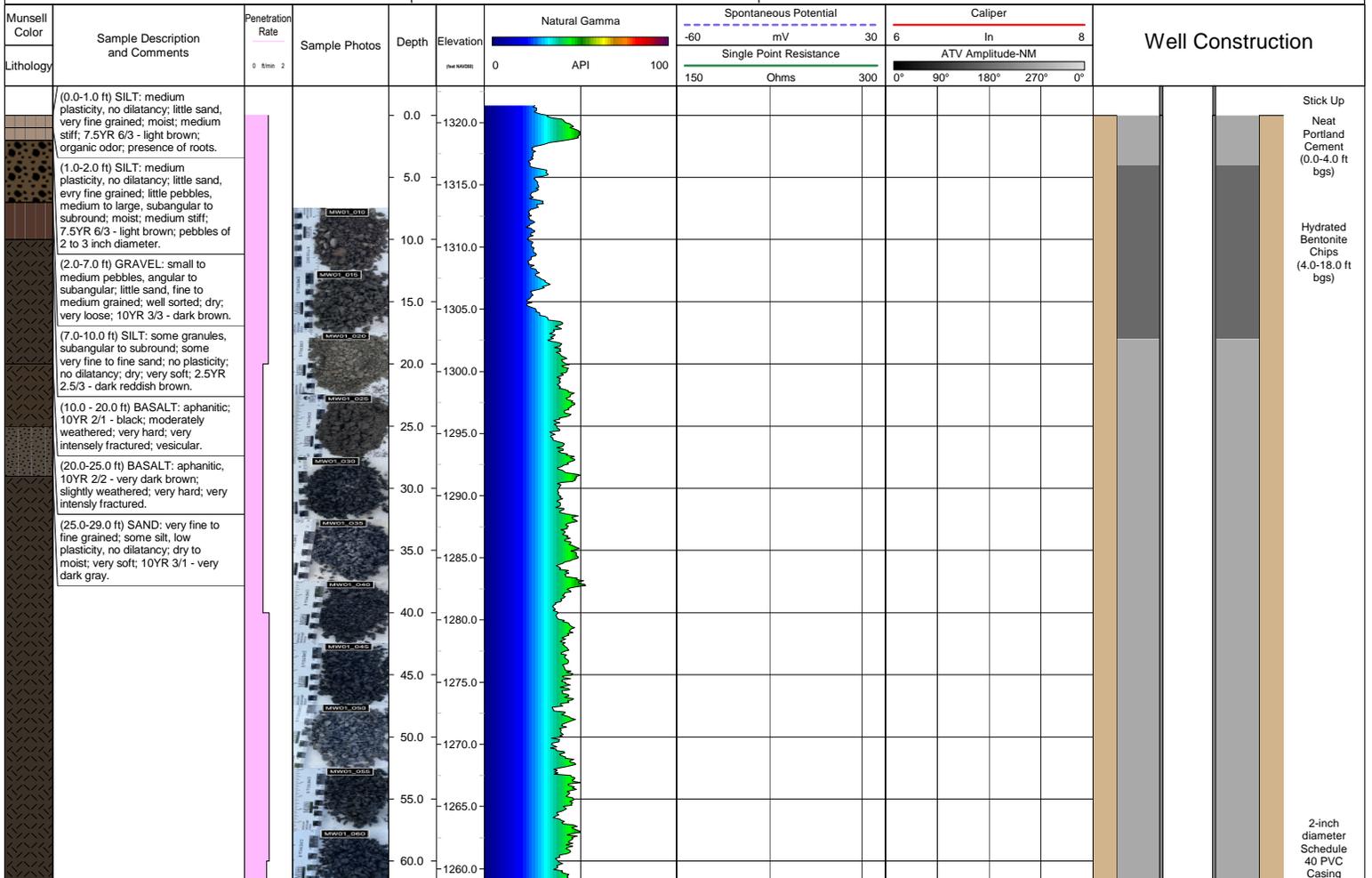
SLOT SIZE: 0.010 inches

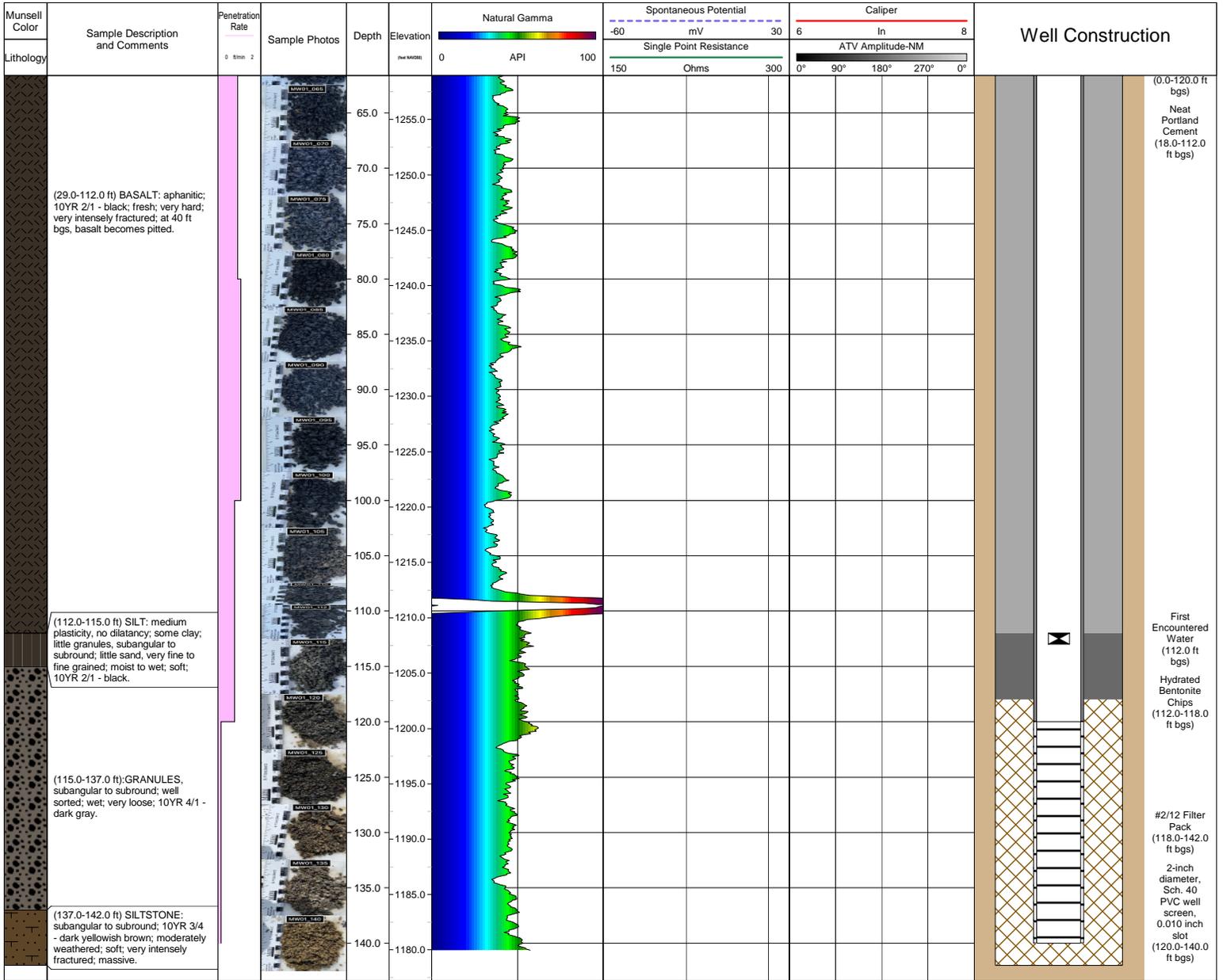
SAND PACK: #12 Sand Pack

ANNULUS SEAL: Hydrated Bentonite Chips

GROUT: Neat Portland Cement

COMPLETION TYPE: Stick Up





ABBREVIATIONS: bgs = beneath ground surface, NA = not available, ft = feet, PVC = polyvinyl chloride, USCS = Unified Soil Classification System, in = inches, mm = millimeters, < = less than, % = percent, NAVD88 = North American Vertical Datum of 1988, NAD83 = North American Datum of 1983

NOTES: Horizontal coordinate projection: SPCS, Washington South, NAD83 Datum, U.S. Survey Feet. Elevation is reported in the NAVD88 (Geoid 18) system. Hand augered to 5.0 ft bgs or to refusal.

WELL ID: MW-02

CLIENT: Army Environmental Command
LOCATION: Yakima Training Center, WA

Project #: 30124009
Reviewed by: Jesse Hemmen, PG#2958

Logging Probes Used

- Natural Gamma
- Fluid Temperature/Resistivity
- Induction Conductivity
- Normal Resistivity
- 3-Arm Caliper
- Acoustic Televierer
- Optical Televierer
- Heat Pulse Flow Meter
- Spinner Flow Meter
- Spectral Gamma
- Full Waveform Sonic
- Nuclear Magnetic Resonance
- CDFM Flow Meter
- SPR/SP
- Other:

DATE DRILLING STARTED: 4/3/2023

DATE DRILLING FINISHED: 4/3/2023

DATE WELL COMPLETE: 4/4/2023

DRILLING COMPANY: Gregory Drilling

DRILLING RIG: Foremost DR-12

DRILLER'S NAME: Chris Gregory

DRILLER'S ASSISTANT(S): M. Grauberer, J. Davis

LOGGED BY: Roberto Piemontese

NORTHING: 490133.45 **EASTING:** 1651470.97

TOC ELEVATION: 1292.26 ft

GROUND SURFACE ELEVATION: 1289.70 ft

TOTAL BOREHOLE DEPTH: 210.0 feet bgs

BOREHOLE DIAMETER: 6.0 inches

DRILLING METHOD: Air Rotary

SAMPLING INTERVAL: Continuous

SAMPLING DEVICE: Cyclone

DRILLING FLUID USED: Water as Needed

WELL CONSTRUCTION

WELL CASING: Schedule 40 PVC

WELL DIAMETER: 2.0 inches

WELL SCREEN: Schedule 40 PVC

SCREEN DIAMETER: 2.0 inches

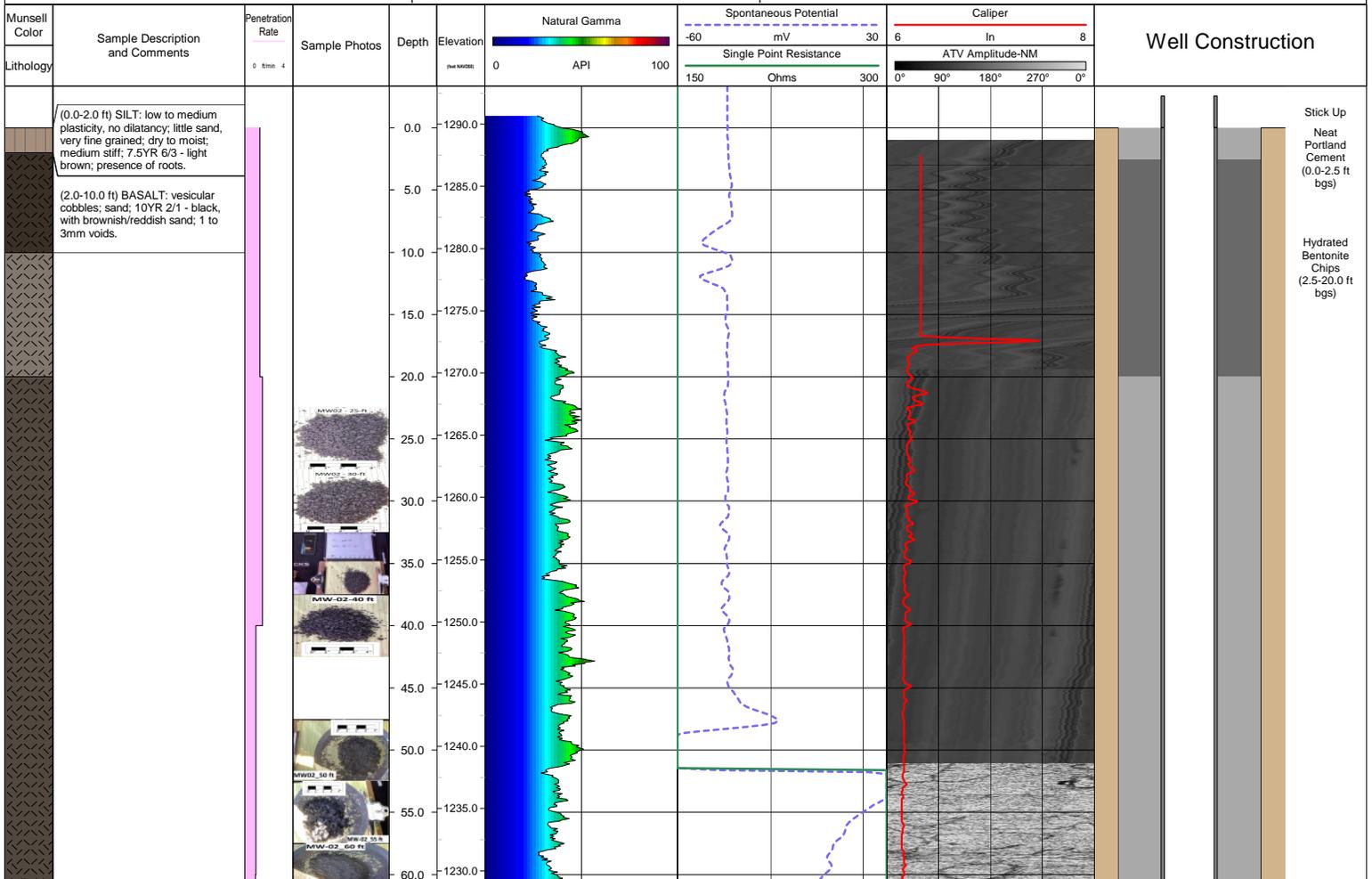
SLOT SIZE: 0.010 inches

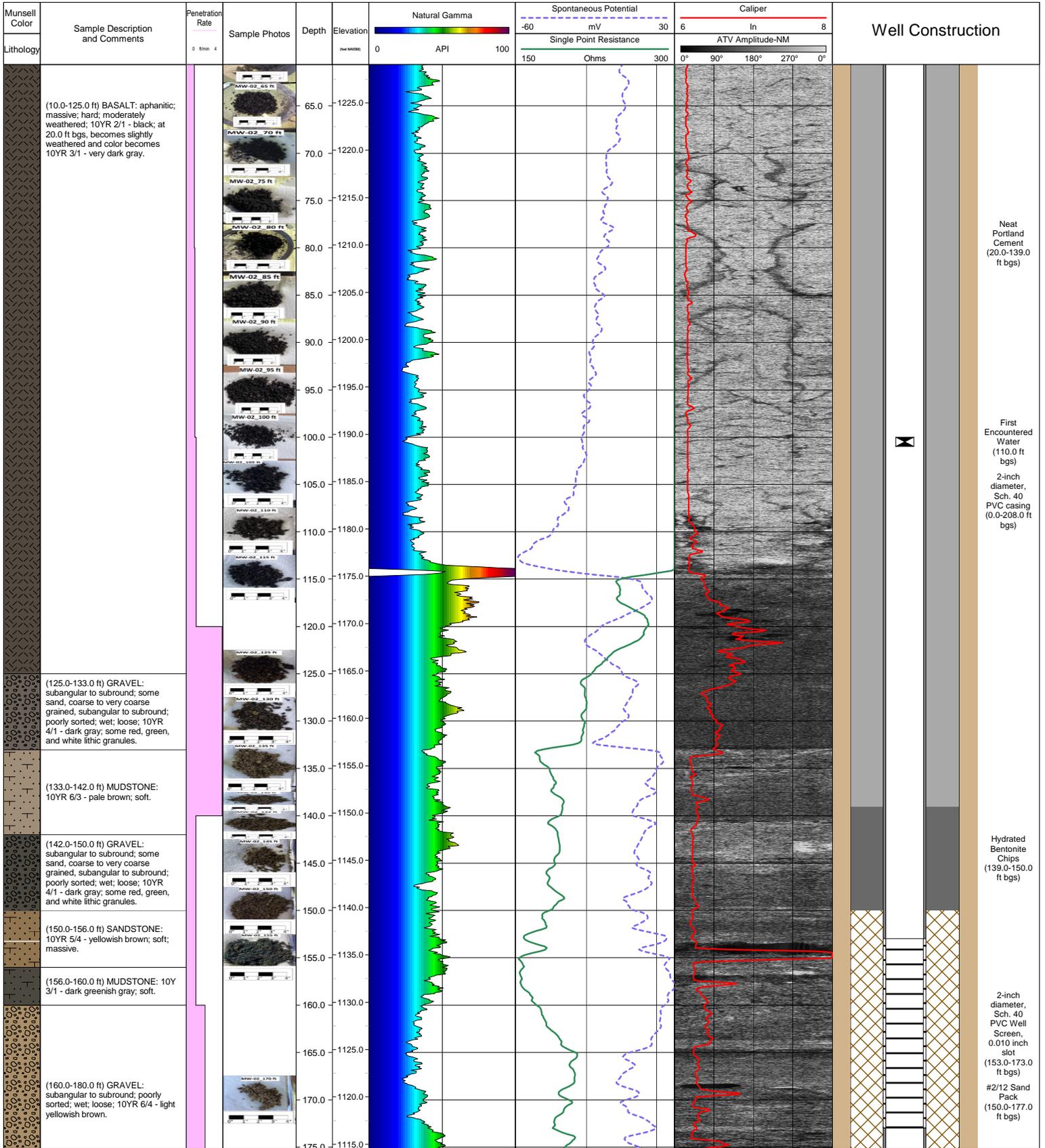
SAND PACK: #2/12 Sand Pack

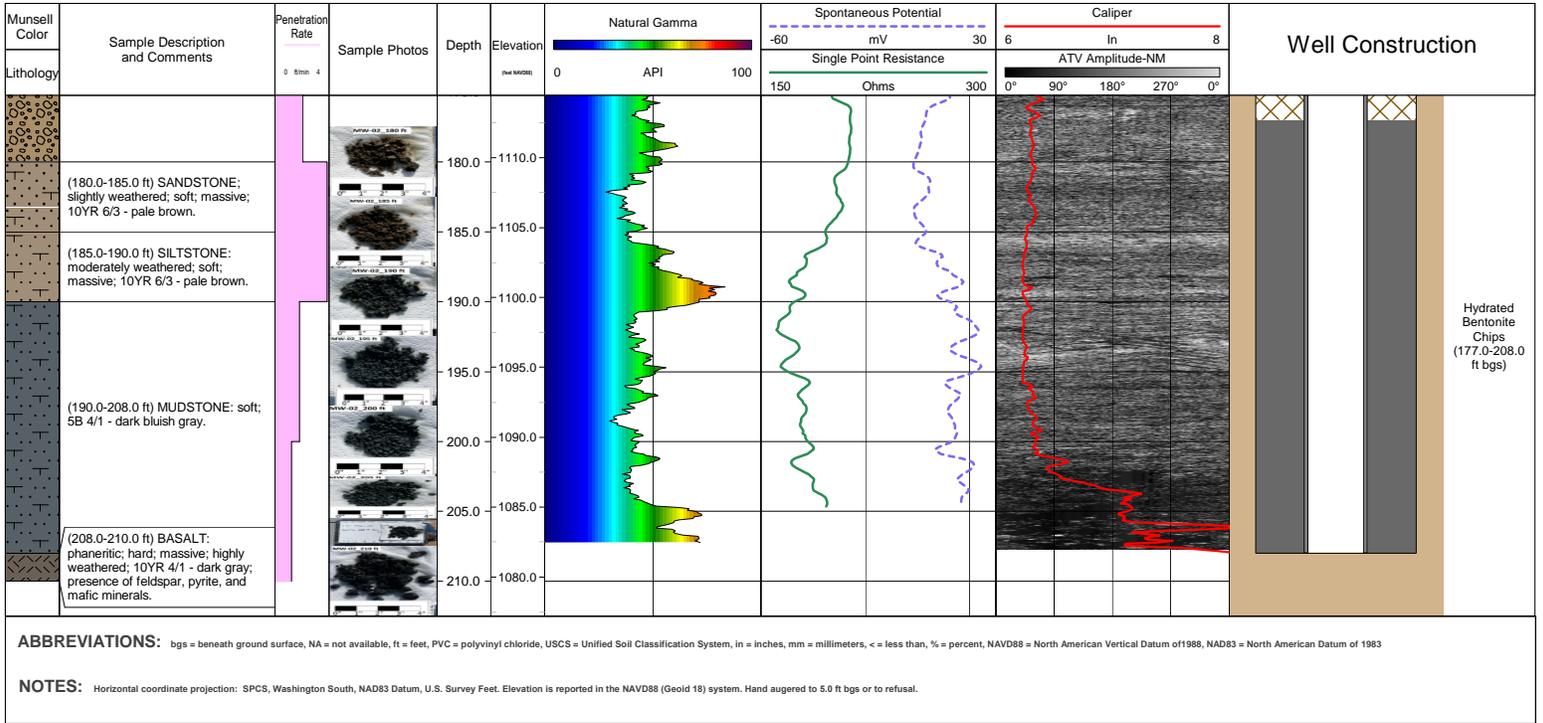
ANNULUS SEAL: Hydrated Bentonite Chips

GROUT: Neat Portland Cement

COMPLETION TYPE: Stick Up







WELL ID: MW-03

CLIENT: Army Environmental Command
LOCATION: Yakima Training Center, WA

Project #: 30124009
Reviewed by: Jesse Hemmen, PG#2958

Logging Probes Used

- Natural Gamma
- Fluid Temperature/Resistivity
- Induction Conductivity
- Normal Resistivity
- 3-Arm Caliper
- Acoustic Televiwer
- Optical Televiwer
- Heat Pulse Flow Meter
- Spinner Flow Meter
- Spectral Gamma
- Full Waveform Sonic
- Nuclear Magnetic Resonance
- CDFM Flow Meter
- SPR/SP
- Other:

DATE DRILLING STARTED: 4/5/2023

NORTHING: 487990.45 **EASTING:** 1648992.98

WELL CONSTRUCTION

DATE DRILLING FINISHED: 4/5/2023

TOC ELEVATION: 1307.66 ft

WELL CASING: Schedule 40 PVC

DATE WELL COMPLETE: 4/6/2023

GROUND SURFACE ELEVATION: 1305.00 ft

WELL DIAMETER: 2.0 inches

DRILLING COMPANY: Gregory Drilling

TOTAL BOREHOLE DEPTH: 210.0 feet bgs

WELL SCREEN: Schedule 40 PVC

DRILLING RIG: Foremost DR-12

BOREHOLE DIAMETER: 6.0 inches

SCREEN DIAMETER: 2.0 inches

DRILLER'S NAME: Chris Gregory

DRILLING METHOD: Air Rotary

SLOT SIZE: 0.010 inches

DRILLER'S ASSISTANT(S): M. Grauberer, J. Davis

SAMPLING INTERVAL: Continuous

SAND PACK: #2/12 Sand Pack

LOGGED BY: Roberto Piemontese

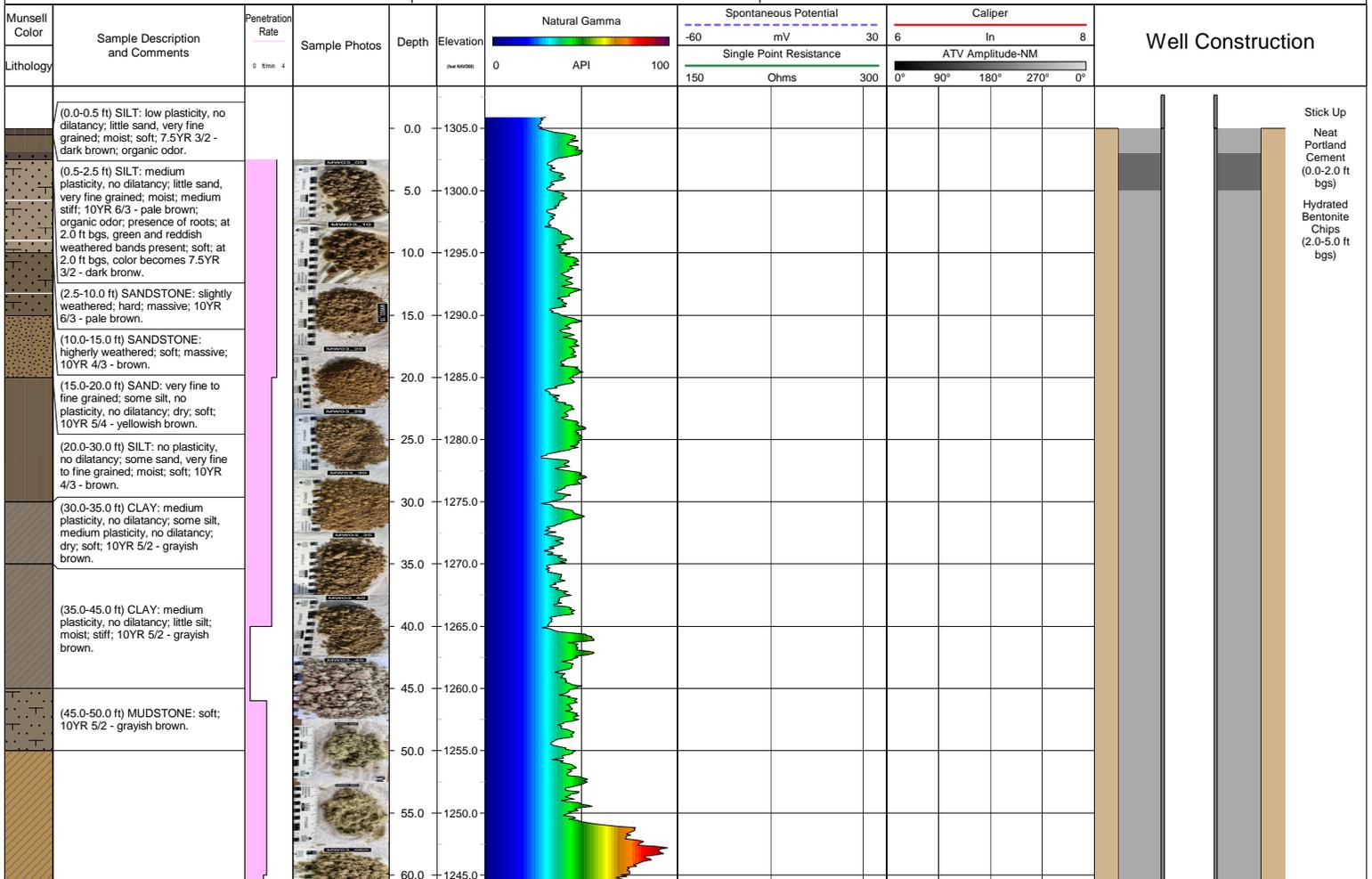
SAMPLING DEVICE: Cyclone

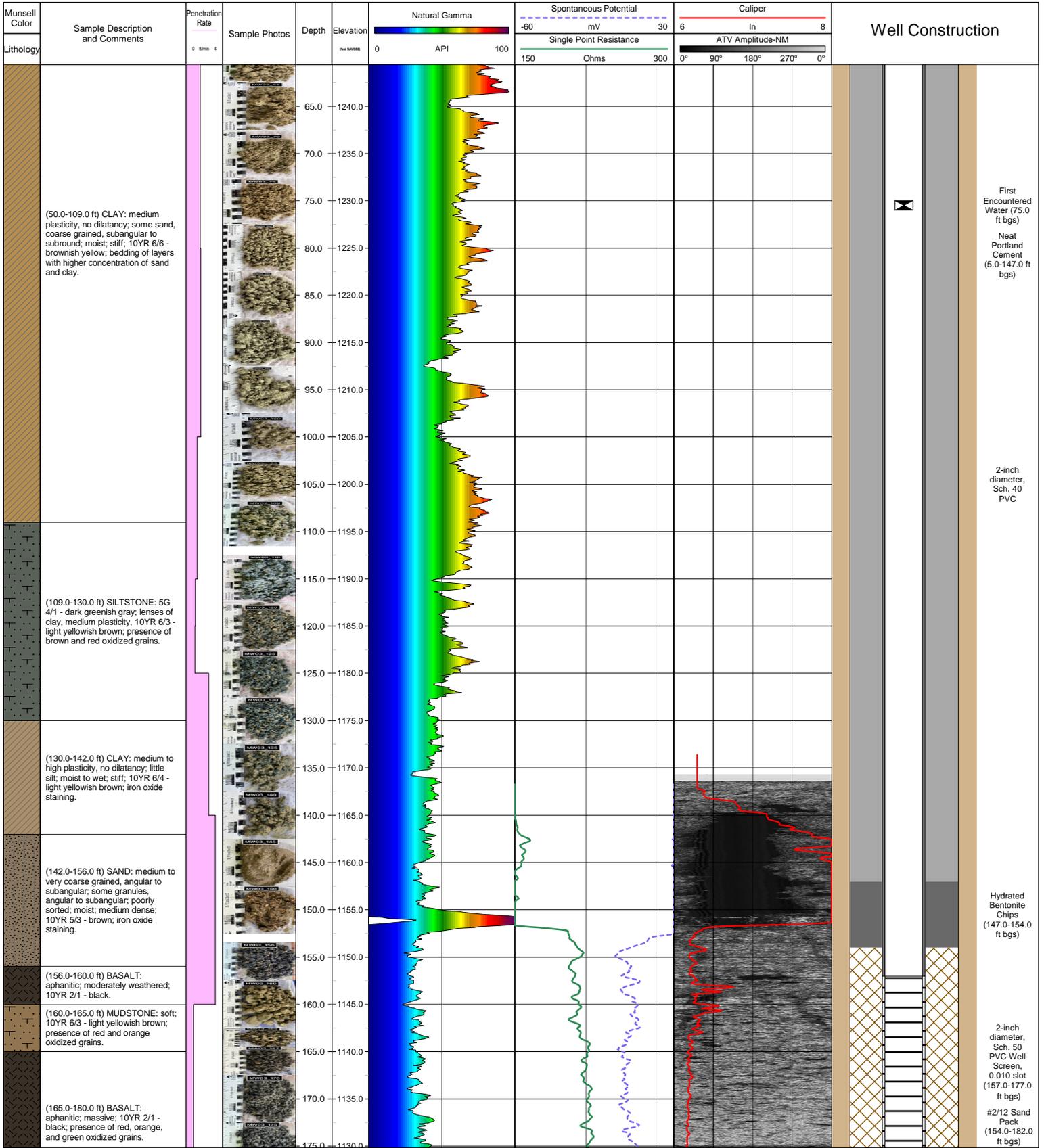
ANNULUS SEAL: Hydrated Bentonite Chips

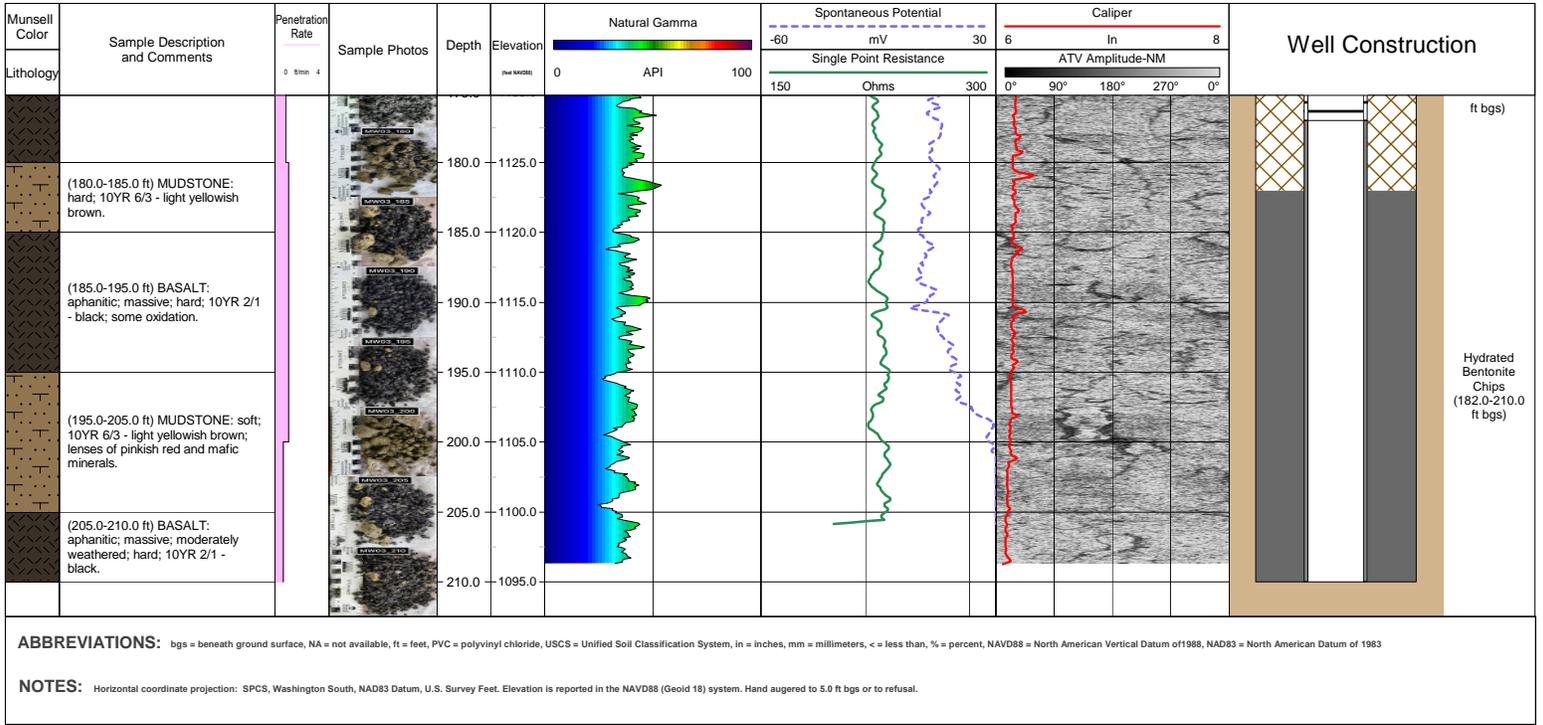
GROUT: Neat Portland Cement

COMPLETION TYPE: Stick Up

DRILLING FLUID USED: Water as Needed







WELL ID: MW-04

CLIENT: Army Environmental Command
LOCATION: Yakima Training Center, WA

Project #: 30124009
Reviewed by: Jesse Hemmen, PG#2958

Logging Probes Used

- Natural Gamma
- Fluid Temperature/Resistivity
- Induction Conductivity
- Normal Resistivity
- 3-Arm Caliper
- Acoustic Televiwer
- Optical Televiwer
- Heat Pulse Flow Meter
- Spinner Flow Meter
- Spectral Gamma
- Full Waveform Sonic
- Nuclear Magnetic Resonance
- CDFM Flow Meter
- SPR/SP
- Other:

DATE DRILLING STARTED: 4/17/2023

NORTHING: 487542.02 **EASTING:** 1649009.88

WELL CONSTRUCTION

DATE DRILLING FINISHED: 4/17/2023

TOC ELEVATION: 1315.69 ft

WELL CASING: Schedule 40 PVC

DATE WELL COMPLETE: 4/18/2023

GROUND SURFACE ELEVATION: 1314.30 ft

WELL DIAMETER: 2.0 inches

DRILLING COMPANY: Gregory Drilling

TOTAL BOREHOLE DEPTH: 180 feet bgs

WELL SCREEN: Schedule 40 PVC

DRILLING RIG: Foremost DR-12

BOREHOLE DIAMETER: 6.0 inches

SCREEN DIAMETER: 2.0 inches

DRILLER'S NAME: Chris Gregory

DRILLING METHOD: Air Rotary

SLOT SIZE: 0.010 inches

DRILLER'S ASSISTANT(S): M. Grauberer, J. Davis

SAMPLING INTERVAL: Continuous

SAND PACK: #2/12 Sand Pack

LOGGED BY: Larissa Sleeper

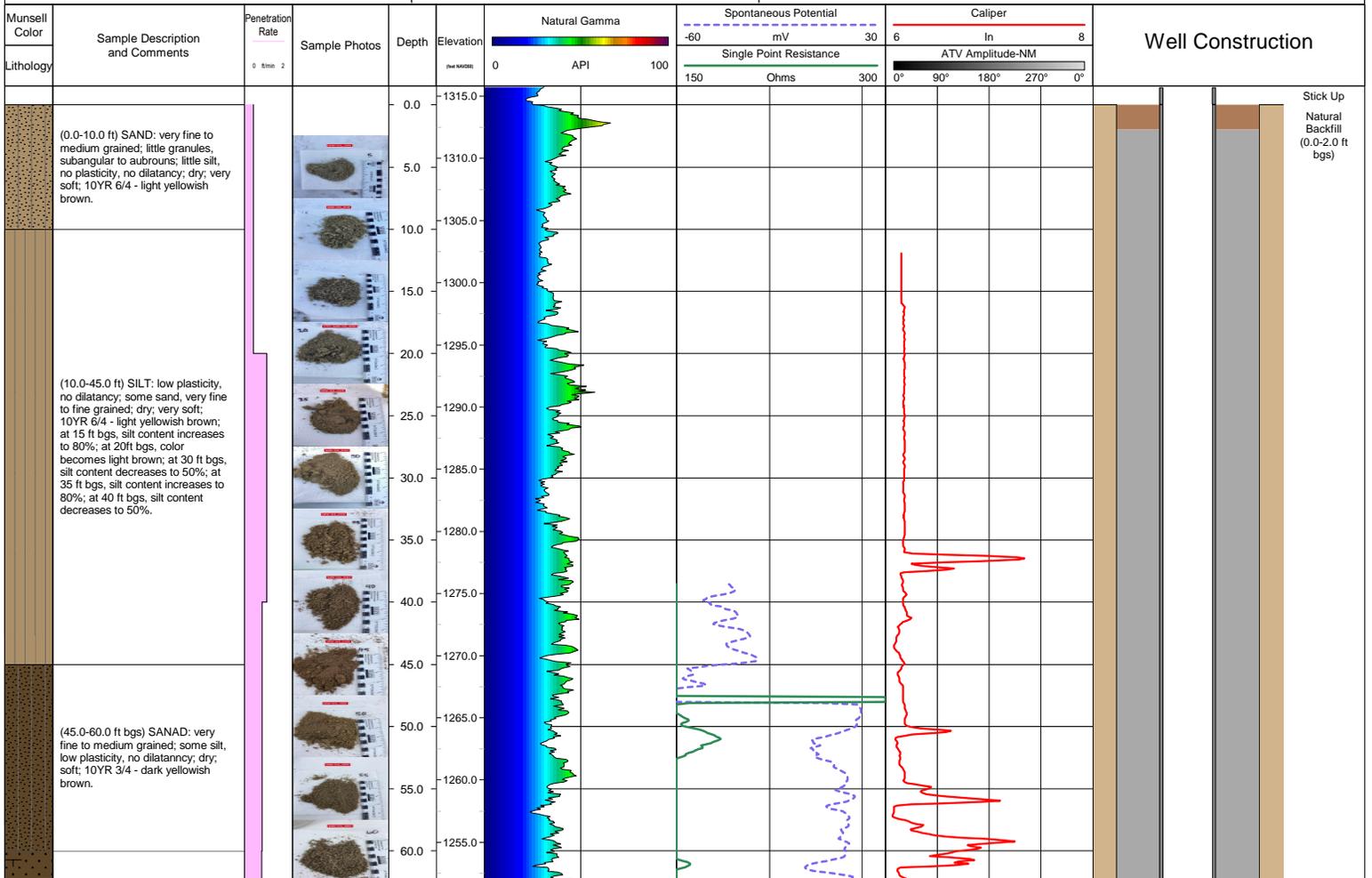
SAMPLING DEVICE: Cyclone

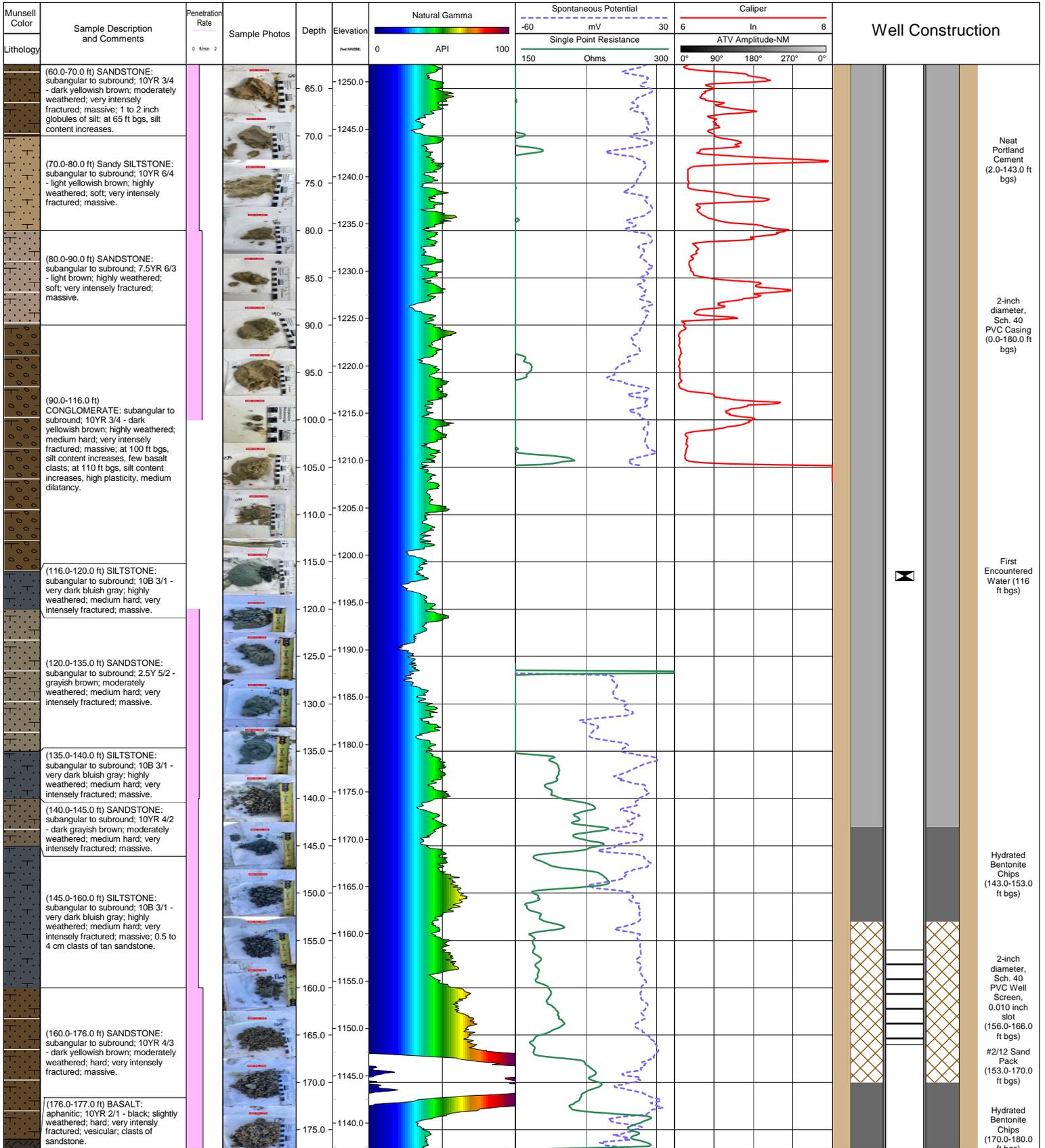
ANNULUS SEAL: Hydrated Bentonite Chips

GROUT: Neat Portland Cement

COMPLETION TYPE: Stick Up

DRILLING FLUID USED: Water as Needed





| Munsell Color | Sample Description and Comments | Penetration Rate | Sample Photos | Depth | Elevation <small>(ft MVD)</small> | Natural Gamma | Spontaneous Potential | Caliper | Well Construction |
|---------------|---|------------------|---|-------|--------------------------------------|---|-----------------------|---------------------|---|
| Lithology | | | | | | API | mV | In | |
| | (177.0-180.0 ft) SANDSTONE: subangular; 10YR 3/4 - dark yellowish brown; moderately weathered; hard; very intensely fractured; massive. | 0 0.000 2 |  | 180.0 | 1135.0 |  | -60 30 | 6 8 |  |
| | | | | | 1130.0 | 0 100 | 150 300 | 0° 90° 180° 270° 0° | |

ABBREVIATIONS: bgs = beneath ground surface, NA = not available, ft = feet, PVC = polyvinyl chloride, USCS = Unified Soil Classification System, in = inches, mm = millimeters, < = less than, % = percent, NAVD88 = North American Vertical Datum of 1988, NAD83 = North American Datum of 1983

NOTES: Horizontal coordinate projection: SPCS, Washington South, NAD83 Datum, U.S. Survey Feet. Elevation is reported in the NAVD88 (Geoid 18) system. Hand augered to 5.0 ft bgs or to refusal.

WELL ID: MW-05

CLIENT: Army Environmental Command
LOCATION: Yakima Training Center, WA

Project #: 30124009
Reviewed by: Jesse Hemmen, PG#2958

Logging Probes Used

- Natural Gamma
- Fluid Temperature/Resistivity
- Induction Conductivity
- Normal Resistivity
- 3-Arm Caliper
- Acoustic Televiwer
- Optical Televiwer
- Heat Pulse Flow Meter
- Spinner Flow Meter
- Spectral Gamma
- Full Waveform Sonic
- Nuclear Magnetic Resonance
- CDFM Flow Meter
- SPR/SP
- Other:

DATE DRILLING STARTED: 4/12/2023

NORTHING: 486631.14 **EASTING:** 1649002.06

WELL CONSTRUCTION

DATE DRILLING FINISHED: 4/12/2023

TOC ELEVATION: 1354.49 ft

WELL CASING: Schedule 40 PVC

DATE WELL COMPLETE: 4/13/2023

GROUND SURFACE ELEVATION: 1351.80 ft

WELL DIAMETER: 2.0 inches

DRILLING COMPANY: Gregory Drilling

TOTAL BOREHOLE DEPTH: 210 feet bgs

WELL SCREEN: Schedule 40 PVC

DRILLING RIG: Foremost DR-12

BOREHOLE DIAMETER: 6.0 inches

SCREEN DIAMETER: 2.0 inches

DRILLER'S NAME: Chris Gregory

DRILLING METHOD: Air Rotary

SLOT SIZE: 0.010 inches

DRILLER'S ASSISTANT(S): M. Grauberer, J. Davis

SAMPLING INTERVAL: Continuous

SAND PACK: #2/12 Sand Pack

LOGGED BY: Larissa Sleeper

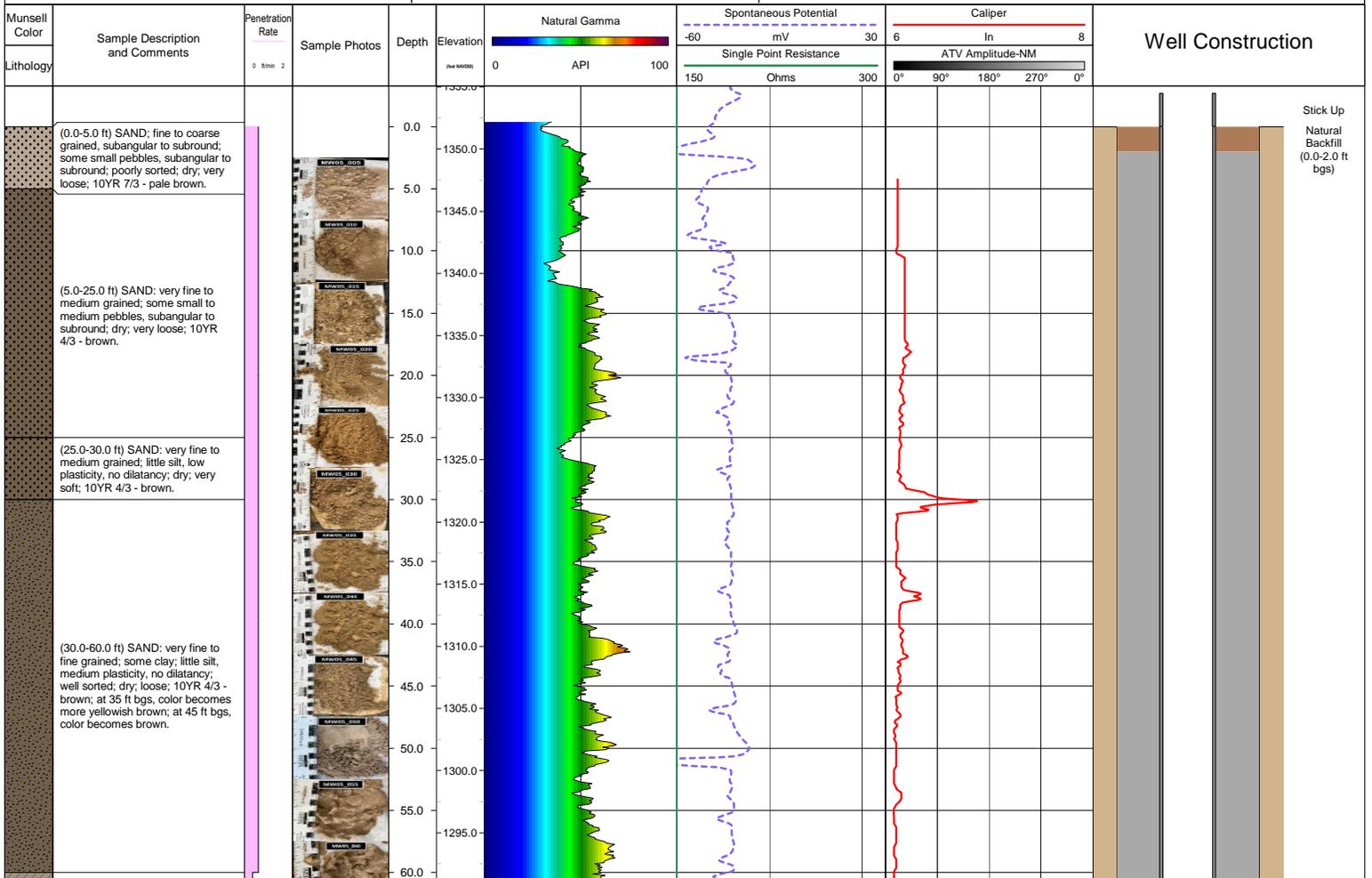
SAMPLING DEVICE: Cyclone

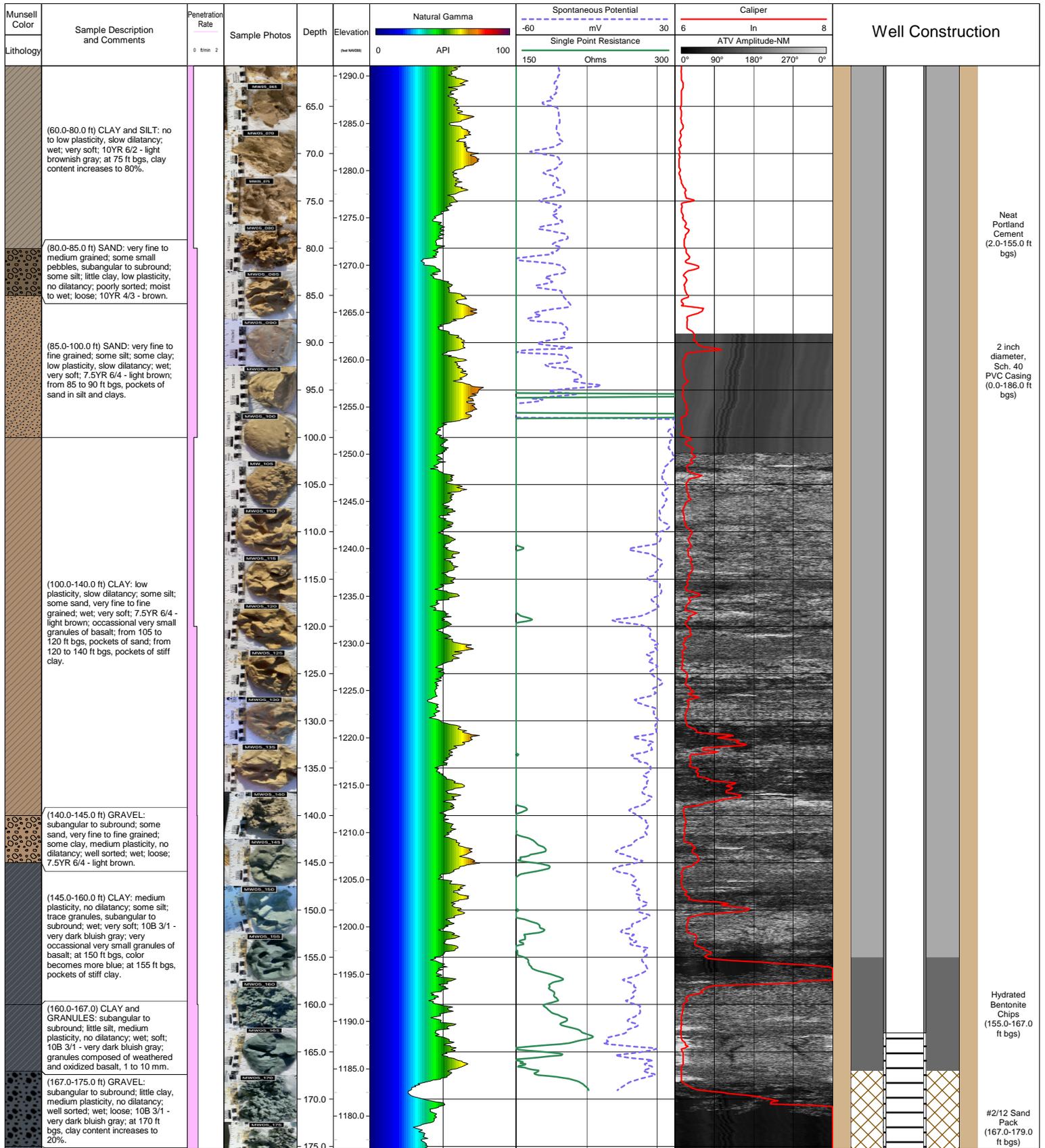
ANNULUS SEAL: Hydrated Bentonite Chips

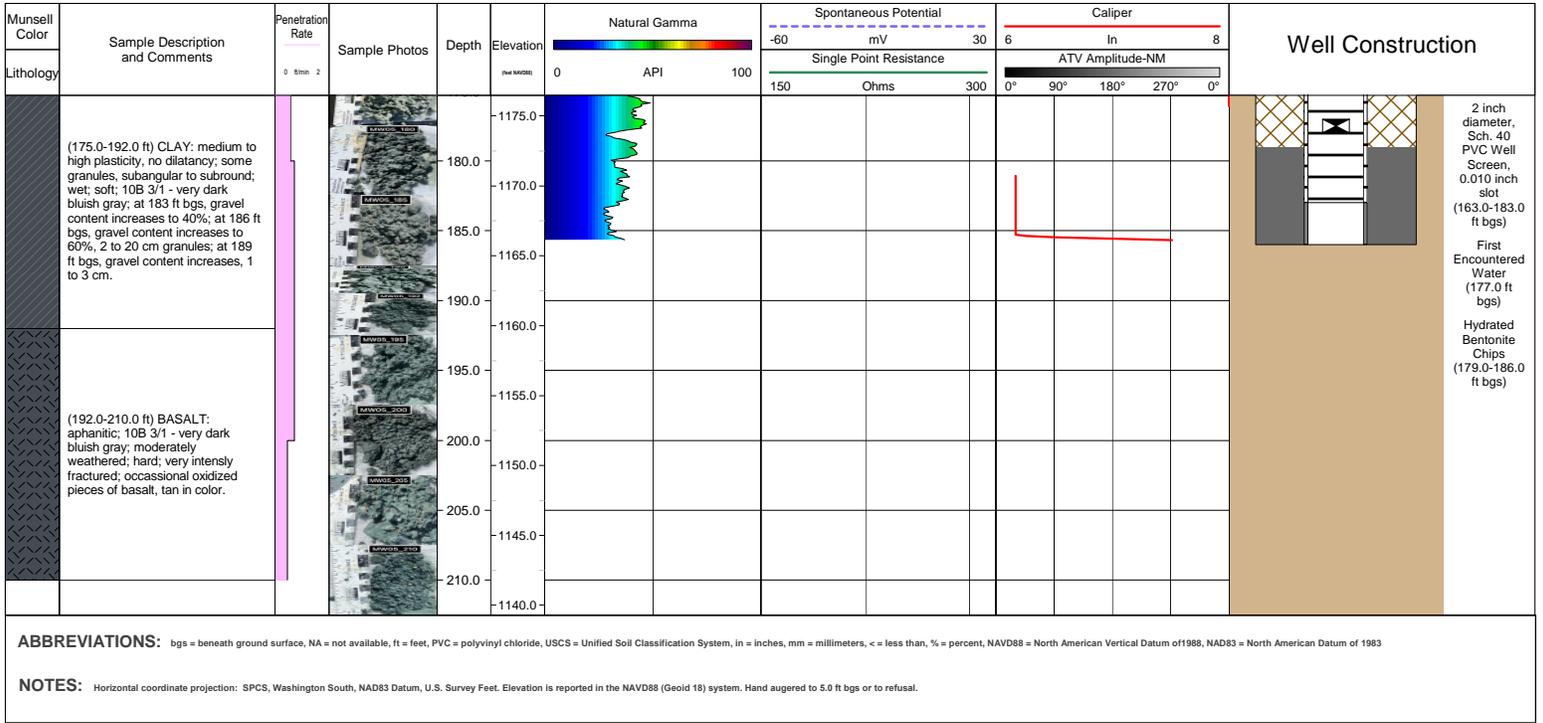
GROUT: Neat Portland Cement

COMPLETION TYPE: Stick Up

DRILLING FLUID USED: Water as Needed







ABBREVIATIONS: bgs = beneath ground surface, NA = not available, ft = feet, PVC = polyvinyl chloride, USCS = Unified Soil Classification System, in = inches, mm = millimeters, < = less than, % = percent, NAVD88 = North American Vertical Datum of 1988, NAD83 = North American Datum of 1983

NOTES: Horizontal coordinate projection: SPCS, Washington South, NAD83 Datum, U.S. Survey Feet. Elevation is reported in the NAVD88 (Geoid 18) system. Hand augered to 5.0 ft bgs or to refusal.

WELL ID: MW-06

CLIENT: Army Environmental Command
LOCATION: Yakima Training Center, WA

Project #: 30124009
Reviewed by: Jesse Hemmen, PG#2958

Logging Probes Used

- Natural Gamma
- Fluid Temperature/Resistivity
- Induction Conductivity
- Normal Resistivity
- 3-Arm Caliper
- Acoustic Televiwer
- Optical Televiwer
- Heat Pulse Flow Meter
- Spinner Flow Meter
- Spectral Gamma
- Full Waveform Sonic
- Nuclear Magnetic Resonance
- CDFM Flow Meter
- SPR/SP
- Other:

DATE DRILLING STARTED: 4/23/2023

NORTHING: 486049.10 **EASTING:** 1648988.98

WELL CONSTRUCTION

DATE DRILLING FINISHED: 4/23/2023

TOC ELEVATION: 1315.81 ft

WELL CASING: Schedule 40 PVC

DATE WELL COMPLETE: 4/24/2023

GROUND SURFACE ELEVATION: 1313.40 ft

WELL DIAMETER: 2.0 inches

DRILLING COMPANY: Gregory Drilling

TOTAL BOREHOLE DEPTH: 236 feet bgs

WELL SCREEN: Schedule 40 PVC

DRILLING RIG: Foremost DR-12

BOREHOLE DIAMETER: 6.0 inches

SCREEN DIAMETER: 2.0 inches

DRILLER'S NAME: Chris Gregory

DRILLING METHOD: Air Rotary

SLOT SIZE: 0.010 inches

DRILLER'S ASSISTANT(S): M. Grauberer, J. Davis

SAMPLING INTERVAL: Continuous

SAND PACK: #2/12 Sand Pack

LOGGED BY: Roberto Piemontese

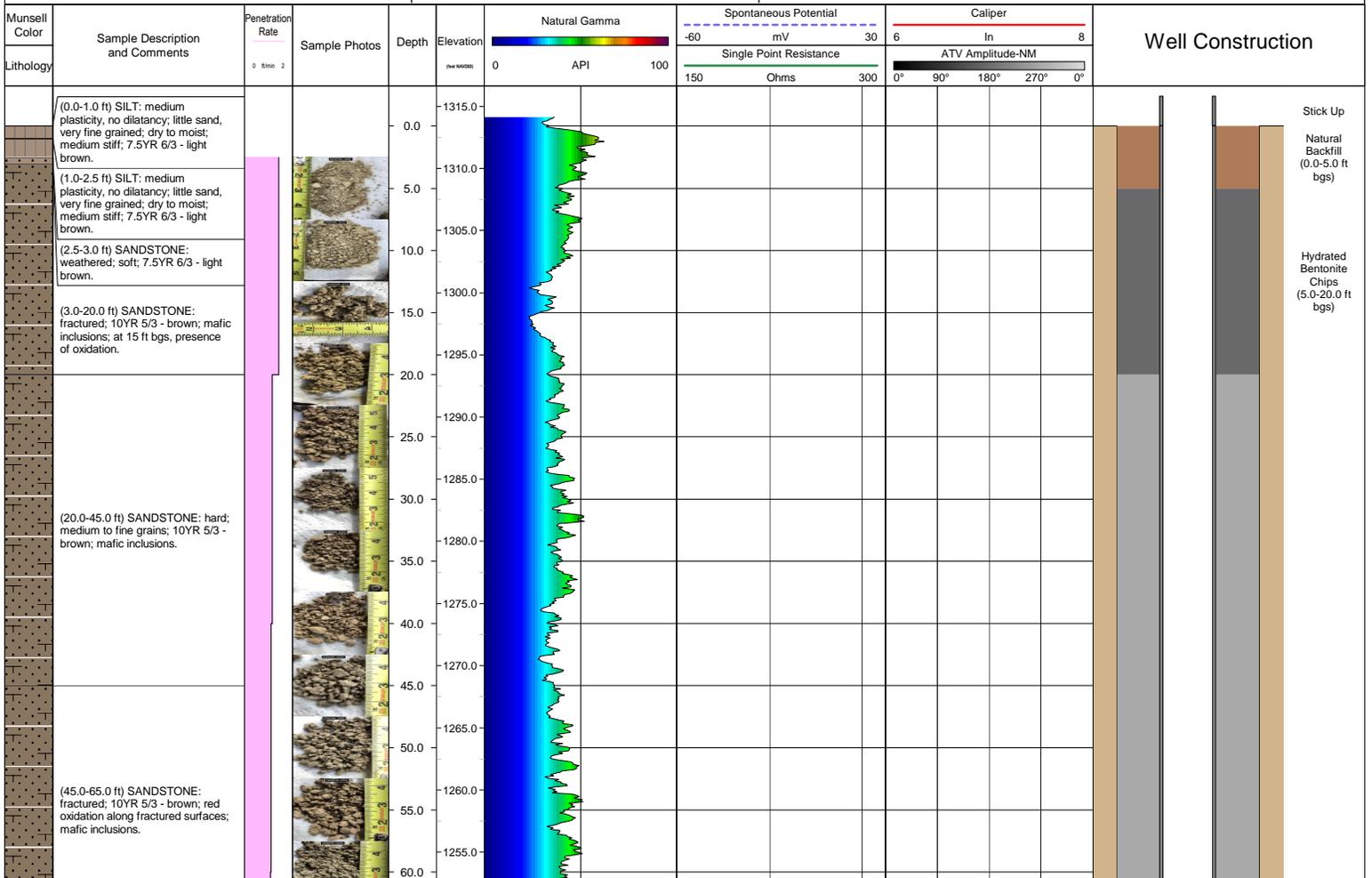
SAMPLING DEVICE: Cyclone

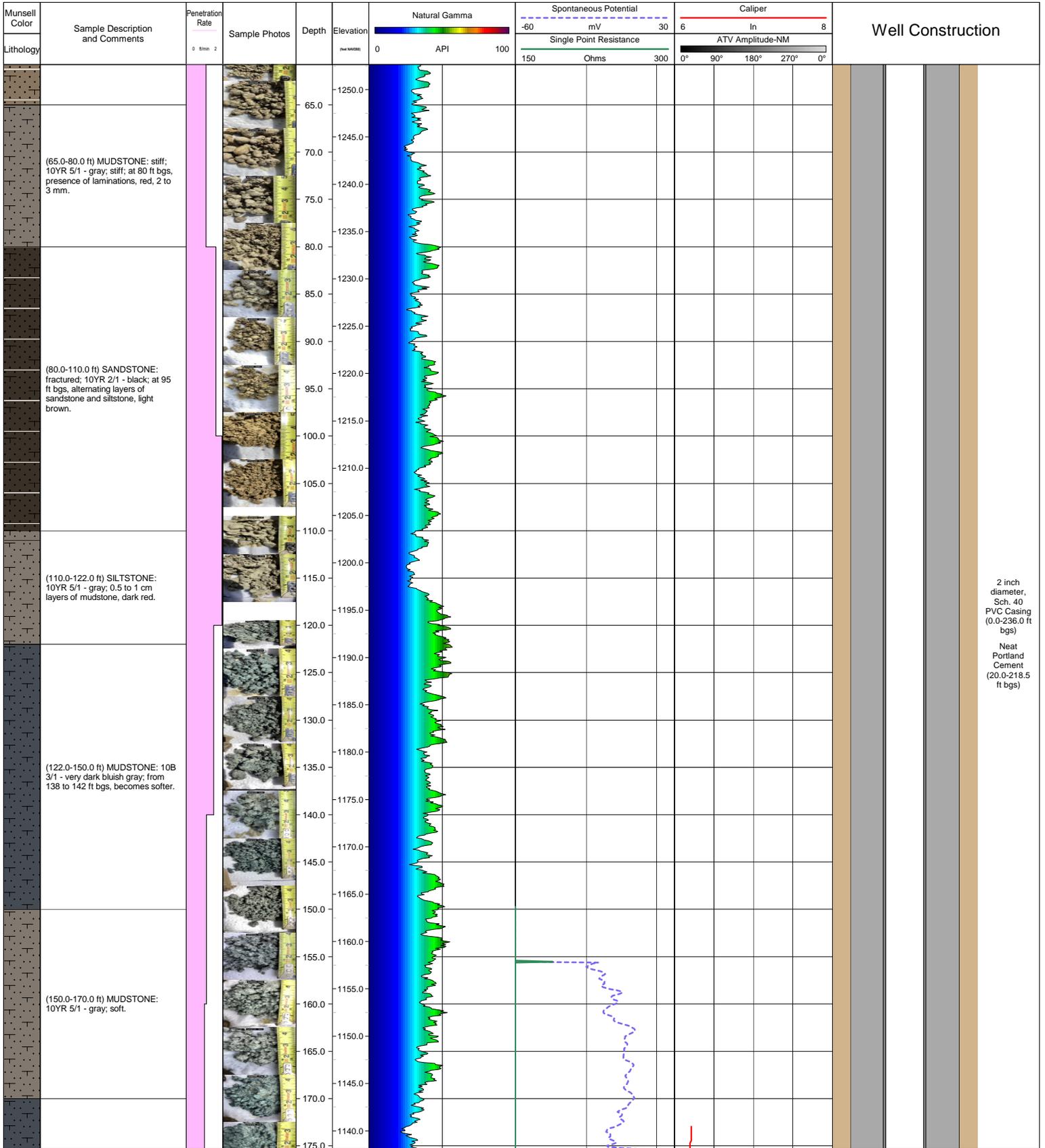
ANNULUS SEAL: Hydrated Bentonite Chips

GROUT: Neat Portland Cement

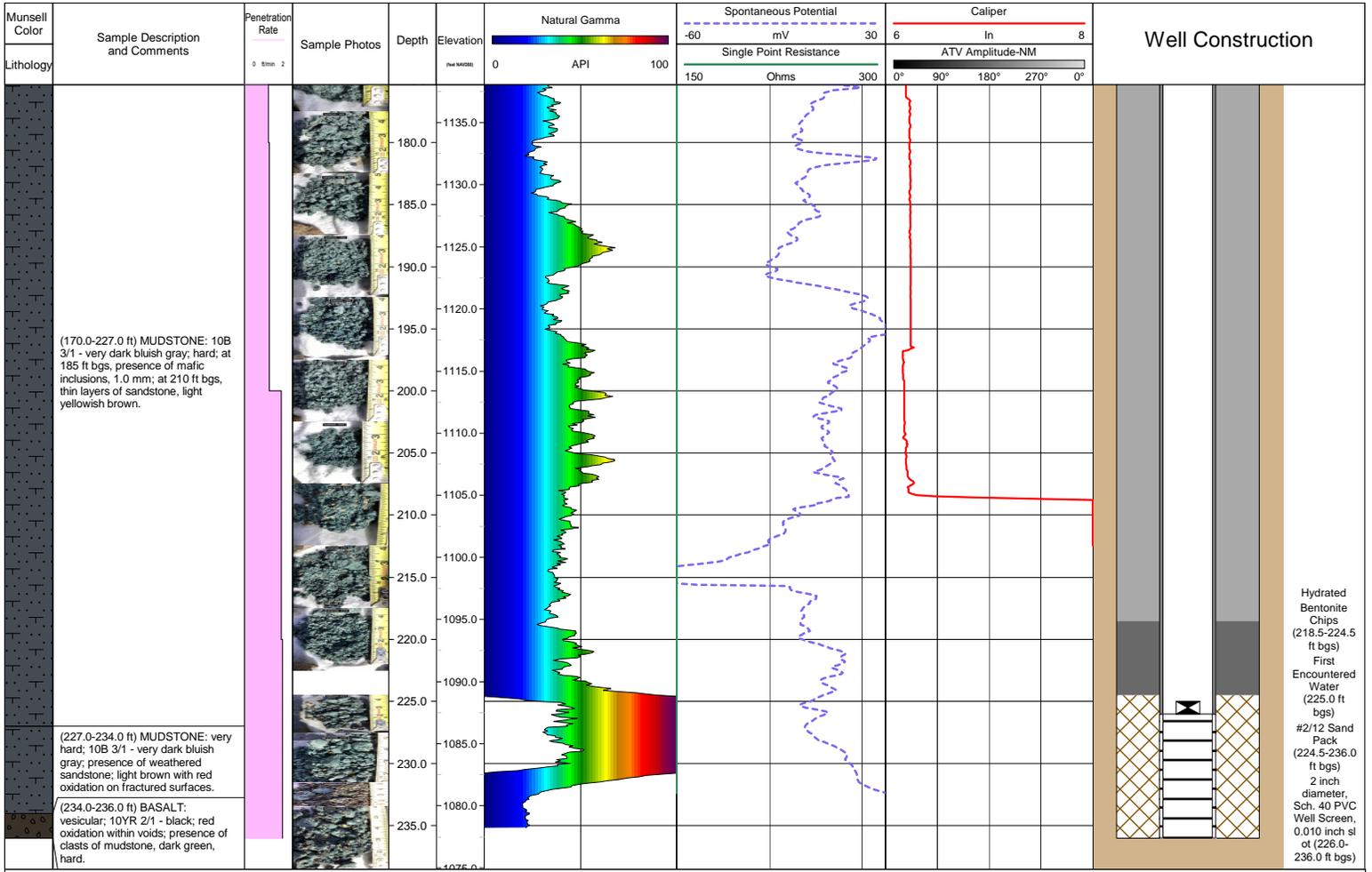
COMPLETION TYPE: Stick Up

DRILLING FLUID USED: Water as Needed





2 inch diameter, Sch. 40 PVC Casing (0.0-236.0 ft bgs)
Neat Portland Cement (20.0-218.5 ft bgs)



ABBREVIATIONS: bgs = beneath ground surface, NA = not available, ft = feet, PVC = polyvinyl chloride, USCS = Unified Soil Classification System, in = inches, mm = millimeters, < = less than, % = percent, NAVD88 = North American Vertical Datum of 1988, NAD83 = North American Datum of 1983

NOTES: Horizontal coordinate projection: SPCS, Washington South, NAD83 Datum, U.S. Survey Feet. Elevation is reported in the NAVD88 (Geoid 18) system. Hand augered to 5.0 ft bgs or to refusal.

WELL ID: MW-07

CLIENT: Army Environmental Command
LOCATION: Yakima Training Center, WA

Project #: 30124009
Reviewed by: Jesse Hemmen, PG#2958

Logging Probes Used

- Natural Gamma
- Fluid Temperature/Resistivity
- Induction Conductivity
- Normal Resistivity
- 3-Arm Caliper
- Acoustic Televiewer
- Optical Televiewer
- Heat Pulse Flow Meter
- Spinner Flow Meter
- Spectral Gamma
- Full Waveform Sonic
- Nuclear Magnetic Resonance
- CDFM Flow Meter
- SPR/SP
- Other:

DATE DRILLING STARTED: 4/23/2023

DATE DRILLING FINISHED: 4/23/2023

DATE WELL COMPLETE: 4/24/2023

DRILLING COMPANY: Gregory Drilling

DRILLING RIG: Foremost DR-12

DRILLER'S NAME: Nicholas Pilar

DRILLER'S ASSISTANT(S): M. Grauberer, J. Davis

LOGGED BY: Roberto Piemontese

NORTHING: 485716.90 **EASTING:** 1649418.98

TOC ELEVATION: 1323.93 ft

GROUND SURFACE ELEVATION: 1321.20 ft

TOTAL BOREHOLE DEPTH: 236 feet bgs

BOREHOLE DIAMETER: 6.0 inches

DRILLING METHOD: Air Rotary

SAMPLING INTERVAL: Continuous

SAMPLING DEVICE: Cyclone

DRILLING FLUID USED: Water as Needed

WELL CONSTRUCTION

WELL CASING: Schedule 40 PVC

WELL DIAMETER: 2.0 inches

WELL SCREEN: Schedule 40 PVC

SCREEN DIAMETER: 2.0 inches

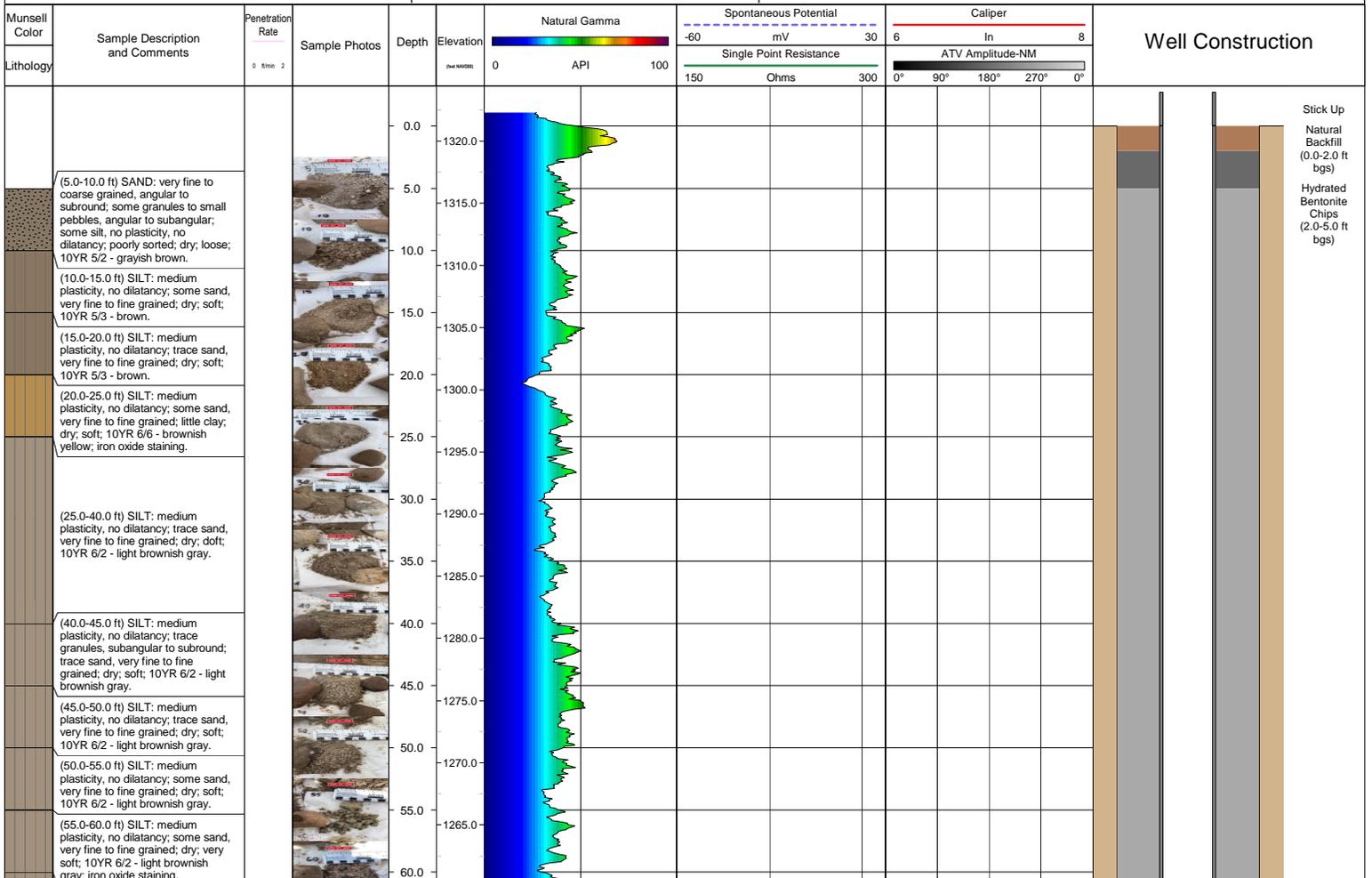
SLOT SIZE: 0.010 inches

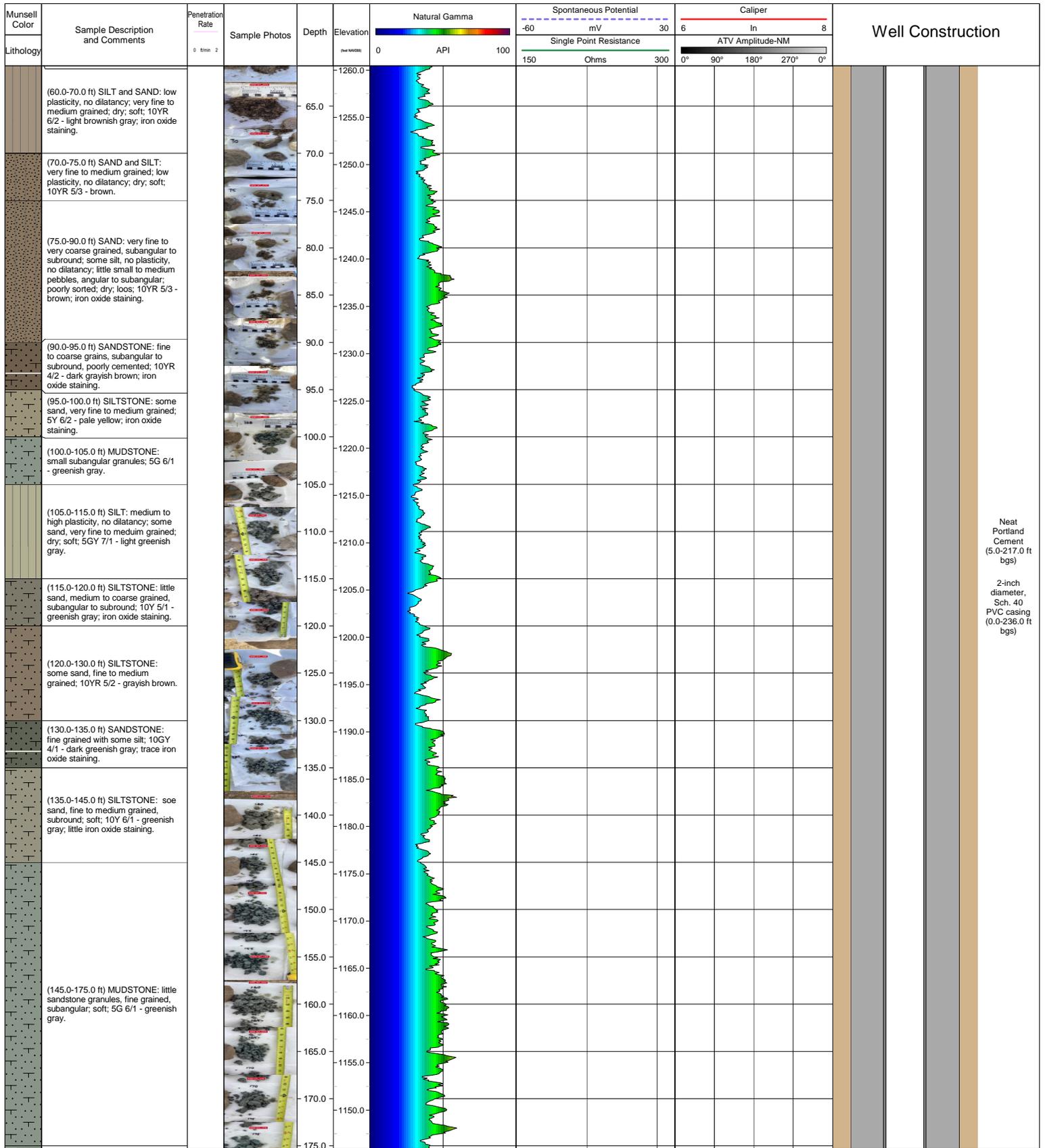
SAND PACK: #12 Sand Pack

ANNULUS SEAL: Hydrated Bentonite Chips

GROUT: Neat Portland Cement

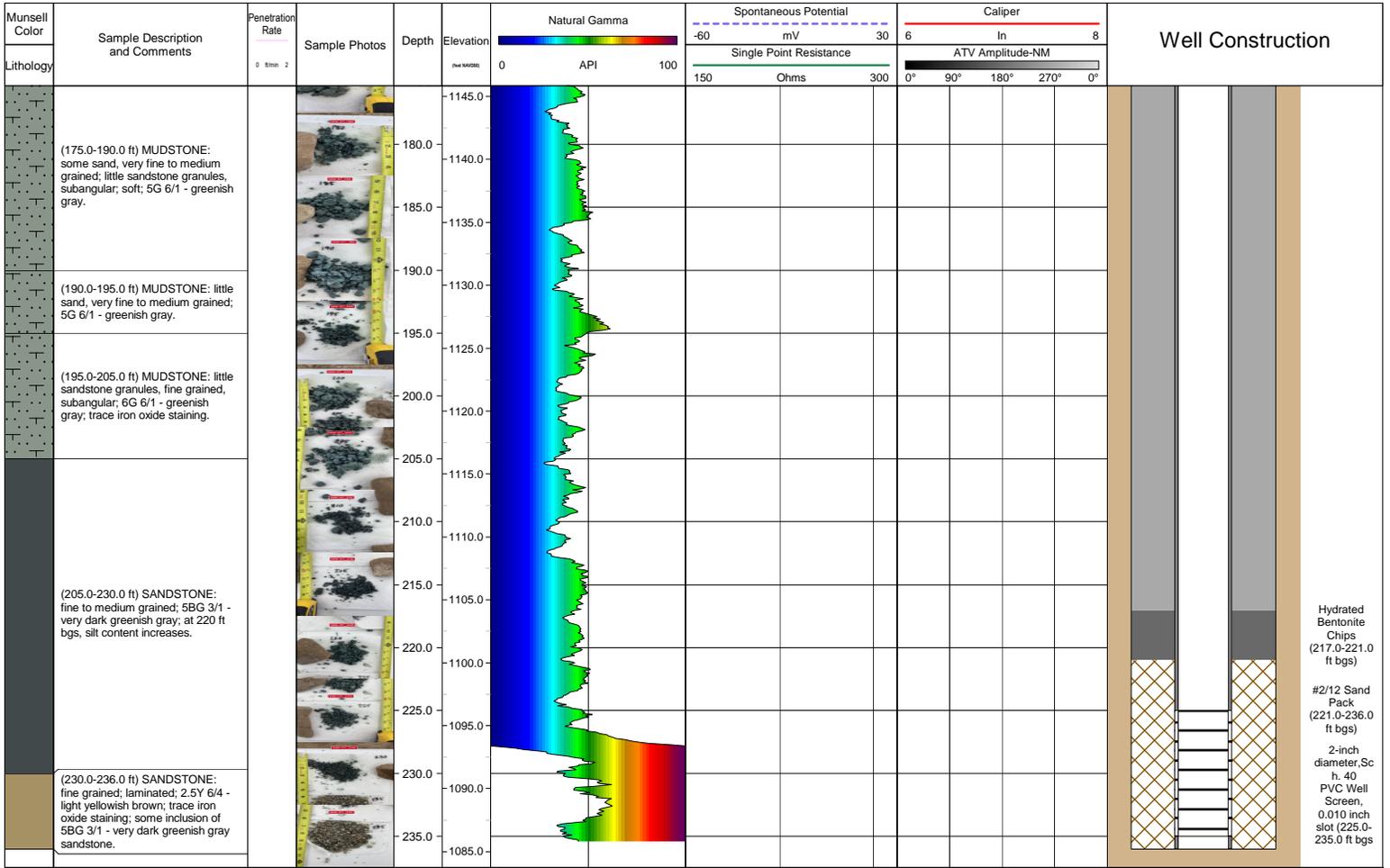
COMPLETION TYPE: Stick Up





Neat Portland Cement (5.0-217.0 ft bgs)

2-inch diameter, Sch. 40 PVC casing (0.0-236.0 ft bgs)



ABBREVIATIONS: bgs = beneath ground surface, NA = not available, ft = feet, PVC = polyvinyl chloride, USCS = Unified Soil Classification System, in = inches, mm = millimeters, < = less than, % = percent, NAVD88 = North American Vertical Datum of 1988, NAD83 = North American Datum of 1983

NOTES: Horizontal coordinate projection: SPCS, Washington South, NAD83 Datum, U.S. Survey Feet. Elevation is reported in the NAVD88 (Geoid 18) system. Hand augered to 5.0 ft bgs or to refusal.

WELL ID: MW-08

CLIENT: Army Environmental Command
LOCATION: Yakima Training Center, WA

Project #: 30124009
Reviewed by: Jesse Hemmen, PG#2958

Logging Probes Used

- Natural Gamma
- Fluid Temperature/Resistivity
- Induction Conductivity
- Normal Resistivity
- 3-Arm Caliper
- Acoustic Televiewer
- Optical Televiewer
- Heat Pulse Flow Meter
- Spinner Flow Meter
- Spectral Gamma
- Full Waveform Sonic
- Nuclear Magnetic Resonance
- CDFM Flow Meter
- SPR/SP
- Other:

DATE DRILLING STARTED: 4/23/2023

NORTHING: 485710.84 **EASTING:** 1650395.35

WELL CONSTRUCTION

DATE DRILLING FINISHED: 4/23/2023

TOC ELEVATION: 1360.67 ft

WELL CASING: Schedule 40 PVC

DATE WELL COMPLETE: 4/24/2023

GROUND SURFACE ELEVATION: 1358.00 ft

WELL DIAMETER: 2.0 inches

DRILLING COMPANY: Gregory Drilling

TOTAL BOREHOLE DEPTH: 210 feet bgs

WELL SCREEN: Schedule 40 PVC

DRILLING RIG: Foremost DR-12

BOREHOLE DIAMETER: 6.0 inches

SCREEN DIAMETER: 2.0 inches

DRILLER'S NAME: Nicholas Pilar

DRILLING METHOD: Air Rotary

SLOT SIZE: 0.010 inches

DRILLER'S ASSISTANT(S): M. Grauberer, J. Davis

SAMPLING INTERVAL: Continuous

SAND PACK: #12/12 Sand Pack

LOGGED BY: Larissa Sleeper

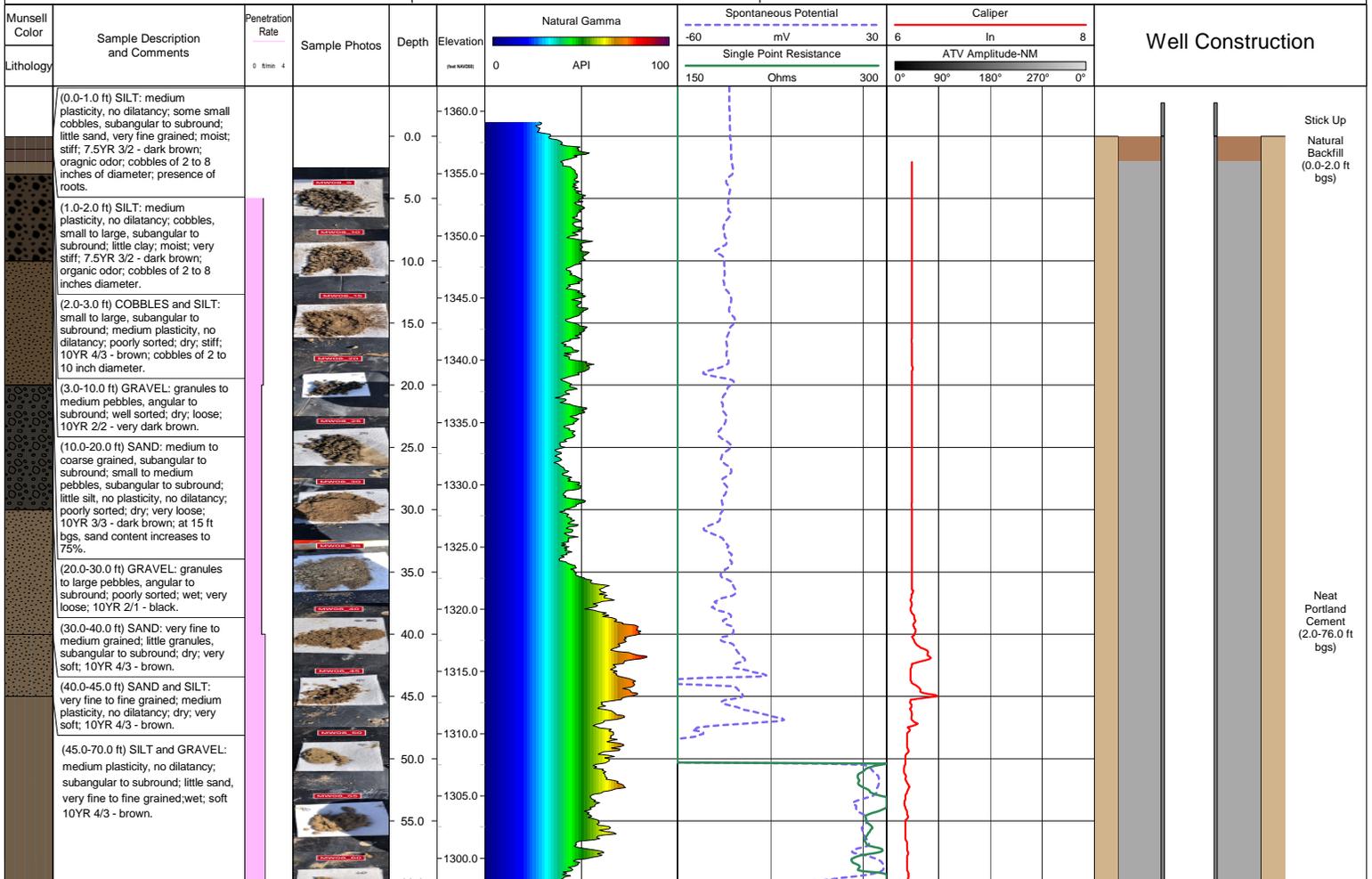
SAMPLING DEVICE: Cyclone

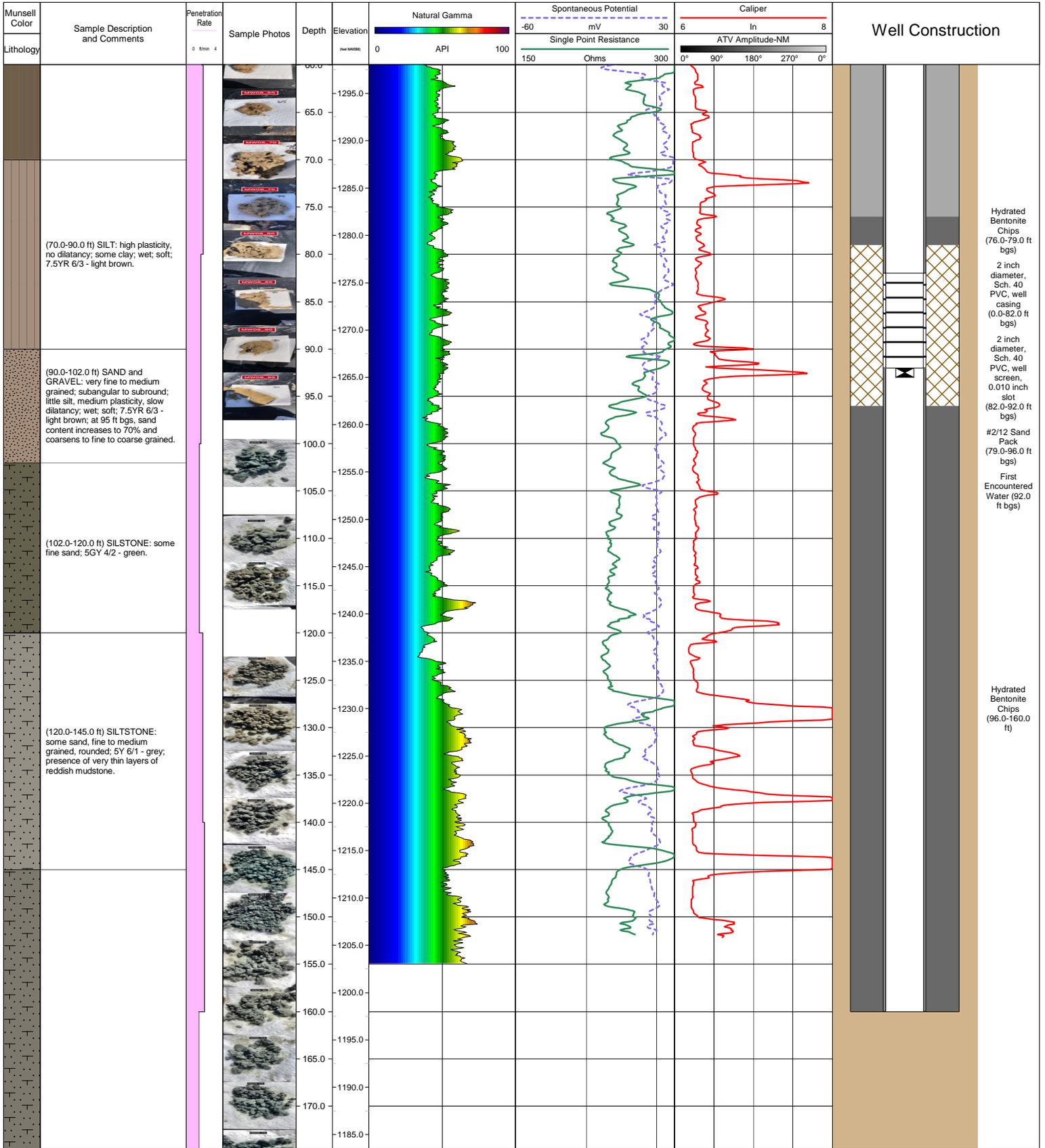
ANNULUS SEAL: Hydrated Bentonite Chips

GROUT: Neat Portland Cement

COMPLETION TYPE: Stick Up

DRILLING FLUID USED: Water as Needed





Hydrated Bentonite Chips (76.0-79.0 ft bgs)
 2 inch diameter, Sch. 40 PVC, well casing (0.0-82.0 ft bgs)
 2 inch diameter, Sch. 40 PVC, well screen, 0.010 inch slot (82.0-92.0 ft bgs)
 #2/12 Sand Pack (79.0-96.0 ft bgs)
 First Encountered Water (92.0 ft bgs)

Hydrated Bentonite Chips (96.0-160.0 ft)

| Munsell Color | Sample Description and Comments | Penetration Rate | Sample Photos | Depth | Elevation <small>(ft MVD)</small> | Natural Gamma | Spontaneous Potential | Caliper | Well Construction | | |
|---------------|--|------------------|---------------|--------|--------------------------------------|---------------|-----------------------|---------|-------------------|--|--|
| Lithology | | | | | | 0 100 API | -60 mV 30 | 6 In 8 | | | |
| | | | | | | 150 Ohms 300 | 0° 90° 180° 270° 0° | | | | |
| | (145.0-210.0 ft) SILTSTONE: some sand, fine to medium grained, round; 5GY 4/2 - green; at 160 ft bgs, presence of thin layers of mudstone, 2.5Y 5/1 - grey; occasional traces of sandstone, 10YR 7/6 - orangish brown. | | | 175.0 | | | | | | | |
| | | | | 1180.0 | | | | | | | |
| | | | | 180.0 | | | | | | | |
| | | | | 1175.0 | | | | | | | |
| | | | | 185.0 | | | | | | | |
| | | | | 1170.0 | | | | | | | |
| | | | | 190.0 | | | | | | | |
| | | | | 1165.0 | | | | | | | |
| | | | | 195.0 | | | | | | | |
| | | | | 1160.0 | | | | | | | |
| | | | | 200.0 | | | | | | | |
| | | | | 1155.0 | | | | | | | |
| | | | | 205.0 | | | | | | | |
| | | | | 1150.0 | | | | | | | |
| | 210.0 | | | | | | | | | | |
| | 1145.0 | | | | | | | | | | |
| | 215.0 | | | | | | | | | | |
| | 1140.0 | | | | | | | | | | |
| | 220.0 | | | | | | | | | | |
| | 1135.0 | | | | | | | | | | |
| | 225.0 | | | | | | | | | | |
| | 1130.0 | | | | | | | | | | |
| | 230.0 | | | | | | | | | | |
| | 1125.0 | | | | | | | | | | |
| | 235.0 | | | | | | | | | | |

ABBREVIATIONS: bgs = beneath ground surface, NA = not available, ft = feet, PVC = polyvinyl chloride, USCS = Unified Soil Classification System, in = inches, mm = millimeters, < = less than, % = percent, NAVD88 = North American Vertical Datum of 1988, NAD83 = North American Datum of 1983

NOTES: Horizontal coordinate projection: SPCS, Washington South, NAD83 Datum, U.S. Survey Feet. Elevation is reported in the NAVD88 (Geoid 16) system. Hand augered to 5.0 ft bgs or to refusal.

Appendix E
Boundary Investigation Data Validation Reports
(pending)

Appendix F
Office of the Secretary of Defense 2022 Memorandum: Investigating Per- and
Polyfluoroalkyl Substances within the Department of Defense Cleanup
Program. July 6



OFFICE OF THE ASSISTANT SECRETARY OF DEFENSE

3400 DEFENSE PENTAGON
WASHINGTON, DC 20301-3400

ENERGY, INSTALLATIONS,
AND ENVIRONMENT

July 6, 2022

MEMORANDUM FOR ASSISTANT SECRETARY OF THE ARMY (INSTALLATIONS,
ENERGY AND ENVIRONMENT)
ASSISTANT SECRETARY OF THE NAVY (ENERGY,
INSTALLATIONS AND ENVIRONMENT)
ASSISTANT SECRETARY OF THE AIR FORCE
(INSTALLATIONS, ENVIRONMENT AND ENERGY)
DIRECTOR, NATIONAL GUARD BUREAU (JOINT STAFF, J8)
DIRECTOR, DEFENSE LOGISTICS AGENCY (INSTALLATION
MANAGEMENT)

SUBJECT: Investigating Per- and Polyfluoroalkyl Substances within the Department of Defense
Cleanup Program

The Department of Defense (DoD) conducts cleanup under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), and the Defense Environmental Restoration Program (DERP). Our goal is protection of human health and the environment in a risk-based, fiscally-sound manner. This memorandum provides clarifying technical guidance on the investigation of perfluorooctane sulfonate (PFOS), perfluorooctanoic acid (PFOA), perfluorobutanesulfonic acid (PFBS), perfluorononanoic acid (PFNA), perfluorohexane sulfonate (PFHxS), and hexafluoropropylene oxide dimer acid (HFPO-DA, or GenX), based on recent U.S. Environmental Protection Agency (EPA) information. This guidance is applicable to investigating these chemicals at Environmental Restoration Account-funded, Base Realignment and Closure Account-funded, and federal Air and Army Guard Operation and Maintenance account-funded sites.

This revised memorandum accounts for the May 2022 EPA screening levels for PFOS, PFOA, PFNA, PFHxS and HFPO-DA. PFBS remains unchanged since the May 2021 update. EPA has provided screening levels for these PFAS compounds using, updated, final, peer-reviewed information from the Agency for Toxic Substances and Disease Registry¹ and the EPA Office of Water.²

PFOS, PFOA, PFBS, PFNA, PFHxS, and HFPO-DA are part of a larger class of chemicals known as per- and polyfluoroalkyl substances (PFAS). PFAS shall be addressed in the same manner as other contaminants of concern within the DERP. HFPO-DA has primarily

¹ Agency for Toxic Substances and Disease Registry (ATSDR), May 2021. *Toxicological Profile for Perfluoroalkyls*.

² U.S. Environmental Protection Agency (EPA), *Provisional Peer-Reviewed Toxicity Values for Perfluorobutane Sulfonic Acid (CASRN 375-73-5)* and October 2021. *Human Health Toxicity Values for Hexafluoropropylene Oxide (HFPO) Dimer Acid and Its Ammonium Salt (CASRN 13252-13-6 and CASRN 62037-80-3), Also Known as "GenX Chemicals."* Office of Water.

been used as a replacement for PFOA in the manufacture of fluoropolymers, so it is not likely to have been released at the vast majority of DoD properties. As with other chemicals, the conceptual site model should be used to determine the necessity for addressing HFPO-DA.

Under CERCLA, site-specific regional screening levels³ (RSLs) for these chemicals are shown in the EPA RSL Tables or may be calculated using the EPA online calculator. The values are provided in the attachment. When multiple PFAS are encountered at a site, RSLs set at a hazard quotient of 0.1 are used for screening purposes. These RSLs should be used to determine if further investigation in the remedial investigation (RI) phase is warranted or if no further action is required. Consistent with the CERCLA process, DoD Components will incorporate these screening values into ongoing and future preliminary assessment/site inspections (PA/SI) and will reevaluate completed PA/SIs with a determination of “no further action,” to assess if an RI is now necessary.

During the RI phase, the RfDs for PFOS, PFOA, PFBS, PFNA, PFHxS, and HPFO-DA and the oral cancer slope factor (CSF) for PFOA of $0.07 \text{ (mg/kg-day)}^{-1}$ will be used to conduct site specific risk assessments in accordance with Risk Assessment Guidance for Superfund Volume I, Part A (EPA/540/1-89/002, December 1989).⁴ Site-specific risk assessment results will depend on the levels of PFAS found at each site, and will be used to determine if any necessary remedial actions are required in accordance with CERCLA, DERP, and the National Oil and Hazardous Substances Pollution Contingency Plan (NCP).

This memorandum is effective immediately and supersedes and cancels the Assistant Secretary of Defense for Sustainment memorandum, “Investigating Per- and Polyfluoroalkyl Substances within the Department of Defense Cleanup Program,” September 15, 2021. The point of contact for this matter is Ms. Alexandria Long, at 703-571-9061 or alexandria.d.long.civ@mail.mil.

MCANDREW.MIC
HAEL.1043243000
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000
Date: 2022.07.06 13:39:15 -04'00'

Michael McAndrew
Deputy Assistant Secretary of Defense for
Construction
Performing the Duties of Principal Deputy
Assistant Secretary of Defense for Energy,
Installations, and Environment

Attachment:
As stated

³ For sites on the National Priorities List, the DoD Components will use the EPA site specific screening levels, if provided.

⁴ Currently there are six PFAS – PFOS, PFOA, PFBS, PFNA, PFHxS, HPFO-DA (GenX) – with established toxicity values that DoD can use to perform a baseline risk assessment to determine whether remedial action is needed under CERCLA.

Attachment: Risk Screening Levels Calculated for PFOS, PFOA, PFBS, PFNA, PFHxA, HFPO-DA in Groundwater or Soil Using EPA's RSL Calculator

| Chemical | Carcinogenic Slope Factor - Oral (SF) (mg/kg-day) ⁻¹ | Non-Carcinogenic Reference Dose (RfD) (mg/kg-day) | Residential Scenario Screening Levels Calculated Using EPA RSL Calculator | | | | | | | | Industrial/Commercial Composite Worker Screening Levels Calculated Using EPA RSL Calculator | | | |
|----------|---|---|---|----------|--------------|--------------|---------------------|----------|--------------|--------------|---|----------|--------------|--------------|
| | | | Tap Water (ng/L or ppt) | | | | Soil (mg/kg or ppm) | | | | Soil (mg/kg or ppm) | | | |
| | | | HQ = 0.1 | HQ = 1.0 | ILCR = 1E-06 | ILCR = 1E-04 | HQ = 0.1 | HQ = 1.0 | ILCR = 1E-06 | ILCR = 1E-04 | HQ = 0.1 | HQ = 1.0 | ILCR = 1E-06 | ILCR = 1E-04 |
| PFOS | NA | 2.00E-06 | 4 | 40 | NA | NA | 0.013 | 0.13 | NA | NA | 0.16 | 1.6 | NA | NA |
| PFOA | 7.00E-02 | 3.00E-06 | 6 | 60 | 1,100 | 111,000 | 0.019 | 0.19 | 7.8 | 775 | 0.25 | 2.5 | 33 | 3,280 |
| PFBS | NA | 3.00E-04 | 601 | 6010 | NA | NA | 1.9 | 19 | NA | NA | 25 | 250 | NA | NA |
| PFNA | NA | 3.00E-06 | 6 | 59 | NA | NA | 0.019 | 0.19 | NA | NA | 0.25 | 2.5 | NA | NA |
| PFHxA | NA | 2.00E-05 | 39 | 394 | NA | NA | 0.13 | 1.30 | NA | NA | 1.6 | 16 | NA | NA |
| HFPO-DA | NA | 3.00E-06 | 6 | 60 | NA | NA | 0.023 | 0.23 | NA | NA | 0.35 | 3.5 | NA | NA |

HQ=Hazard Quotient

ILCR=Incremental Lifetime Cancer Risk

NA=Not available/applicable

NOTES:

- The table represents screening levels based on residential and industrial/commercial worker receptor scenarios for either direct ingestion of groundwater (residential scenario only) or incidental ingestion of soil (both residential and composite worker scenarios).
- Default exposure assumptions for each potential receptor scenario, contained in EPA's RSL Calculator on May 2022.
- Final peer reviewed toxicity values considered valid for risk assessment, and the screening levels may be found in EPA's RSL table or EPA's RSL calculator used to develop them.
- Other potential receptor scenarios (e.g., recreational user, site trespasser, construction worker) are not included in the above table, but could be relevant receptors at a site potentially containing PFAS. These receptors, and their associated exposure scenarios, should be further considered in the scoping phase and completion of the Baseline Human Health Risk Assessment typically completed during an RI.
- The shaded values represent conservative screening levels in groundwater or soil that when exceeded should be considered a contaminant of potential concern in the risk assessment process and calculations of site-specific risk posed.

Appendix G
Alternate Laboratory (ELLE) UFP-QAPP Worksheets, Certifications and
Standard Operating Procedures



SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

EUROFINS LANCASTER LABORATORIES ENVIRONMENT TESTING LLC

2425 New Holland Pike

Lancaster, PA 17601

Kenneth Boley Phone: 717-556-9413

ENVIRONMENTAL

Valid To: November 30, 2024

Certificate Number: 0001.01

In recognition of the successful completion of the A2LA evaluation process (including an assessment of the laboratory's compliance with the 2009 TNI Environmental Testing Laboratory Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.4 of the DoD/DOE Quality Systems Manual for Environmental Laboratories, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP-MS Spectrometry, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.-Electronic Probes (pH, F⁻, O₂), Oxygen Demand, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, TCLP, Total Organic Carbon, Turbidity, Liquid Chromatography/Mass Spectrometry/Mass Spectrometry, High Resolution Gas Chromatography/Mass Spectrometry

| <u>Parameter/Analyte</u> | <u>Drinking Water</u> | <u>Non-Potable Water</u> | <u>Solid Hazardous Waste</u> | |
|--------------------------|-----------------------|-------------------------------------|-------------------------------------|--|
| | | | <u>Aqueous</u> | <u>Solid</u> |
| Demands | | | | |
| COD | ----- | EPA 410.4 | EPA 410.4 | ----- |
| Total Organic Carbon | ----- | EPA 9060A SM 5310C-2014 | EPA 9060A SM 5310C-2014 | EPA 9060A SM 5310C-2014 Lloyd Kahn |
| Anions | | | | |
| Ammonia | ----- | EPA 350.1 | EPA 350.1 | SM 4500-NH3 B/C-2011 |
| Fluoride | ----- | EPA 300.0 EPA 9056A | EPA 300.0 EPA 9056A | EPA 300.0 EPA 9056A |
| Nitrate (as N) | ----- | EPA 300.0 EPA 353.2 EPA 9056A | EPA 300.0 EPA 353.2 EPA 9056A | EPA 300.0 EPA 353.2 EPA 9056A |
| Nitrite (as N) | ----- | EPA 300.0 EPA 353.2 EPA 9056A | EPA 300.0 EPA 353.2 EPA 9056A | EPA 300.0 EPA 353.2 EPA 9056A |

| Parameter/Analyte | Drinking Water | Non-Potable Water | Solid Hazardous Waste | |
|-------------------------------|----------------|--|--|--------------------------|
| | | | Aqueous | Solid |
| Nitrate Nitrite Total | ----- | EPA 300.0 EPA 9056A | EPA 300.0 EPA 9056A | EPA 300.0 EPA 9056A |
| Bromide | ----- | EPA 300.0 EPA 9056A | EPA 300.0 EPA 9056A | ----- |
| Chloride | ----- | EPA 300.0 EPA 9056A | EPA 300.0 EPA 9056A | EPA 300.0 EPA 9056A |
| Sulfate | ----- | EPA 300.0 EPA 9056A | EPA 300.0 EPA 9056A | EPA 300.0 EPA 9056A |
| Wet Chemistry | | | | |
| Alkalinity | ----- | SM 2320B-2011 | SM 2320B-2011 | ----- |
| Corrosivity | ----- | ----- | SW-846 Chapter 7 | SW-846 Chapter 7 |
| Conductivity | | SM 2510B-2011 | SM 2510B-2011 | ----- |
| Cyanide | ----- | EPA 9012B | EPA 9012B | EPA 9012B |
| Filterable Residue (TDS) | ----- | SM 2540C-2015 | SM 2540C-2015 | ----- |
| Flashpoint | ----- | EPA 1010A/B | EPA 1010A/B | EPA 1010A/B |
| Grain Size | ----- | ----- | ----- | ASTM D422 MOD |
| Hardness | ----- | EPA 130.2 SM 2340B-2011 SM 2340C-2011 | EPA 130.2 SM 2340B-2011 SM 2340C-2011 | ----- |
| Hexavalent Chromium Digestion | ----- | ----- | ----- | EPA 3060A |
| Hexavalent Chromium | ----- | EPA 218.6 EPA 7196A EPA 7199 | EPA 7196A EPA 7199 | EPA 7196A EPA 7199 |
| Ignitability | ----- | ----- | 40 CFR 261.21 | 40 CFR 261.21 |
| Non-filterable Residue (TSS) | ----- | SM 2540D-2015 | SM 2540D-2015 | ----- |
| Paint Filter | ----- | ----- | ----- | EPA 9095B |
| pH | ----- | SM 4500 H+B-2011 EPA 9040B/C | EPA 9040B/C | EPA 9045C/D |
| Phenol | ----- | EPA 9066 | EPA 9066 | ----- |
| Reactivity Prep | ----- | ----- | SW-846 Chapter 7.3 | SW-846 Chapter 7.3 |
| Reactive Cyanide | ----- | ----- | EPA 9012B | EPA 9012B |
| Reactive Sulfide | ----- | ----- | EPA 9034 | EPA 9034 |
| Sulfide | ----- | EPA 376.1 EPA 376.2 SM 4500 S2D-2011 SM 4500 S2F-2011 | EPA 376.1 EPA 376.2 SM 4500 S2D-2011 SM 4500 S2F-2011 | ----- |
| Total Kjeldahl Nitrogen (TKN) | ----- | EPA 351.2 | EPA 351.2 | EPA 351.2 |
| Total Residue | ----- | SM 2540B-2015 | SM 2540B-2015 | SM 2540G-2015 |
| Metals | | | | |
| Metals Digestion | ----- | EPA 3005A | EPA 3005A | EPA 3050B |
| Aluminum | EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |

| <u>Parameter/Analyte</u> | <u>Drinking Water</u> | <u>Non-Potable Water</u> | <u>Solid Hazardous Waste</u> | |
|--------------------------|------------------------|--|------------------------------|--------------------------|
| | | | <u>Aqueous</u> | <u>Solid</u> |
| Antimony | EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Arsenic | EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Barium | EPA 200.7 EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Beryllium | EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Boron | ----- | EPA 200.7 EPA 6010C/D | EPA 6010C/D | EPA 6010C/D |
| Cadmium | EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Calcium | EPA 200.7 EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Chromium | EPA 200.7 EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Cobalt | EPA 200.7 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Copper | EPA 200.7 EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Iron | EPA 200.7 EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Lead | EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |

| <u>Parameter/Analyte</u> | <u>Drinking Water</u> | <u>Non-Potable Water</u> | <u>Solid Hazardous Waste</u> | |
|--------------------------|------------------------|--|------------------------------|--------------------------|
| | | | <u>Aqueous</u> | <u>Solid</u> |
| Lithium | EPA 200.7 | EPA 200.7 EPA 6010C/D | EPA 6010C/D | EPA 6010C/D |
| Molybdenum | ----- | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Magnesium | EPA 200.7 EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Manganese | EPA 200.7 EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Mercury | EPA 245.1 | EPA 245.1 EPA 7470A | EPA 245.1 EPA 7470A | EPA 7471A EPA 7471B |
| Nickel | EPA 200.7 EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Potassium | EPA 200.7 EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Selenium | EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Silicon | ----- | EPA 200.7 EPA 6010C/D | EPA 6010C/D | EPA 6010C/D |
| Silver | EPA 200.7 EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Sodium | EPA 200.7 EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Strontium | EPA 200.7 EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Sulfur | EPA 200.7 | EPA 200.7 EPA 6010C/D | EPA 6010C/D | EPA 6010C/D |
| Thallium | EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |

| <u>Parameter/Analyte</u> | <u>Drinking Water</u> | <u>Non-Potable Water</u> | <u>Solid Hazardous Waste</u> | |
|---|------------------------|--|------------------------------|--------------------------|
| | | | <u>Aqueous</u> | <u>Solid</u> |
| Thorium | ----- | EPA 6010C/D | EPA 6010C/D | EPA 6010C/D |
| Tin | EPA 200.7 | EPA 200.7 EPA 6010C/D | EPA 6010C/D | EPA 6010C/D |
| Titanium | ----- | EPA 200.7 EPA 200.8 EPA 6010C/D | EPA 6010C/D | EPA 6010C/D |
| Tungsten | ----- | EPA 6010C/D | EPA 6010C/D | EPA 6010C/D |
| Uranium | ----- | EPA 200.8 EPA 6020B | EPA 6020B | EPA 6020B |
| Vanadium | EPA 200.7 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Zinc | EPA 200.7 EPA 200.8 | EPA 200.7 EPA 200.8 EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B | EPA 6010C/D EPA 6020B |
| Zirconium | ----- | EPA 6010C/D | EPA 6010C/D | EPA 6010C/D |
| Purgeable Organics (Volatiles) | | | | |
| Volatile Preparation | ----- | EPA 5030C | EPA 5030C | EPA 5035A |
| Acetone | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Acetonitrile | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Acrolein | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Acrylonitrile | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Allyl chloride | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| tert-Amyl Alcohol | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| tert-Amyl Methyl Ether | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| tert-Butyl Alcohol | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| tert-Butyl Formate | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Benzene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Bromobenzene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Bromochloromethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Bromodichloromethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Bromoform | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Bromomethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 2-Butanone | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| n-Butylbenzene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| sec-Butylbenzene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| tert-Butylbenzene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Carbon disulfide | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Carbon tetrachloride | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 2-Chloro-1,3-butadiene | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |

| <u>Parameter/Analyte</u> | <u>Drinking Water</u> | <u>Non-Potable Water</u> | <u>Solid Hazardous Waste</u> | |
|-----------------------------|-----------------------|--------------------------------|--------------------------------|--------------------------------|
| | | | <u>Aqueous</u> | <u>Solid</u> |
| Chloroacetonitrile | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Chlorobenzene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1-Chlorobutane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Chlorodifluoromethane | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Chloroethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 2-Chloroethyl Vinyl Ether | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Chloroform | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1-Chlorohexane | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Chloromethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 2-Chlorotoluene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 4-Chlorotoluene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Cyclohexane | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Cyclohexanone | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Di-Isopropyl ether | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Dibromochloromethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,2-Dibromo-3-chloropropane | EPA 524.2 | EPA 8260C/D EPA 8011 | EPA 8260C/D EPA 8011 | EPA 8260C/D |
| Dibromomethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,2-Dibromoethane (EDB) | ----- | EPA 8260C/D EPA 8011 | EPA 8260C/D EPA 8011 | EPA 8260C/D |
| 1,2-Dichlorobenzene | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,3-Dichlorobenzene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,4-Dichlorobenzene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| trans-1,4-dichloro-2-butene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Dichlorodi-fluoromethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,1-Dichloroethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,2-Dichloroethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,1-Dichloroethene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| cis-1,2-Dichloroethene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| trans-1,2-Dichloroethene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Dichlorofluoromethane | EPA 524.2 | ----- | ----- | ----- |
| 1,2-Dichloropropane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,3-Dichloropropane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 2,2-Dichloropropane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,1-Dichloropropene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| cis-1,3-Dichloropropene | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| trans-1,3-Dichloropropene | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,4-Dioxane | ----- | EPA 8260C/D EPA 8260C/D SIM | EPA 8260C/D EPA 8260C/D SIM | EPA 8260C/D EPA 8260C/D SIM |
| Ethanol | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Ethylbenzene | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Ethyl ether | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |

| Parameter/Analyte | Drinking Water | Non-Potable Water | Solid Hazardous Waste | |
|--|----------------|---|---|---|
| | | | Aqueous | Solid |
| Ethyl Methacrylate | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Ethyl Tert-Butyl Ether | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Freon-113 | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Gasoline Range Organics (GRO) [Volatile Petroleum Hydrocarbons (VPH)] | ----- | EPA 8015C EPA 8015D EPA 8260C/D NW TPH-Gx MA VPH AK101 | EPA 8015C EPA 8015D EPA 8260C/D NW TPH-Gx MA VPH AK101 | EPA 8015C EPA 8015D EPA 8260C/D NW TPH-Gx MA VPH AK101 |
| Heptane | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Hexane | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 2-Hexanone | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Hexachlorobutadiene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Hexachloroethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Isopropyl Alcohol | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Isopropylbenzene | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,4-Isopropyltoluene | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Methylacrylonitrile | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Methyl Acetate | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Methyl Acrylate | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Methyl Iodide | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Methylene Chloride | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Methyl Methacrylate | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Methyl Tert-Butyl Ether | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 4-Methyl-2-pentanone | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Methylcyclohexane | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 2-Nitropropane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Naphthalene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Pentachloroethane | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Propionitrile | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| n-Propylbenzene | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Styrene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Tert-Amyl Ethyl Ether | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,1,1,2-Tetrachloroethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,1,2,2-Tetrachloroethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Tetrachloroethene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Tetrahydrofuran | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Toluene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,2,3-Trichlorobenzene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,2,4-Trichlorobenzene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,1,1-Trichloroethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,1,2-Trichloroethane | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |

| Parameter/Analyte | Drinking Water | Non-Potable Water | Solid Hazardous Waste | |
|---|----------------|--------------------------------|--------------------------------|--------------------------------|
| | | | Aqueous | Solid |
| Trichloroethene | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Trichlorofluoromethane | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,2,3-Trichloropropane | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,2,4-Trimethylbenzene | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,3,5-Trimethylbenzene | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 130BVinyl Acetate | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Vinyl Chloride | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Xylenes, Total | ----- | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,2-Xylene (o-Xylene) | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| 1,3+1,4-Xylene (m+p Xylene) | EPA 524.2 | EPA 8260C/D | EPA 8260C/D | EPA 8260C/D |
| Extractable Organics (Semivolatiles) | | | | |
| Acenaphthene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Acenaphthylene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Acetophenone | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2-Acetylaminofluorene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Alkylated PAHs | ----- | EPA 8270D/E SIM | EPA 8270D/E SIM | EPA 8270D/E SIM |
| 4-Aminobiphenyl | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2-Amino-4,6-dinitrotoluene | ----- | EPA 8330B | EPA 8330B | ----- |
| 4-Amino-2,6-dinitrotoluene | ----- | EPA 8330B | EPA 8330B | ----- |
| Aniline | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Anthracene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Atrazine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Benzaldehyde | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Benzidine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Benzoic acid | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Benzo (a) anthracene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Benzo (b) fluoranthene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Benzo (k) fluoranthene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Benzo (ghi) perylene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Benzo (a) pyrene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Benzo (e) pyrene | ----- | EPA 8270D/E SIM | EPA 8270D/E SIM | EPA 8270D/E SIM |
| Benzyl Alcohol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Biphenyl | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |

| Parameter/Analyte | Drinking Water | Non-Potable Water | Solid Hazardous Waste | |
|--|----------------|---|---|---|
| | | | Aqueous | Solid |
| bis (2-Chloroethoxy) Methane | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| bis (2-Chloroethyl) Ether | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| bis (2-Ethylhexyl) Phthalate | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| 4-Bromophenylphenyl Ether | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Butyl benzyl Phthalate | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Caprolactam | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Carbazole | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Carbon Range Organics C8-C44 (including subsets of this range i.e. HRO, MRO, ORO, RRO) | ----- | EPA 8015C EPA 8015D | EPA 8015C EPA 8015D | EPA 8015C EPA 8015D |
| 4-Chloroaniline | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 4-Chloro-3-methylphenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Chlorobenzilate | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 1-Chloronaphthalene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2-Chloronaphthalene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2-Chlorophenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 4-Chlorophenyl phenyl ether | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Chrysene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Cresols (Methyl phenols) | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| cis-/trans-Diallate | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2,4-Diamino-6-nitrotoluene | ----- | EPA 8330B | EPA 8330B | ----- |
| 2,6-Diamino-4-nitrotoluene | ----- | EPA 8330B | EPA 8330B | ----- |
| Dibenzo (a,h) acridine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Dibenzo (a,h) anthracene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Dibenzofuran | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| 1,2-Dichlorobenzene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 1,3-Dichlorobenzene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 1,4-Dichlorobenzene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 3,3-Dichlorobenzidine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Diesel Range Organics (DRO) [Extractable Petroleum Hydrocarbons (EPH)] | ----- | EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005 AK102/103 AK102/103-SV | EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005 AK102/103 AK102/103-SV | EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005 AK102/103 |

| Parameter/Analyte | Drinking Water | Non-Potable Water | Solid Hazardous Waste | |
|---|----------------|--------------------------------|--------------------------------|--------------------------------|
| | | | Aqueous | Solid |
| 2,4-Dichlorophenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2,6-Dichlorophenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Diethyl Phthalate | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Dimethoate | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| p-Dimethylaminoazobenze | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 7,12-Dimethylbenz (a) anthracene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2,4-Dimethylphenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Dimethyl Phthalate | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| 3,3'-Dimethylbenzidine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Di-n-butyl Phthalate | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Di-n-octyl phthalate | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| 3,5-Dinitroaniline | ----- | EPA 8330B | EPA 8330B | ----- |
| 1,3-Dinitrobenzene | ----- | EPA 8270D/E EPA 8330B | EPA 8270D/E EPA 8330B | EPA 8270D/E |
| 1,4-Dinitrobenzene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2,4-Dinitrophenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2,4-Dinitrotoluene | ----- | EPA 8270D/E EPA 8330B | EPA 8270D/E EPA 8330B | EPA 8270D/E |
| 2,6-Dinitrotoluene | ----- | EPA 8270D/E EPA 8330B | EPA 8270D/E EPA 8330B | EPA 8270D/E |
| 1,4-Dioxane | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Diphenylamine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Diphenyl ether | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 1,2-Diphenylhydrazine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Ethyl Methanesulfonate | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Fluoroanthene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Fluorene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Hexachlorobenzene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Hexachlorobutadiene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Hexachlorocyclopentadiene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Hexachloroethane | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Hexachloropropene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) | ----- | EPA 8330B | EPA 8330B | ----- |

| Parameter/Analyte | Drinking Water | Non-Potable Water | Solid Hazardous Waste | |
|----------------------------|----------------|--------------------------------|--------------------------------|--------------------------------|
| | | | Aqueous | Solid |
| Indeno (1,2,3-cd) Pyrene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Isodrin | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Isophorone | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Isosafrole | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 3-Methycolanthrene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2-Methyl-4,6-dinitrophenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Methyl methane sulfonate | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 1-Methylnaphthalene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| 2-Methylnaphthalene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| 2-Methylphenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 4-Methylphenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Naphthalene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| 1,4-Naphthoquinone | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 1-Naphthylamine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2-Naphthylamine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 4-Nitroquinoline-1-oxide | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2-Nitroaniline | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 3-Nitroaniline | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 4-Nitroaniline | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Nitrobenzene | ----- | EPA 8270D/E EPA 8330B | EPA 8270D/E EPA 8330B | EPA 8270D/E |
| Nitroglycerin | ----- | EPA 8330B | EPA 8330B | ----- |
| 2-Nitrophenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 4-Nitrophenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2-Nitrotoluene | ----- | EPA 8330B | EPA 8330B | ----- |
| 3-Nitrotoluene | ----- | EPA 8330B | EPA 8330B | ----- |
| 4-Nitrotoluene | ----- | EPA 8330B | EPA 8330B | ----- |
| 5-Nitro-o-toluidine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| n-Nitroso-di-n-butylamine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| n-Nitrosodiethylamine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| n-Nitrosodimethylamine | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| n-Nitrosomethylethylamine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| n-Nitrosomorpholine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| n-Nitrosodi-n-propylamine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| n-Nitrosodiphenylamine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| n-Nitrosopiperidine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| n-Nitrosopyrrolidine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |

| Parameter/Analyte | Drinking Water | Non-Potable Water | Solid Hazardous Waste | |
|--|----------------|--------------------------------|--------------------------------|--------------------------------|
| | | | Aqueous | Solid |
| Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) | ----- | EPA 8330B | EPA 8330B | EPA 8330B MOD |
| 2,2-Oxybis (1-chloropropane) | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Pentachlorobenzene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Pentachloronitrobenzene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Pentachlorophenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Pentaerythritol Tetranitrate (PETN) | ----- | EPA 8330B | EPA 8330B | ----- |
| Perylene | ----- | EPA 8270D/E SIM | EPA 8270D/E SIM | EPA 8270D/E SIM |
| Phenacetin | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Phenanthrene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Phenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2-Picoline | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Pronamide | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Pyrene | ----- | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM | EPA 8270D/E EPA 8270D/E SIM |
| Pyridine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Safrole | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 1,2,4,5- Tetrachlorobenzene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2,3,4,6-Tetrachlorophenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Tetraethyl dithiopyrophosphate | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Tetraethyl lead | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Tetryl | ----- | EPA 8330B | EPA 8330B | ----- |
| Thionazin | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| o-Toluidine | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 1,2,4-Trichlorobenzene | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 1,3,5-Trinitrobenzene | ----- | EPA 8330B | EPA 8330B | ----- |
| 2,4,5-Trichlorophenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2,4,6-Trichlorophenol | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| O,O,O-Tri-ethylphosphorothioate | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| 2,4,6-Trinitrotoluene | ----- | EPA 8330B | EPA 8330B | ----- |
| Organochlorine Pesticides | | | | |
| Aldrin | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| alpha-BHC | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| beta-BHC | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| delta-BHC | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| gamma-BHC (Lindane) | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| alpha-Chlordane | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Chlordane (Technical) | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| 2,4'-DDD | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| 2,4'-DDE | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| 2,4'-DDT | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| 4,4'-DDD | ----- | EPA 8081B | EPA 8081B | EPA 8081B |

| <u>Parameter/Analyte</u> | <u>Drinking Water</u> | <u>Non-Potable Water</u> | <u>Solid Hazardous Waste</u> | |
|---------------------------|-----------------------|--------------------------|------------------------------|------------------------|
| | | | <u>Aqueous</u> | <u>Solid</u> |
| 4,4'-DDE | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| 4,4'-DDT | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Dieldrin | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Dinoseb | ----- | EPA 8270D/E | EPA 8270D/E | EPA 8270D/E |
| Endosulfan I (alpha) | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Endosulfan II (beta) | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Endosulfan Sulfate | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Endrin | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Endrin Aldehyde | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Endrin Ketone | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| gamma-Chlordane | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Heptachlor | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Heptachlor Epoxide | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Hexachlorobenzene | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Hexachlorocyclopentadiene | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Methoxychlor | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Mirex | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| Toxaphene | ----- | EPA 8081B | EPA 8081B | EPA 8081B |
| PCBs (Aroclors) | | | | |
| PCB-1016 (Arochlor) | ----- | EPA 8082A | EPA 8082A | EPA 8082A |
| PCB-1221 | ----- | EPA 8082A | EPA 8082A | EPA 8082A |
| PCB-1232 | ----- | EPA 8082A | EPA 8082A | EPA 8082A |
| PCB-1242 | ----- | EPA 8082A | EPA 8082A | EPA 8082A |
| PCB-1248 | ----- | EPA 8082A | EPA 8082A | EPA 8082A |
| PCB-1254 | ----- | EPA 8082A | EPA 8082A | EPA 8082A |
| PCB-1260 | ----- | EPA 8082A | EPA 8082A | EPA 8082A |
| PCB-1262 | ----- | EPA 8082A | EPA 8082A | EPA 8082A |
| PCB-1268 | ----- | EPA 8082A | EPA 8082A | EPA 8082A |
| PCB congeners (209) | ----- | EPA 1668A EPA 1668C | EPA 1668A EPA 1668C | EPA 1668A EPA 1668C |
| Herbicides | | | | |
| 2,4,5-T | ----- | EPA 8151A | EPA 8151A | EPA 8151A |
| 2,4,5-TP (Silvex) | ----- | EPA 8151A | EPA 8151A | EPA 8151A |
| 2,4-D | ----- | EPA 8151A | EPA 8151A | EPA 8151A |
| 2,4-DB | ----- | EPA 8151A | EPA 8151A | EPA 8151A |
| Dalapon | ----- | EPA 8151A | EPA 8151A | EPA 8151A |
| Dicamba | ----- | EPA 8151A | EPA 8151A | EPA 8151A |
| Dichlorprop | ----- | EPA 8151A | EPA 8151A | EPA 8151A |
| Dinoseb | ----- | EPA 8151A | EPA 8151A | EPA 8151A |
| MCPA | ----- | EPA 8151A | EPA 8151A | EPA 8151A |
| MCPP | ----- | EPA 8151A | EPA 8151A | EPA 8151A |
| Pentachlorophenol | ----- | EPA 8151A | EPA 8151A | EPA 8151A |
| PCB Homologues | | | | |
| Monochlorobiphenyls | ----- | EPA 680 | EPA 680 | EPA 680 |
| Dichlorobiphenyls | ----- | EPA 680 | EPA 680 | EPA 680 |
| Trichlorobiphenyls | ----- | EPA 680 | EPA 680 | EPA 680 |

| Parameter/Analyte | Drinking Water | Non-Potable Water | Solid Hazardous Waste | |
|---|----------------|-------------------|-----------------------|-----------|
| | | | Aqueous | Solid |
| Tetrachlorobiphenyls | ----- | EPA 680 | EPA 680 | EPA 680 |
| Pentachlorobiphenyls | ----- | EPA 680 | EPA 680 | EPA 680 |
| Hexachlorobiphenyls | ----- | EPA 680 | EPA 680 | EPA 680 |
| Heptachlorobiphenyls | ----- | EPA 680 | EPA 680 | EPA 680 |
| Octachlorobiphenyls | ----- | EPA 680 | EPA 680 | EPA 680 |
| Nonachlorobiphenyls | ----- | EPA 680 | EPA 680 | EPA 680 |
| Decachlorobiphenyls | ----- | EPA 680 | EPA 680 | EPA 680 |
| Dioxins/Furans | | | | |
| 2,3,7,8-TCDD | EPA 1613B | EPA 8290A | EPA 8290A | EPA 8290A |
| 2,3,7,8-TCDF | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| 1,2,3,7,8-PeCDF | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| 2,3,4,7,8-PeCDF | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| 1,2,3,7,8-PeCDD | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| 1,2,3,4,7,8-HxCDF | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| 1,2,3,6,7,8-HxCDF | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| 2,3,4,6,7,8-HxCDF | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| 1,2,3,7,8,9-HxCDF | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| 1,2,3,4,7,8,-HxCDD | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| 1,2,3,6,7,8-HxCDD | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| 1,2,3,7,8,9-HxCDD | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| 1,2,3,4,6,7,8-HpCDF | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| 1,2,3,4,7,8,9-HpCDF | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| 1,2,3,4,6,7,8-HpCDD | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| OCDF | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| OCDD | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| Total HpCDD | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| Total HpCDF | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| Total HxCDD | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| Total HxCDF | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| Total PeCDD | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| Total PeCDF | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| Total TCDD | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| Total TCDF | ----- | EPA 8290A | EPA 8290A | EPA 8290A |
| Misc. Headspace Analysis | | | | |
| Carbon dioxide | ----- | RSK-175 | RSK-175 | ----- |
| Ethane | ----- | RSK-175 | RSK-175 | ----- |
| Ethene | ----- | RSK-175 | RSK-175 | ----- |
| Methane | ----- | RSK-175 | RSK-175 | ----- |
| Acetylene | ----- | RSK-175 | RSK-175 | ----- |
| Propane | ----- | RSK-175 | RSK-175 | ----- |
| Hazardous Waste Characteristics | | | | |
| 342B Toxicity Characteristic Leaching Procedure | ----- | ----- | EPA 1311 | EPA 1311 |
| 343B Synthetic Precipitation Leaching Procedure | ----- | ----- | EPA 1312 | EPA 1312 |

| Parameter/Analyte | Drinking Water | Non-Potable Water | Solid Hazardous Waste | |
|---------------------------------|----------------|---|---|--|
| | | | Aqueous | Solid |
| 344BASTM Leaching Procedure | ----- | ----- | ASTM D3987-85 | ASTM D3987-85 |
| Other | | | | |
| Perchlorate | ----- | EPA 6850 | EPA 6850 | EPA 6850 |
| Hydrazine | ----- | EPA 8315A MOD | EPA 8315A MOD | EPA 8315A MOD |
| Formaldehyde | ----- | ----- | EPA 8315A | EPA 8315A |
| Methylhydrazine | ----- | EPA 8315A MOD | EPA 8315A MOD | EPA 8315A MOD |
| 1,1-Dimethylhydrazine | ----- | EPA 8315A MOD | EPA 8315A MOD | EPA 8315A MOD |
| Acetic Acid | ----- | EPA 8015D | EPA 8015D | ----- |
| Butyric acid | ----- | EPA 8015D | EPA 8015D | ----- |
| Lactic Acid | ----- | EPA 8015D | EPA 8015D | ----- |
| Propionic Acid | ----- | EPA 8015D | EPA 8015D | ----- |
| Pyruvic Acid | ----- | EPA 8015D | EPA 8015D | ----- |
| Citric Acid | ----- | EPA 8015D | EPA 8015D | ----- |
| Formic Acid | ----- | EPA 8015D | EPA 8015D | ----- |
| Oxalic Acid | ----- | EPA 8015D | EPA 8015D | ----- |
| Quinic Acid | ----- | EPA 8015D | EPA 8015D | ----- |
| Succinic Acid | ----- | EPA 8015D | EPA 8015D | ----- |
| Tartaric Acid | ----- | EPA 8015D | EPA 8015D | ----- |
| Volatile Preparation | ----- | EPA 5030C | EPA 5030C | EPA 5035 EPA 5035A |
| 352B Organic Extraction/Cleanup | ----- | EPA 3510C EPA 3511 EPA 3660B, 3620C, 3665A | EPA 3510C EPA 3511 EPA 3660B, 3620C, 3665A | EPA 3546 EPA 3550C EPA 3660B, 3620C, 3665A, 3640A |

| Parameter/Analyte | Drinking Water | Nonpotable Water | Solid Haz. Waste |
|---|---------------------------------|---|---|
| Per and Polyfluoroalkyl Substances (PFAS) | | | |
| N-ethyl Perfluorooctanesulfonamidoacetic Acid (NEtFOSAA) | EPA 537 EPA 537.1 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| N-methyl perfluorooctanesulfonamidoacetic Acid (NMeFOSAA) | EPA 537 EPA 537.1 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluorobutanesulfonic Acid (PFBS) | EPA 537 EPA 537.1 EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluorodecanoic Acid (PFDA) | EPA 537 EPA 537.1 EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 |

| Parameter/Analyte | Drinking Water | Nonpotable Water | Solid Haz.Waste |
|--------------------------------------|---------------------------------|---|---|
| | | EPA Draft Method 1633 | EPA Draft Method 1633 |
| Perfluorododecanoic Acid (PFDoA) | EPA 537 EPA 537.1 EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluoroheptanoic Acid (PFHpA) | EPA 537 EPA 537.1 EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluorohexanesulfonic Acid (PFHxS) | EPA 537 EPA 537.1 EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluorohexanoic Acid (PFHxA) | EPA 537 EPA 537.1 EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluorononanoic Acid (PFNA) | EPA 537 EPA 537.1 EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluorooctanesulfonic Acid (PFOS) | EPA 537 EPA 537.1 EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluorooctanoic Acid (PFOA) | EPA 537 EPA 537.1 EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluorotetradecanoic Acid (PFTeDA) | EPA 537 EPA 537.1 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluorotridecanoic Acid (PFTrDA) | EPA 537 EPA 537.1 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluoroundecanoic Acid (PFUnA) | EPA 537 EPA 537.1 EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 |

| Parameter/Analyte | Drinking Water | Nonpotable Water | Solid Haz.Waste |
|--|-----------------------|---|---|
| | | EPA Draft Method 1633 | EPA Draft Method 1633 |
| Hexafluoropropylene oxide dimer acid (HF-PODA) | EPA 537.1 EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| 4,8-Dioxa-3H-perfluorononanoic acid (ADONA) | EPA 537.1 EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS) | EPA 537.1 EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS) | EPA 537.1 EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluorobutanoic Acid (PFBA) | EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluoropentanoic Acid (PFPeA) | EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| 1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2FTS) | EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| 1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2-FTS) | EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluoropentanesulfonic Acid (PFPeS) | EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |

| Parameter/Analyte | Drinking Water | Nonpotable Water | Solid Haz.Waste |
|---|-----------------------|---|---|
| 1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2-FTS) | EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluoroheptanesulfonic Acid (PFHpS) | EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluorononanesulfonic Acid (PFNS) | ----- | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| 374BPerfluorodecanesulfonic Acid (PFDS) | ----- | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| 10:2 Fluorotelomersulfonic Acid (10:2-FTS) | ----- | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 |
| Perfluorododecanesulfonic Acid (PFDoS) | ----- | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluorohexadecanoic Acid (PFHxDA) | ----- | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 |
| Perfluorooctadecanoic Acid (PFODA) | ----- | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 |
| Perfluorooctanesulfonamide (PFOSA) | ----- | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| N-methyl perfluorooctanesulfonamidoethanol (NMeFOSE) | ----- | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| N-methyl perfluorooctanesulfonamide (NMeFOSA) | ----- | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 |

| Parameter/Analyte | Drinking Water | Nonpotable Water | Solid Haz.Waste |
|---|----------------|---|---|
| | | EPA Draft Method 1633 | EPA Draft Method 1633 |
| N-ethyl perfluorooctanesulfonamidoethanol (NEtFOSE) | ----- | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| N-ethylperfluorooctanesulfonamide (NEtFOSA) | ----- | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) | EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluoro-3-methoxypropanoic acid (PFMPA) | EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluoro-4-methoxybutanoic acid (PFMBA) | EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA) | EPA 533 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| 3-Perfluoropropylpropanoic acid (3:3 FTCA) | --- | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| 2H,2H,3H,3H-Perfluorooctanoic acid (5:3 FTCA) | --- | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |
| 3-Perfluoroheptylpropanoic acid (7:3 FTCA) | --- | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 | PFAS by LCMSMS Compliant with QSM 5.3/5.4 Table B-15 EPA Draft Method 1633 |

End of DoD ELAP section of scope

In addition, in recognition of the successful completion of the A2LA evaluation process (including an assessment of the



laboratory's compliance with ISO IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, and for the test methods applicable to Kentucky Statute KRS 224.60-130(2)(a), and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program), accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP-MS Spectrometry, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.-Electronic Probes (pH, F⁻, O₂), Oxygen Demand, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, TCLP, Total Organic Carbon, Turbidity, Liquid Chromatography/Mass Spectrometry/Mass Spectrometry, High Resolution Gas Chromatography/Mass Spectrometry

| <u>Parameter/Analyte</u> | <u>Tissue</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste</u> | |
|-----------------------------|---|--|--|---|
| | | | <u>Aqueous</u> | <u>Solid</u> |
| Other | | | | |
| Perchlorate | Food & Food Products EPA 6850 | EPA 6850 | EPA 6850 | EPA 6850 |
| Hydrazine | ----- | EPA 8315A MOD | EPA 8315A MOD | EPA 8315A MOD |
| Methylhydrazine | ----- | EPA 8315A MOD | EPA 8315A MOD | EPA 8315A MOD |
| 1,1-Dimethylhydrazine | ----- | EPA 8315A MOD | EPA 8315A MOD | EPA 8315A MOD |
| Volatile Preparation | ----- | EPA 5030A EPA 5030C | EPA 5030A EPA 5030C | EPA 5035 EPA 5035A |
| Organic Extraction/ Cleanup | EPA 3546 EPA 3550C EPA 3660B EPA 3620C EPA 3665A EPA 3640A | EPA 3510C EPA 3511 EPA 3660B EPA 3620C EPA 3665A | EPA 3510C EPA 3511 EPA 3660B EPA 3620C EPA 3665A | EPA 3546 EPA 3550C EPA 3660B EPA 3620C EPA 3665A EPA 3640A |

| <u>Parameter/Analyte</u> | <u>Tissue</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste</u> | |
|---------------------------------------|---------------|-------------------------|------------------------------|--------------|
| | | | <u>Aqueous</u> | <u>Solid</u> |
| Kentucky UST Program | | | | |
| Metals | | | | |
| Arsenic | ----- | ----- | EPA 6010B | EPA 6010B |
| Barium | ----- | ----- | EPA 6010B | EPA 6010B |
| Cadmium | ----- | ----- | EPA 6010B | EPA 6010B |
| Chromium | ----- | ----- | EPA 6010B | EPA 6010B |
| Lead | ----- | ----- | EPA 6010B | EPA 6010B |
| Mercury | ----- | ----- | EPA 7470A | EPA 7471A |
| Selenium | ----- | ----- | EPA 6010B | EPA 6010B |
| Silver | ----- | ----- | EPA 6010B | EPA 6010B |
| Purgeable Organics (Volatiles) | | | | |



| <u>Parameter/Analyte</u> | <u>Tissue</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste</u> | |
|---|---------------|-------------------------|-------------------------------------|-------------------------------------|
| | | | <u>Aqueous</u> | <u>Solid</u> |
| Diesel Range Organics (DRO) | ----- | EPA 8015C EPA 8015D | EPA 8015C EPA 8015D | EPA 8015C EPA 8015D |
| Gasoline Range Organics (GRO) | ----- | EPA 8015C EPA 8015D | EPA 8015C EPA 8015D | EPA 8015C EPA 8015D |
| Wyoming Storage Tank Program | | | | |
| Metals | | | | |
| Cadmium | ----- | ----- | EPA 6010C | EPA 6010C |
| Chromium | ----- | ----- | EPA 6010C | EPA 6010C |
| Chromium (Total, hexavalent) | ----- | ----- | EPA 7196A | EPA 7196A |
| Lead | ----- | ----- | EPA 6010C | EPA 6010C |
| Purgeable Organics (Volatiles) | | | | |
| Volatile Preparation | ----- | ----- | EPA 5030C EPA 5030C | EPA 5035 EPA 5035A |
| Benzene | ----- | ----- | EPA 5030C EPA 8260D | EPA 8260D |
| 1,2-Dichloroethane | ----- | ----- | EPA 8260D | EPA 8260D |
| 1,2-Dibromoethane | ----- | ----- | EPA 8011 | EPA 8011 |
| Diisopropyl Ether | ----- | ----- | EPA 5030C EPA 8260D | EPA 8260D |
| Ethyl Benzene | ----- | ----- | EPA 5030C EPA 8260D | EPA 8260D |
| Ethyl tert-butyl Ether | ----- | ----- | EPA 8260D | EPA 8260D |
| Methyl tert-butyl Ether | ----- | ----- | EPA 5030C EPA 8260D | EPA 8260D |
| Naphthalene | ----- | ----- | EPA 5030C EPA 8260D | EPA 8260D |
| Toluene | ----- | ----- | EPA 5030C EPA 8260D | EPA 8260D |
| Tert-amyl Methyl Ether | ----- | ----- | EPA 5030C EPA 8260D | EPA 8260D |
| Tert-butyl Alcohol | ----- | ----- | EPA 5030C EPA 8260D | EPA 8260D |
| Xylenes, total | ----- | ----- | EPA 5030C EPA 8260D | EPA 8260D |
| Gasoline Range Organics (GRO C6-C10) | ----- | ----- | EPA 5030C EPA 8260D | EPA 8260D |
| Extractable Organics (Semivolatiles) | | | | |
| Diesel Range Organics (DRO C10-C32) | ----- | ----- | EPA 8015C w/ EPA 3630 cleanup | EPA 8015C w/ EPA 3630 cleanup |

End of KY, WY, and ISO 17025 section of scope



In recognition of the successful completion of the A2LA evaluation process, including an assessment of the laboratory's compliance with ISO/IEC 17025:2017 accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and, in the analyte, categories identified below:

| | |
|--|------------------|
| Food and Feed (WHO 29) | Food/Feed |
| 2,3,7,8-TCDD | EPA 1613B |
| 2,3,7,8-TCDF | EPA 1613B |
| 1,2,3,7,8-PeCDF | EPA 1613B |
| 2,3,4,7,8-PeCDF | EPA 1613B |
| 1,2,3,7,8-PeCDD | EPA 1613B |
| 1,2,3,4,7,8-HxCDF | EPA 1613B |
| 1,2,3,6,7,8-HxCDF | EPA 1613B |
| 2,3,4,6,7,8-HxCDF | EPA 1613B |
| 1,2,3,7,8,9-HxCDF | EPA 1613B |
| 1,2,3,4,7,8-HxCDD | EPA 1613B |
| 1,2,3,6,7,8-HxCDD | EPA 1613B |
| 1,2,3,7,8,9-HxCDD | EPA 1613B |
| 1,2,3,4,6,7,8-HpCDF | EPA 1613B |
| 1,2,3,4,7,8,9-HpCDF | EPA 1613B |
| 1,2,3,4,6,7,8-HpCDD | EPA 1613B |
| OCDF | EPA 1613B |
| OCDD | EPA 1613B |
| Food and Feed (WHO 29) | Food/Feed |
| 6 marker PCBs (PCB28, PCB52, PCB101, PCB138, PCB153, and PCB180) | EPA 1668C |
| (PCB77, PCB81, PCB105, PCB114, PCB118, PCB123, PCB126, PCB156, PCB157, PCB167, PCB169, and PCB189) | EPA 1668C |

| <u>Parameter/Analyte</u> | <u>Tissue</u> | <u>Nonpotable Water</u> | <u>Solid Hazardous Waste</u> | |
|--|---------------|-------------------------|------------------------------|--------------|
| | | | <u>Aqueous</u> | <u>Solid</u> |
| 12 Dioxin-like PCBs (dl-PCBs)/coplanar PCBs (PCB77, PCB81, PCB105, PCB114, PCB118, PCB123, PCB126, PCB156, PCB157, PCB167, PCB169, & PCB189) | EPA 1668C | ----- | ----- | ----- |

| <u>Parameter/Analyte</u> | <u>Drinking Water</u> | <u>Nonpotable Water</u> | <u>Solid Haz.Waste</u> |
|--|-------------------------------|-------------------------|------------------------|
| 441BPer and Polyfluoroalkyl Substances (PFAS) | | | |
| 442BN-ethyl perfluorooctane-sulfonamidoacetic acid (NetFOSAA) | EPA 537 Ver. 1.1 EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 443BN-methyl perfluorooctane-sulfonamidoacetic acid (NMeFOSAA) | EPA 537 Ver. 1.1 EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 444BPerfluorobutanesulfonic acid (PFBS) | EPA 537 Ver. 1.1 EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 445BPerfluorodecanoic acid (PFDA) | EPA 537 Ver. 1.1 EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 446BPerfluorododecanoic acid (PFDoDA) | EPA 537 Ver. 1.1 EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 447BPerfluoroheptanoic acid (PFHpA) | EPA 537 Ver. 1.1 EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 448BPerfluorohexanesulfonic acid (PFHxS) | EPA 537 Ver. 1.1 EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 449BPerfluorohexanoic acid (PFHxA) | EPA 537 Ver. 1.1 EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 450BPerfluorononanoic acid (PFNA) | EPA 537 Ver. 1.1 EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 451BPerfluorooctanesulfonic acid (PFOS) | EPA 537 Ver. 1.1 EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 452BPerfluorooctanoic acid (PFOA) | EPA 537 Ver. 1.1 EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 453BPerfluorotetradecanoic acid (PFTeDA) | EPA 537 Ver. 1.1 EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 454BPerfluorotridecanoic acid (PFTrDA) | EPA 537 Ver. 1.1 EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 455BPerfluoroundecanoic acid (PFUnDA) | EPA 537 Ver. 1.1 EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid (HFPODA) | EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 4,8-Dioxa-3H-perfluorononanoic acid (DONA) | EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS) | EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS) | EPA 537.1 | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 456BPerfluoro-n-butanoic acid (PFBA) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 457BPerfluoro-n-pentanoic acid (PFPeA) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |

| <u>Parameter/Analyte</u> | <u>Drinking Water</u> | <u>Nonpotable Water</u> | <u>Solid Haz.Waste</u> |
|---|-----------------------|-------------------------|------------------------|
| 458B8:2 Fluorotelomersulfonic acid (8:2FTS) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 459B4:2 Fluorotelomersulfonic acid (4:2-FTS) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 460BPerfluoropentanesulfonic acid (PFPeS) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 461B6:2 Fluorotelomersulfonic acid (6:2-FTS) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| Perfluoroheptanesulfonic acid (PFHpS) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 462BPerfluorononanesulfonic acid (PFNS) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 463BPerfluorodecanesulfonic acid (PFDS) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 464B10:2 Fluorotelomersulfonic acid (10:2-FTS) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 465BPerfluorododecanesulfonic acid (PFDoDS) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 466BPerfluorohexadecanoic acid (PFHxDA) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 467BPerfluorooctadecanoic acid (PFODA) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 468BPerfluorooctanesulfonamide (PFOSA) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 469B2-(N-methylperfluoro-1-octanesulfonamido)-ethanol (NMePFOSAE) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 470BN-methylperfluoro-1-octanesulfonamide (NMePFOSA) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 471B2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol (NEtPFOSAE) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |
| 472BN-ethylperfluoro-1-octanesulfonamide (NEtPFOSA) | ----- | EPA 537 Ver.1.1 Mod | EPA 537 Ver.1.1 Mod |



Accredited Laboratory

A2LA has accredited

EUROFINS LANCASTER LABORATORIES ENVIRONMENTAL, LLC

Lancaster, PA

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.4 of the DoD/DOE Quality System Manual for Environmental Laboratories (QSM), accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).



Presented this 21st day of November 2022.

A blue ink signature of Mr. Trace McInturff, written over a horizontal line.

Mr. Trace McInturff, Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 1.01
Valid to November 30, 2024

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

| | | |
|--|---|---|
|  | Always check on-line for validity. | Level:  |
| Document number: T-PFAS-WI46412 | <p style="text-align: center;">Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples by LC-MS/MS Using Draft Method 1633/QSM5.4 Table B24</p> | Work Instruction |
| Old Reference: | | |
| Version: 2 | | Organisation level: 5-Sub-BU |
| Approved by: XL3S Effective Date 31-AUG-2022 | Document users: 5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep | Responsible: 5_EUUSLA_PFAS_Manager |

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Revision Log

| | | |
|------------------------|--------------------------|---|
| Revision: | <u>2</u> | Effective date: <u>This version</u> |
| Section | Justification | Changes |
| Revision Log | Formatting Requirement | Revision logs up to the previous version |
| Reference | Enhancement | Updated to 2 nd version of draft june 2022 |
| Cross Reference | Enhancement | Add T-WC-WI53304 |
| Reagents and Standards | Reflect current practice | Remove NH4OH 5M. this is not used B.standards-updated to standardized verbiage |
| Attachments | Enhancement | Updated attachments 5-20 for added clarity |

| | | |
|------------------|--|--|
| Revision: | <u>2</u> | Effective date: <u>This version</u> |
| Section | Justification | Changes |
| Procedure | Clarification/reflect current practice | B.6, change PFC_ST_XXXX to PFC_1633_SS_XXXX B.7 updated for clarity B.8 add volume of 5ml for 1:1 0.1M formic acid:MeOH rinse B.8 update drying instructions. C.6 updated example sequence Add verbiage about TSS |

| | | |
|------------------|----------------------|---|
| Revision: | <u>1</u> | Effective date: <u>03-JUN-2022</u> |
| Section | Justification | Changes |
| Revision Log | NEW | NEW |

Reference

1. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by LC/MS/MS (EPA Draft method 1633), Department of Defense Quality System Manual Version 5.4, Table B-24.
2. US EPA Method 1633, *Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS, Version 2nd DRAFT, June 2022.*
3. *Chemical Hygiene Plan*, current version.

Cross Reference

| Document | Document Title |
|--------------------------------|---|
| T-PFAS-WI21568 | Manifold and N-EVAP Cleaning for PFAS Extractions |
| T-PEST-WI9847 | Common Equations Used During Chromatographic Analyses |
| T-WC-WI53304 | Total Suspended Solids (TSS Gravimetric) Prescreen by EPA Draft Method 1633 Revision 2 in Aqueous Samples |
| QA-SOP11178 | Demonstrations of Capability |
| QA-SOP11892 | Determining Method Detection Limits and Limits of Quantitation |

Scope

This method is applicable for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in aqueous samples to include non-potable waters and non-regulatory potable water when directed by the client. The compounds analyzed in this method are listed in the table below. The most current MDLs and LOQs are listed in the LIMS. Compounds other than those listed may be analyzed by client request.

| Analyte | Acronym | CAS# |
|--|----------------|---------------|
| Perfluorobutanesulfonic acid | PFBS | 375-73-5 |
| Perfluorodecanoic acid | PFDA | 335-76-2 |
| Perfluorododecanoic acid | PFDODA | 307-55-1 |
| Perfluoroheptanoic acid | PFHpA | 375-85-9 |
| Perfluorohexanesulfonic acid | PFHxS | 355-46-4 |
| Perfluorohexanoic acid | PFHxA | 307-24-4 |
| Perfluorononanoic acid | PFNA | 375-95-1 |
| Perfluorooctanesulfonic acid | PFOS | 1763-23-1 |
| Perfluorooctanoic acid | PFOA | 335-67-1 |
| Perfluorotetradecanoic acid | PFTeDA | 376-06-7 |
| Perfluorotridecanoic acid | PFTTrDA | 72629-94-8 |
| Perfluoroundecanoic acid | PFUnDA | 2058-94-8 |
| Perfluoro-n-butanoic acid | PFBA | 375-22-4 |
| Perfluoro-n-pentanoic acid | PFPeA | 2706-90-3 |
| 8:2 - Fluorotelomersulfonic acid | 8:2FTS | 39108-34-4 |
| N-methylperfluoro-1-octanesulfonamidoacetic acid | NMeFOSAA | 2355-31-9 |
| N-ethylperfluoro-1-octanesulfonamidoacetic acid | NEtFOSAA | 2991-50-6 |
| 4:2-Fluorotelomersulfonic acid | 4:2-FTS | 757124-72-4 |
| Perfluoropentanesulfonic acid | PFPeS | 2706-91-4 |
| 6:2-Fluorotelomersulfonic acid | 6:2-FTS | 27619-97-2 |
| Perfluoroheptanesulfonic acid | PFHpS | 375-92-8 |
| Perfluorononanesulfonic acid | PFNS | 68259-12-1 |
| Perfluorodecanesulfonic acid | PFDS | 335-77-3 |
| Perfluorododecanesulfonic acid | PFDODS | 79780-39-5 |
| Perfluorooctanesulfonamide | PFOSA | 754-91-6 |
| 2-(N-methylperfluoro-1-octanesulfonamido)-ethanol | NMePFOSAE | 24448-09-7 |
| N-methylperfluoro-1-octanesulfonamide | NMePFOSA | 31506-32-8 |
| 2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol | NEtPFOSAE | 1691-99-2 |
| N-ethylperfluoro-1-octanesulfonamide | NEtPFOSA | 4151-50-2 |
| 2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid; (Hexafluoropropylene oxide dimer acid) | HFPODA | 13252-13-6 |
| Ammonium 4,8-dioxa-3H-perfluorononanoic acid | DONA ** | 919005-14-4 * |

| Analyte | Acronym | CAS# |
|---|--------------------------|---------------|
| Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid | 9Cl-PF3ONS, F53B major | 756426-58-1 * |
| Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid | 11Cl-PF3OUdS, F53B minor | 763051-92-9 * |
| 3-Perfluoropropylpropanoic acid | 3:3 FTCA | 356-02-5 |
| 3-Perfluoropentylpropanoic acid | 5:3 FTCA | 914637-49-3 |
| 3-Perfluoroheptylpropanoic acid | 7:3 FTCA | 812-70-4 |
| Perfluoro-3-methoxypropanoic acid | PFMPA | 377-73-1 |
| Perfluoro-4-methoxybutanoic acid | PFMBA | 863090-89-5 |
| Nonafluoro-3,6-dioxaheptanoic acid | NFDHA | 151772-58-6 |
| Perfluoro(2-ethoxyethane)sulfonic acid | PFEESA | 113507-82-7 |

*CAS# for the free acid form of the analyte

**Acronym for the free acid form of the analyte

Basic Principles

A 500-mL aqueous sample is fortified with isotopically-labeled extraction standards and is passed through a solid phase extraction (SPE) cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. Carbon cleanup is performed on each sample extract. The extract is filtered and fortified with Isotopically-labeled injection internal standards. It is then analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.

Interferences

Compounds which have similar structures to the compounds of interest and similar molecular weights would potentially interfere. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, etc. A laboratory blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Precaution to Minimize Method Interference

1. LC system components contain many of the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK™ solvent frits and tubing where possible.
2. A precolumn, Phenomenex Luna, 30 x 2 mm, 5 µm C18 column, is installed before the injection valve to separate PFAS in standards/samples from those from the LC system and mobile phases.
3. All part of the SPE manifold must be cleaned as per [T-PFAS-WI21568](#).

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. PFOA has been described as "likely to be carcinogenic to humans". Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal into the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is capable of independent preparation. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

Each LC/MS/MS analyst must work with an experienced employee for a period of time until they can independently calibrate the LC/MS/MS, review and process data, and perform maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC and DOC consist of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. IDOC trials are spiked at the OPR Level.

See [QA-SOP11178](#) for additional information on IDOC and DOC.

Sample Collection, Preservation, and Handling

A. Sample Collection

The samples are collected in 500-mL HDPE containers. The second aliquot may be collected in a smaller sample container (e.g. 250 mL or 125 mL). All sample containers must have linerless HDPE or polypropylene caps. Keep the sample sealed from time of collection until extraction.

NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

B. Sample Storage and Shipment

1. Samples must be chilled during shipment and must not exceed 6°C during the first 48 hours after collection. Sample temperature must be confirmed to be at 0° to 6°C when the samples are received at the laboratory.
2. Samples stored in the lab must be held at a temperature of 0° to 6°C, not frozen, and protected from light until extraction. Alternatively, to meet project requirements, samples may be stored at $\leq -20^{\circ}\text{C}$ and protected from light until extraction.
3. Water samples must be extracted within 28 days when stored at a temperature of 0° to 6°C, not frozen, and protected from light. Water samples must be extracted within 90 days when stored at a temperature $\leq -20^{\circ}\text{C}$ and protected from light. Extracts must be analyzed within 28 days after extraction. Extracts are stored at a temperature of 0° to 6°C.

Apparatus and Equipment

A. Apparatus

1. 500 mL HDPE bottles: Scientific Specialties; #334008-blk-1, or equivalent.
2. Centrifuge tubes – 15-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 05-539-5 or equivalent
3. 10-mL polypropylene volumetric flask, Class A – Fisher Scientific, Cat. No. S02288 or equivalent.
4. HDPE bottles for extraction fluid storage: L; Environmental Sampling Supply, Cat. No. 1000-1902-PC.
5. Analytical Balance – Capable of weighing to 0.0001 g
5. Top-Loading Balance – Capable of weighing to 0.01 g
7. Solid phase extraction (SPE) Weak Anion Exchange ("WAX") cartridge – Agilent; Sampli-Q WAX Polymer; 150mg/6mL; Cat. # 5982-3667.
3. Large-volume SPE Reservoir (25-mL) - Millipore-Sigma; Product # 54258-U.
9. SPE Tube Adapter - Millipore-Sigma; Product # 57020-U.
10. SPE vacuum extraction manifold –"Resprep" 24-port manifold; Restek Corp catalog # 26080, or equivalent.
11. Polypropylene SPE delivery needles – Agilent; Cat. No. 12234511.
12. Centrifuge – "Q-Sep 3000"; Restek Corp. Cat. No. 26230, or equivalent, capable of a minimum rotational speed of 3000 rpm.
13. Disposable polyethylene pipette – Fisher Scientific, Cat. No. S30467-1 or equivalent.
14. Auto Pipettes – Eppendorf; capable of accurately dispensing 10- to 1000- μL . FisherScientific cat # 14-287-150, or equivalent.
15. Polypropylene pipette tips: 0-200 μL . Fisher; Cat. No. 02-681-135
16. Polypropylene pipette tips: 101-1000 μL . Fisher, Cat. No. 02-707-508
17. Pipettes – Disposable transfer. FisherScientific, Cat. No. 13-711-7M

18. Vortex mixer, variable speed, Fisher Scientific or equivalent.
19. N-Evap sample extract concentrator with N₂ supply and water bath for temperature control. Organomation, Inc. Cat. #11250, or equivalent.
20. Reagent Water Purification System: Capable of producing ultrapure "Type 1/Milli-Q"-grade water from in-house deionized water system. Millipore SAS; Cat. No. FTF08831.
21. Thermo Target PP Polyspring inserts, catalog number C4010-630P
22. Agilent 9mm vial kit pack, catalog number 5190-2278, or equivalent
23. Centrifuge tubes – 50-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 06-443-21 or equivalent
24. Polypropylene bottles for standard storage - 4 mL; Fisher Scientific, Cat. No. 2006-9125
25. Stainless steel spatula/scoop set. Bel-Art SP Scienceware; Product # 11-865-130.
26. pH paper, range 0-14, Whatman Panpeha or equivalent, 0.5 unit readability
27. Syringe filter - Acrodisc, Syringe Filter, GHP, 13 mm, 0.2 µm, Aqueous, 100/pkg, Part # WAT097962.
28. Silanized glass wool (Sigma-Alrich, Cat #20411 or equivalent
29. Disposable syringe filter, 25-mm, 0.2µm Nylon membrane, PALL/Acrodisc or equivalent
30. Glass fiber filter, 47 mm, 1 µm, PALL A/E or equivalent

B. Equipment

1. AB Sciex Triple Quad 4500/5500/5500 Plus Turbo V Ion Source

ExionLC Controller
ExionLC AC Pump
ExionLC AC Autosampler
Exion AC Column Oven
Data system –Analyst 1.6.3

2. HPLC columns

- a. Analytical column: Gemini 3µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4439-YO or equivalent
- b. Pre-column: Luna, 5µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4252-Y0, or equivalent

Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions.

A. Reagents:

1. Methanol (MeOH) – Honeywell Burdick and Jackson "Chromasolv LC-MS" grade Cat. No. BJ34966-4L or equivalent
2. Acetonitrile (ACN) – Fisher Scientific, Optima Cat. No. A955-4 or equivalent

3. Ammonium acetate – Fisher Scientific, Cat. No. A637-500 or equivalent
4. Ammonium hydroxide, 30% in water, certified ACS+ grade or equivalent, store at room temperature
5. Aqueous ammonium hydroxide (3%) – add ammonium hydroxide (10 mL, 30%) to reagent water (90 mL), store at room temperature, replace after 3 months
6. Methanolic ammonium hydroxide (1%) - add ammonium hydroxide (3.3 mL, 30%) to methanol (97 mL), store at room temperature, replace after 1 month
7. Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid - add ammonium hydroxide (3.3 mL, 30%), reagent water (1.7 mL) and acetic acid (0.625 mL) to methanol (92 mL), store at room temperature, replace after 1 month.
8. Acetic Acid – ACS grade or equivalent, store at room temperature
9. Acetic Acid (0.1%) – dissolve acetic acid (1 mL) in reagent water (1 L), store at room temperature, replace after 3 months.
10. Formic acid
 - a. Formic acid (aqueous, 0.1 M) - dissolve formic acid (4.6 g) in reagent water (1 L), store at room temperature, replace after 2 years
 - b. Formic acid (aqueous, 0.3 M) - dissolve formic acid (13.8 g) in reagent water (1 L), store at room temperature, replace after 2 years
 - c. Formic acid (aqueous, 5% v/v) - mix 5 mL formic acid with 95 mL reagent water, store at room temperature, replace after 2 years
 - d. Formic acid (aqueous, 50% v/v) - mix 50 mL formic acid with 50 mL reagent water, store at room temperature, replace after 2 years
 - e. Formic acid (methanolic 1:1, 0.1 M formic acid/methanol) - mix equal volumes of methanol and 0.1 M formic acid, store at room temperature, replace after 2 years
11. "Superclean Envi-Carb"; bulk sorbent. Millipore-Sigma; 50g; Product # 57210-U.
12. 20 mM ammonium acetate solution in 95:5(v/v) Milli-Q water/acetonitrile-Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 1900 mL Milli-Q water and mix well to dissolve the ammonium acetate. Add 100 mL acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.
13. 20 mM ammonium acetate solution in 90:10 acetonitrile/Milli-Q water – Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 200 mL of Milli-Q water and mix well to dissolve the Ammonium Acetate. Add 1800 mL of acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.

B. Standards:

Standards are prepared using calibrated pipettes, polypropylene microcentrifuge tubes, polypropylene bottles, and 10 ml Class A PP volumetric flasks to create solutions at desired concentrations. The concentrated solution is injected below the surface of the diluting solvent. After preparation is completed, standards should be vortexed to ensure complete mixing. Measurement of volumes less than 5 µl should be avoided in routine production operations.

All stock, intermediate and spiking solutions are prepared using Methanol.

All initial calibration, initial calibration verification, and linear branched working standard solutions are prepared using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid.

All diluted solutions must be stored in HDPE containers that have been thoroughly rinsed with methanol.

Stock standard and intermediate standard solutions are stored in the refrigerator in labeled polypropylene screw-top vials, PP bottles, or PP centrifuge tubes.

Expiration dates are managed through LIMS Reagent. Solutions transferred from sealed glass ampules to screw-capped vials are given expiration dates of 1 year from the date opened or the expiration date provided by the vendor, whichever occurs sooner. Intermediate solutions are given an expiration date of 6 months from the preparation date, or the expiration date from the ampule provided by the vendor, whichever occurs sooner. The ampules and transferred solutions are stored in the refrigerator.

Working native and labeled (extraction surrogate and internal standard) compound spiking solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working solution, whichever occurs sooner. The solutions are stored in labeled polypropylene screw-top vials in the refrigerator. When these solutions are prepared they must be tested prior to use in the PFAS extraction lab and verified monthly until they are consumed by operations or expire. Records of the standard verification are maintained by the laboratory. Prior to use, the working spiking solution should be evaluated against recovery windows of 85-115% for all compounds that will be analyzed using that solution. Should a standard fail to meet these criteria, the data must be reviewed by departmental management for acceptability and/or corrective action.

Working initial calibration solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working initial calibration solution, whichever occurs sooner.

The primary/preferred standard vendor is Wellington Laboratories, Inc. Ontario, Canada. Listed catalog numbers are taken from Wellington product lists. Equivalent standards may be substituted, if the listed standards are unavailable.

The solution concentration listed is as presented on the certificate of analysis and includes adjustment for purity and the salt form of the compound used.

Note: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of analysis (CofA). See [Attachment 4](#).

If the compound purity is assayed to be 96% or greater, weight can be used without correction to calculate concentrations.

Log purchased standards into LIMS Reagent. Select the solution category SOURCE for purchased mixes and/or single-compound ampules. LIMS Reagent system will assign formatted names to the purchased standard solutions. The automatically-generated name can be overwritten with a manually created name if desired. Use labels printed through the LIMS Reagent to identify and track standard solutions after transfer from original ampule to storage vial. The CofA for the ampulated stock standard is attached in LIMS Reagent for reference.

Standards are prepared by transferring a known quantity of Standard to a final volume of solvent. Standard Preparation is documented in LIMS Reagent. Solutions are stored by Type in LIMS Reagent, i.e., INTERMEDIATE=working solutions and intermediate standards and SOURCE=stocks (ampulated solutions). Each Standard is given a unique name.

The following attachments provide examples of standard preparation and purchasing information. Refer to the documentation in LIMS Reagent for standards preparation information.

Attachment 5 - Native PFAS Intermediate A
Attachment 6 - Native PFAS Intermediate B
Attachment 7 - Working Labeled Extraction Standard Spike
Attachment 8 - Internal Standard Spike
[Attachment 9](#) - Native 1633 Mid-Level Spike
[Attachment 10](#) - Native 1633 Low-Level Spike
[Attachment 11](#) - 1633 Initial Calibration Standards Preparation
[Attachment 12](#) - 1633 Initial calibration Standards Concentrations
[Attachment 13](#) - TDCA Stock Solution
[Attachment 14](#) - TDCA Working Solution A
[Attachment 15](#) - TDCA Working Solution B
[Attachment 16](#) - 1633 Linear/Branched TDCA Intermediate
[Attachment 17](#) - 1633 Linear/Branched TDCA Solution
[Attachment 18](#) - PFAS 1633 ICV Working Standard
[Attachment 19](#) - 1633 Labeled Ampulated Standards
[Attachment 20](#) - 1633 Native Ampulated Standards

Calibration

A. Initial Calibration

1. A minimum of six calibration standards are required when using an average or linear curve fit. A minimum of seven calibration standards are required for a second-order curve fit. In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, and NEtFOSAA. S/N ratio must be greater than or equal to 3:1 for all ions used for quantification.
2. Analyze a Cal4 level standard that contains TDCA retention time marker and linear and branch chained isomers of PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE. The analysis of this standard is used to evaluate the interference from bile salts in tissue samples, as well as evaluate where the branch chained isomers elute and not included in the calibration curve. This will assist the chemist in identifying and properly integrating this compound in samples.

Example Initial Calibration Sequence:

1. Instrument Blank
 2. Instrument Blank
 3. Instrument Blank
 4. CAL 1
 5. CAL 2
 6. CAL 3
 7. CAL 4
 8. CAL 5
 9. CAL 6
 10. CAL 7
 11. ICB (Instrument Blank)
 12. ICV
 13. MDL
 14. WDM (Linear Branched/TDCA standard)
3. Isotopically-labeled compounds are not available for some compounds. See below for compounds and their referenced extraction standards. See [Attachment 2](#) for additional information about compound relationships.
 4. Analyze a standard at a concentration of 100 ppb containing Taurodeoxycholic Acid (TDCA). The analysis of this standard is used to evaluate the chromatographic program relative to the risk of an interference from bile salts in tissue samples. The analytical conditions must be set to allow a separation of at least 1 minute between the bile salts and PFOS.

NOTE: For better accuracy, PFTrDA is quantitated using the average of the areas of labeled compounds 13C2-PFTeDA and 13C2-PFDoDA.

| Compound | Extraction Standard |
|----------|--------------------------------|
| PFBA | 13C4-PFBA |
| PFPeA | 13C5-PFPeA |
| 3:3FTCA | |
| PFMPA | |
| PFMBA | |
| PFHxA | 13C5-PFHxA |
| NFDHA | |
| 5:3FTCA | |
| 7:3FTCA | |
| PFEESA | |
| PFHpA | 13C4-PFHpA |
| PFOA | 13C8-PFOA |
| PFNA | 13C9-PFNA |
| PFDA | 13C6-PFDA |
| PFUnA | 13C7-PFUnA |
| PFDoA | 13C2-PFDoA |
| PFTrDA | Avg 13C2-PFTeDA and 13C2-PFDoA |
| PFTeDA | 13C2-PFTeDA |
| PFBS | 13C3-PFBS |
| PFPeS | 13C3-PFHxS |
| PFHxS | |
| PFHpS | 13C8-PFOS |
| PFOS | |
| PFNS | |
| PFDS | |
| PFDoS | |
| 4:2-FTS | 13C2-4:2-FTS |
| 6:2-FTS | 13C2-6:2-FTS |
| 8:2-FTS | 13C2-8:2-FTS |
| PFOSA | 13C8-PFOSA |

| | |
|--------------|--------------|
| NMeFOSA | D3-NMeFOSA |
| NEtFOSA | D5-NEtFOSA |
| NMeFOSAA | D3-NMeFOSAA |
| NEtFOSAA | D5-N-EtFOSAA |
| NMeFOSE | D7-NMeFOSE |
| NEtFOSE | D9-NEtFOSE |
| HFPO-DA | 13C3-HFPO-DA |
| DONA | |
| 9Cl-PF3ONS | |
| 11Cl-PF3OUdS | |

5. Fit the curve

- If the %RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data.
- If the %RSD is greater than 20%, a linear regression with a concentration weighing factor of 1/x is tried for the compounds not meeting the criteria in 5.a. The RSE for all method analytes must be less than or equal to 20%.
- For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point should be within $\pm 30\%$ of its true value.
- If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

NOTE: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of Analysis (CofA). See [Attachment 4](#).

6. Initial Calibration Verification (ICV)

A check standard prepared from a second source (ICV) is injected to confirm the validity of the calibration curve/standard. If a second source is not available, a separate preparation from the same stock by a second analyst may be used. The calculated amount for each analyte must be within $\pm 30\%$ of the true value. If this criteria is not met, re-inject or remake the standard. If the criteria is still not met, recalibration is necessary. Instrument maintenance may be needed prior to recalibrating.

B. Continuing calibration

- Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Subsequent CCV standards should use the Cal4 level standard.
- Acceptance criteria
 - The calculated amount for each compound (native and extraction standard) in the CCV standard must be within $\pm 30\%$ of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target

analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.

- b. The absolute areas of the injection internal standards should be greater than 30% of the average areas measured during the initial calibration.

Procedure

All water samples should be evaluated for Total Suspended Solids(TSS) as per T-WC-WI53304. Although the full container volume of sample should be extracted, this method is applicable to aqueous samples containing up to 100mg/L of suspended solids. For samples containing > 100mg/l of suspended solids, or when unavoidable due to high levels of PFAS, smaller samples volumes may be analyzed. However, ultimately, the TSS results, visual inspection, and analyst experience are all used to determine the volume to be used to extract the sample.

A. Sample Preparation

1. Weigh sample container with contents on a calibrated top loading balance, and record the first reading in the automated prep entry system.
 - a. For all samples, the full bottle must be extracted. The sample must remain in the original sample container until after spiking solutions have been added.
 - b. If limited sample is submitted, spike sample in original container, then add Milli-Q water to bring to final volume of 500 mL prior to SPE extraction (see B.6 for spiking details).
2. Use a 500 mL HDPE bottle for the method blank, the laboratory control sample (LCS), and the low level laboratory control sample (LLCS). Fill each bottle with 500 mL of Milli-Q water. Record 500 mL as the volume for the batch QC samples on the batchlog.
3. Check that the pH is 6.5 ± 0.5 . If necessary, adjust the pH with 50% formic acid or ammonium hydroxide (or with 5% formic acid and 3% aqueous ammonium hydroxide).

B. Solid Phase Extraction (SPE)

1. Pack clean silanized glass wool to half the height of the WAX SPE cartridge barrel.
2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.
3. Condition each SPE cartridge with the following reagents in the following order without allowing the cartridges to go dry:
 - a. 15 mL 1% methanolic ammonium hydroxide
 - b. 5 mL 0.3M formic acid
 - c. Discard conditioning eluent(s)
4. Label each sample bottle, cap and reservoir with the same number to ensure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).
5. Vortex all spike solutions prior to use.
6. Spike QC and all samples with 25 μ l of Working labeled extraction standard spike solution (PFC_1633_SS_XXXXX). Spike LCS/MS/MSD with 200 μ l of native 1633 mid-level spike solution (PFC_1633_MID_XXXXX). Spike LLCS with 400 μ l of native 1633 low-level spike solution (PFC_1633_LOW_XXXXX). Vortex/Shake containers to mix thoroughly.
7. Attach a 25-mL SPE reservoir to each cartridge. Load the QC and samples to their respective cartridges.

8. Rinse the walls of the reservoir with 5mL reagent water (twice) followed by 5 mL 1:1 0.1M formic acid/methanol and pass the rinses through the cartridge using vacuum. Apply full vacuum (not exceeding 20" Hg) to the cartridges and dry until they are visually similar to an unused cartridge. This may take up to 15 minutes for some cartridges. Discard the rinse solution.
9. Place labeled 15-mL polypropylene centrifuge collection tubes under each respective SPE cartridge ensuring the delivery needles to do not touch the sides of the tubes.
10. Rinse the inside of each empty sample/QC bottle with 5mL of 1% methanolic ammonium hydroxide.
11. Using a glass pipette, transfer the rinse from the bottles to the SPE reservoirs, washing the walls of the reservoirs. Set empty bottles aside to air dry.
12. Apply a slight vacuum to the manifold in order to reclaim as much solvent as possible from the SPE cartridges.
13. Disconnect the cartridge/adaptor from the manifold. Remove the collection tubes.
14. Add 25 uL of concentrated acetic acid to each collection tube and vortex to mix.
15. Place each empty sample bottle on the top-loading balance and weigh. Record the second reading in the automated prep entry system. The prep entry system will calculate the sample weight. Record the calculated weight as the sample volume on the batchlog.

Note: The instrument lab chemist performs the next steps.

16. Add 10 mg of Superclean Envi-Carb to each sample and batch QC extracts using a 10 mg scoop.
17. Handshake occasionally for no more than 5 minutes. Immediately vortex and centrifuge for 10 minutes.
18. Add 25 uL of Internal Standard Spike Solution (PFC_ST_XXXXX) to a clean 15-mL polypropylene centrifuge collection tube.
19. Place a syringe filter (25-mm filter, 0.2-um nylon membrane) on a 5 mL polypropylene syringe. Take the plunger out and carefully decant the sample supernatant into the syringe barrel. Replace the plunger and filter the entire extract into the new collection tube containing the internal standard.
20. QS each sample extract using methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.
21. Cap and vortex to mix.
22. Transfer a portion of the final extract to the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.
23. Cap the centrifuge tube. The remaining centrifuged extracts are stored in the refrigerator for dilution or reinjection if needed.

C. LC/MS/MS Analysis

1. Mass Calibration and Tuning

- a. At instrument set up and installation, after the performance of major maintenance, or annually calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.

- b. When masses fall outside of the ± 0.5 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.5 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.
2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (4500/5500/5500 Plus) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.
3. Acquisition method: See [Attachment 3](#). Mass Transitions: See [Attachment 1](#).
4. Instrument Sensitivity Check (ISC) and Instrument Blanks
 - a. Prior to sample analysis, an instrument sensitivity check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the LOQ. The CAL1 standard will be analyzed. All analyte concentrations must be within $\pm 30\%$ of their true values for 90% of the native and isotopically labeled compounds, with the other recoveries achieving 50-150%. The signal-to-noise ratio must be greater than or equal to 3:1. If the criteria is not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.
 - b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily or at the start of a sequence. The concentration of all analytes must be less than or equal to 1/2 the LOQ. If acceptance criteria are not met the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.
5. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.
6. After the initial calibration and when analyzing samples within the same tune, inject an instrument blank, followed by the ICV, Linear branched (L/B) standard, instrument sensitivity check, CCV standard using the CAL4, qualitative identification standard (includes TDCA RT marker), Instrument blank, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard at the CAL4 level, followed by an instrument blank.

Example Sample Sequence:

1. Instrument blank
 2. Instrument blank
 3. Instrument blank
 4. Instrument Sensitivity Check (CCV0 _CAL1)
 5. CCV 2_CAL4
 6. Linear Branched/TDCA marker (WDM)
 7. Instrument Blank (ICB)
 8. Method Blank (MB)
 9. Low Level LCS (LLCS)
 10. LCS
 11. Sample (10 or less)
 12. CCV 3_CAL4
 13. Instrument Blank
7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

8. Quantitate results for the extraction blank. No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank for acceptable batch results. If this criteria is not met, the samples must be re-extracted.
9. Calculate the recoveries of spiked analytes for the LLCS, LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.
 - a. LLCS, LCS, MS, extraction standard recoveries and RPDs are calculated and compared to the limits stored on the LIMS.
 - b. If LLCS and LCS recoveries are acceptable, proceed to sample quantitation.
 - c. If the LCS and LLCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, the samples associated with the LCS must be reextracted.
 - d. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.
10. Isotopically-labeled extraction standards are added to all samples, extraction blank, LLCS/LCS, and MS/MSD prior to extraction. The recovery of the extraction standards should be within QC acceptance criteria. If the extraction standard recovery(ies) is(are) outside the QC limit(s), reextract using a reduced sample volume. If the extraction standard recovery(ies) is(are) again outside the QC limit(s), consult a supervisor to determine the appropriate course of action based on batch and sample results.
11. Isotopically-labeled injection standards are added to each QC and field sample extract prior to analysis. The absolute areas of the injection standards should be within 30-200% of the average areas measured during the initial calibration. If the internal standards are recovered outside 30-200%, consult a supervisor to determine the appropriate course of action based on batch and sample results.
12. Compare the retention times of all of the analytes, surrogates, and internal standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than
 - a. 0.4 minutes for isotopically-labeled compounds
 - b. 0.1 minutes from their analog for native compounds with an exact isotopically-labeled compound
 - c. 0.4 minutes from their assigned analog for native compounds without an exact isotopically-labeled compound.

If the retention time is outside of the criteria, the compound is considered a false positive unless it is a compound with branched isomers. Compounds with branched isomers can vary in intensity of the individual isomers that are used for reporting and must be reviewed and compared to the preceding CCV to determine if it should be reported.

13. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of 13C4-PFBA, 13C5-PFPeA, 13C4-PFHpA, 13C8-PFOA, 13C9-PFNA, 13C6-PFDA, 13C7-PFUnA, 13C2-PFDA, 13C2-PFDoDA, 13C2-PFTeDA, 13C8-PFOSA, D3-NMePFOSA, D5-NEtFOSAA, D3-NMeFOSAA, D5-NEtPFOSA, D7-NMePFOSAE, D9-NEtPFOSAE, 13C3-PFBA, 13C4-PFOA, 13C5-PFNA, 13C2-PFOA, 18O2-PFHxS, PFBA, PFECA F(PFMPA), PFECA A(PFMBAA), NMePFOSAE, and NEtPFOSAE. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards. When an ion ratio for a compound differs from the expected ion ratio by more than 50%, a qualifier is placed on the raw data and on the sample report. No corrective action is required.
14. The linear/branch chain standard is used when assessing the correctness of the computer generated peak integrations for PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE.

15. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract with Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution and adjust the amount of internal standard spike solution in the diluted extract. Select the dilution so that the expected EIS recoveries in the diluted extract are >5%. Extracts requiring dilutions greater than 10X should be reextracted using a reduced aliquot.

Dilution Example 1/10: Mix 895 µl of Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution with 100 µl of sample extract and 5 µl of internal standard spike solution. Vortex to mix. Using an auto-pipette, transfer an aliquot of the mixed solution into a labeled auto-sampler vial. Cap and vortex thoroughly to mix.

Calculations

1. Peak Area Ratio

$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

2. On-Column Analyte Concentration using average RRF

$$\text{On-column Concentration} = \text{peak area ratio} \div \text{AVE RRF}$$

3. On-Column Analyte Concentration using linear curve

$$\text{On-column Concentration} = (\text{peak area ratio} - \text{intercept}) \div \text{slope}$$

4. Sample Concentration

$$\text{Sample concentration (ng/l)} = (\text{On-column concentration} \times \text{Final Sample Volume} \times \text{DF}) \div \text{Initial Sample Volume}$$

5. Ion Ratio

$$\text{ion ratio} = (\text{peak area or height of quantifier}) / (\text{peak area or height of qualifier})$$

5. See [T-PEST-WI9847](#) for additional calculations used to evaluate the calibrations and quality control samples.

Statistical Information/Method Performance

The LCS should contain all compounds of interest. LCS, MS, and extraction standard recoveries are compared to the limits stored on the LIMS. These limits are statistically derived when sufficient data points are available. If sufficient data points are not available to generate statistical windows advisory limits will be used.

| QC parameter | Lower acceptance limit | High acceptance limit |
|-----------------------------------|------------------------|-----------------------|
| Extracted Internal standard (EIS) | 20% | 150% |

| | | |
|---------------------------------------|--|------|
| Non-extracted Internal Standard (NIS) | >30% of the average NIS from the initial calibration | 200% |
| Analyte recoveries LCS/LLCS/MS/MSD | 40% | 150% |

Note: lower acceptance limit for EIS cannot not be <20%, lower acceptance limit for analyte recovery cannot be <40%.

Historical data for MS/Ds, LCSs, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to [QA-SOP11892](#) for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

Quality Assurance/Quality Control

For each batch of samples extracted, a method blank and an LCS/LLCS (Milli Q water spiked with all compounds to be determined carried through the entire procedure) must be extracted and analyzed. MS/MSD is extracted only if submitted by the client. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared.

If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

Attachment:

- [Attachment 1 - Mass Transitions \(.doc\)](#)
- [Attachment 10 - Native Low Level Spike \(.pdf\)](#)
- [Attachment 11 - 1633 Initial Calibration Standards Preparation \(.pdf\)](#)
- [Attachment 12 - 1633 Initial Calibration Standard Concentrations \(.pdf\)](#)
- [Attachment 13 - TDCA Stock Solution \(.pdf\)](#)
- [Attachment 14 - TDCA Working Solution A \(.pdf\)](#)
- [Attachment 15 - TDCA Working Solution B \(.pdf\)](#)
- [Attachment 16 - 1633 Linear Branched and TDCA Intermediate \(.pdf\)](#)
- [Attachment 17 - 1633 Linear Branched and TDCA Solution \(.pdf\)](#)
- [Attachment 18 - PFAS ICV Working Standard \(.pdf\)](#)
- [Attachment 19 - 1633 Labeled Ampulated Standards \(.pdf\)](#)
- [Attachment 2 - Standard Relationships \(.docx\)](#)
- [Attachment 20 - 1633 Native Ampulated Standards \(.pdf\)](#)
- [Attachment 3 - Acquisition Parameters \(.pdf\)](#)
- [Attachment 4 - Example Certificate of Analysis \(.pdf\)](#)
- [Attachment 5 - 1633 Native PFAS Intermediate A \(.pdf\)](#)
- [Attachment 6 - 1633 Native PFAS Intermediate B \(.pdf\)](#)
- [Attachment 7 - Working Labeled Extraction Standard Spike \(.pdf\)](#)
- [Attachment 8 - Internal Standard Spike \(.pdf\)](#)
- [Attachment 9 - Native Mid Level Spike \(.pdf\)](#)

[QA-SOP11178 Demonstrations of Capability](#)

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)

[T-PEST-WI9847 Common Equations Used During Chromatographic Analyses](#)

[T-PFAS-WI21568 Manifold and N-EVAP Cleaning for PFAS Extractions](#)

[Attachment: Attachment 1 - Mass Transitions \(doc\)](#)

[Attachment: Attachment 10 - Native Low Level Spike \(pdf\)](#)

[Attachment: Attachment 11 - 1633 Initial Calibration Standards Preparation \(pdf\)](#)

[Attachment: Attachment 12 - 1633 Initial Calibration Standard Concentrations \(pdf\)](#)

[Attachment: Attachment 13 - TDCA Stock Solution \(pdf\)](#)

[Attachment: Attachment 14 - TDCA Working Solution A \(pdf\)](#)

[Attachment: Attachment 15 - TDCA Working Solution B \(pdf\)](#)

[Attachment: Attachment 16 - 1633 Linear Branched and TDCA Intermediate \(pdf\)](#)

Attachment: [Attachment 17 - 1633 Linear Branched and TDCA Solution \(pdf\)](#)

Attachment: [Attachment 18 - PFAS ICV Working Standard \(pdf\)](#)

Attachment: [Attachment 19 - 1633 Labeled Ampulated Standards \(pdf\)](#)

Attachment: [Attachment 2 - Standard Relationships \(docx\)](#)

Attachment: [Attachment 20 - 1633 Native Ampulated Standards \(pdf\)](#)

Attachment: [Attachment 3 - Acquisition Parameters \(pdf\)](#)

Attachment: [Attachment 4 - Example Certificate of Analysis \(pdf\)](#)

Attachment: [Attachment 9 - Native Mid Level Spike \(pdf\)](#)

End of document

Version history

| Version | Approval | Revision information | |
|---------|-------------|----------------------|--|
| 1 | 20.MAY.2022 | | |
| 2 | 31.AUG.2022 | | |

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|  | Always check on-line for validity. | Level:  | |
| | Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Solid Samples by LC-MS/MS Using Draft Method 1633/QSM5.4 Table B24 | Work Instruction | |
| | | Document number: T-PFAS-WI48593 | Organisation level: 5-Sub-BU |
| | | Old Reference: | Responsible: 5_EUUSLA_PFAS_Manager |
| Version: 2 | Document users: 5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep | | |
| Approved by: X6TJ Effective Date: 05-OCT-2022 | | | |

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- [Revision Log](#)
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- [Personnel Training and Qualifications](#)
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- [Procedure](#)
- [Calculations](#)
- [Statistical Information/Method Performance](#)
- [Quality Assurance/Quality Control](#)

Revision Log

| Revision: | <u>2</u> | Effective date: | <u>This version</u> |
|-------------------------|--------------------------|---|---------------------|
| Section | Justification | Changes | |
| Revision Log | Formatting Requirement | Revision logs up to the previous version | |
| Reference | Enhancement | Updated to 2 nd version of draft june 2022 | |
| Apparatus and Equipment | Enhancement | Add 15ml bottles used to store standards | |
| Reagents and Standards | Reflect current practice | Remove NH4OH 5M. this is not used. reagent 5. Updated to 0.3% solution in Methanol B.standards-updated to standarized verbiage | |

| | | |
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| | Version: 2 | Organisation level: 5-Sub-BU |
| Approved by: X6TJ Effective Date: 05-OCT-2022 | Document users: 5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep | Responsible: 5_EUUSLA_PFAS_Manager |

| | | |
|------------------|--|---|
| Revision: | <u>2</u> | Effective date: <u>This version</u> |
| Section | Justification | Changes |
| Attachments | Enhancement | Updated attachments 5-20 for added clarity |
| Procedure | Clarification/reflect current practice | A: updated for clarity as needed B.8 add volume of 5ml for 1:1 0.1M formic acid:MeOH rinse C.6 updated example sequence B.16 Replace QC with bring to final volume 5ml |

| | | |
|------------------|----------------------|---|
| Revision: | <u>1</u> | Effective date: <u>26-MAY-2022</u> |
| Section | Justification | Changes |
| Revision Log | NEW | NEW |

Reference

1. Per- and Polyfluoroalkyl Substances (PFAS) Analysis by LC/MS/MS (EPA Draft method 1633), Department of Defense Quality System Manual Version 5.4, Table B-24.
2. US EPA Method 1633, *Analysis of Per and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS*, Version 2nd DRAFT, June 2022.
3. *Chemical Hygiene Plan*, current version.

Cross Reference

| Document | Document Title |
|--------------------------------|--|
| T-PFAS-WI21568 | Manifold and N-EVAP Cleaning for PFAS Extractions |
| T-PEST-WI9847 | Common Equations Used During Chromatographic Analyses |
| QA-SOP11178 | Demonstrations of Capability |
| QA-SOP11892 | Determining Method Detection Limits and Limits of Quantitation |

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| Approved by: X6TJ Effective Date: 05-OCT-2022 | | | |

Scope

This method is applicable for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in solid samples. The compounds analyzed in this method are listed in the table below. The most current MDLs and LOQs are listed in the LIMS. Compounds other than those listed may be analyzed by client request.

| Analyte | Acronym | CAS# |
|--|----------|-------------|
| Perfluorobutanesulfonic acid | PFBS | 375-73-5 |
| Perfluorodecanoic acid | PFDA | 335-76-2 |
| Perfluorododecanoic acid | PFDoDA | 307-55-1 |
| Perfluoroheptanoic acid | PFHpA | 375-85-9 |
| Perfluorohexanesulfonic acid | PFHxS | 355-46-4 |
| Perfluorohexanoic acid | PFHxA | 307-24-4 |
| Perfluorononanoic acid | PFNA | 375-95-1 |
| Perfluorooctanesulfonic acid | PFOS | 1763-23-1 |
| Perfluorooctanoic acid | PFOA | 335-67-1 |
| Perfluorotetradecanoic acid | PFTeDA | 376-06-7 |
| Perfluorotridecanoic acid | PFTrDA | 72629-94-8 |
| Perfluoroundecanoic acid | PFUnDA | 2058-94-8 |
| Perfluoro-n-butanoic acid | PFBA | 375-22-4 |
| Perfluoro-n-pentanoic acid | PFPeA | 2706-90-3 |
| 8:2 - Fluorotelomersulfonic acid | 8:2FTS | 39108-34-4 |
| N-methylperfluoro-1-octanesulfonamidoacetic acid | NMeFOSAA | 2355-31-9 |
| N-ethylperfluoro-1-octanesulfonamidoacetic acid | NEtFOSAA | 2991-50-6 |
| 4:2-Fluorotelomersulfonic acid | 4:2-FTS | 757124-72-4 |
| Perfluoropentanesulfonic acid | PFPeS | 2706-91-4 |
| 6:2-Fluorotelomersulfonic acid | 6:2-FTS | 27619-97-2 |

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| Approved by: X6TJ Effective Date: 05-OCT-2022 | | | |

| Analyte | Acronym | CAS# |
|---|--------------------------|---------------|
| Perfluoroheptanesulfonic acid | PFHpS | 375-92-8 |
| Perfluorononanesulfonic acid | PFNS | 68259-12-1 |
| Perfluorodecanesulfonic acid | PFDS | 335-77-3 |
| Perfluorododecanesulfonic acid | PFDoDS | 79780-39-5 |
| Perfluorooctanesulfonamide | PFOSA | 754-91-6 |
| 2-(N-methylperfluoro-1-octanesulfonamido)-ethanol | NMePFOSAE | 24448-09-7 |
| N-methylperfluoro-1-octanesulfonamide | NMePFOSA | 31506-32-8 |
| 2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol | NEtPFOSAE | 1691-99-2 |
| N-ethylperfluoro-1-octanesulfonamide | NEtPFOSA | 4151-50-2 |
| 2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid; (Hexafluoropropylene oxide dimer acid) | HFPODA | 13252-13-6 |
| Ammonium 4,8-dioxa-3H-perfluorononanoic acid | DONA ** | 919005-14-4 * |
| Potassium 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid | 9Cl-PF3ONS, F53B major | 756426-58-1 * |
| Potassium 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid | 11Cl-PF3OUdS, F53B minor | 763051-92-9 * |
| 3-Perfluoropropylpropanoic acid | 3:3 FTCA | 356-02-5 |
| 3-Perfluoropentylpropanoic acid | 5:3 FTCA | 914637-49-3 |
| 3-Perfluoroheptylpropanoic acid | 7:3 FTCA | 812-70-4 |
| Perfluoro-3-methoxypropanoic acid | PFMPA | 377-73-1 |
| Perfluoro-4-methoxybutanoic acid | PFMBA | 863090-89-5 |
| Nonafluoro-3,6-dioxaheptanoic acid | NFDHA | 151772-58-6 |
| Perfluoro(2-ethoxyethane)sulfonic acid | PFEESA | 113507-82-7 |

*CAS# for the free acid form of the analyte

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**Acronym for the free acid form of the analyte

Basic Principles

A solid sample is fortified with isotopically-labeled extraction standards. The sample extract is shaken, centrifuged, and the supernatant decanted. Carbon cleanup is performed on each sample extract. Sample extract is diluted to volume and then concentrated. The sample is then passed through a solid phase extraction (SPE) cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. The extract is fortified with Isotopically-labeled injection internal standards and filtered. It is then analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.

Interferences

Compounds which have similar structures to the compounds of interest and similar molecular weights would potentially interfere. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, etc. A laboratory blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Precaution to Minimize Method Interference

1. LC system components contain many of the target analytes. To minimize the background PFAS peaks, PTFE solvent frits and tubing are replaced by PEEK™ solvent frits and tubing where possible.
2. A precolumn, Phenomenex Luna, 30 x 2 mm, 5 µm C18 column, is installed before the injection valve to separate PFAS in standards/samples from those from the LC system and mobile phases.
3. All parts of the SPE manifold must be cleaned as per [T-PFAS-WI21568](#).

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

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The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. PFOA has been described as "likely to be carcinogenic to humans". Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal into the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is capable of independent preparation. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

Each LC/MS/MS analyst must work with an experienced employee for a period of time until they can independently calibrate the LC/MS/MS, review and process data, and perform maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC and DOC consist of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. IDOC trials are spiked at the OPR Level.

See [QA-SOP11178](#) for additional information on IDOC and DOC.

Sample Collection, Preservation, and Handling

A. Sample Collection

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The samples are collected in 500 mL HDPE widemouth sample bottle or jar with linerless HDPE or polypropylene caps. Collect samples as grab samples using wide-mouth jar and fill no more than $\frac{3}{4}$ full. Keep the sample sealed from time of collection until extraction.

NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

B. Sample Storage and Shipment

1. Solid and Biosolid samples must be chilled during shipment and must not exceed 6°C during the first 48 hours after collection. Sample temperature must be confirmed to be at 0° to 6°C when the samples are received at the laboratory.
2. Solid and Biosolid Samples stored in the lab must protected from light and held at a temperature of 0° to 6°C, or \leq -20°C until extraction.
3. Solid and Biosolid samples must be extracted within 90 days. Extracts must be analyzed within 28 days after extraction. Extracts are stored at a temperature of 0° to 6°C.

Note: Biosolid samples stored under refrigeration may produce gases that may cause sample to be expelled from the container when opened. This may produce noxious odors. It is recommended to store frozen if extraction will not occur for a few days.

Apparatus and Equipment

A. Apparatus

1. 500-mL HDPE bottles: Scientific Specialties; # 334008-blk-1, or equivalent.
2. Centrifuge tubes – 15-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 05-539-5 or equivalent
3. 10-mL polypropylene volumetric flask, Class A – Fisher Scientific, Cat. No. S02288 or equivalent.
4. HDPE bottles for extraction fluid storage: L; Environmental Sampling Supply, Cat. No. 1000-1902-PC.
5. Analytical Balance – Capable of weighing to 0.0001 g
6. Top-Loading Balance – Capable of weighing to 0.01 g
7. Solid phase extraction (SPE) Weak Anion Exchange ("WAX") cartridge – Agilent; Sampli-Q WAX Polymer; 150mg/6mL; Cat. # 5982-3667.

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8. Large-volume SPE Reservoir (25-mL) - Millipore-Sigma; Product # 54258-U.
9. SPE Tube Adapter - Millipore-Sigma; Product # 57020-U.
10. SPE vacuum extraction manifold –“Resprep” 24-port manifold; Restek Corp catalog # 26080, or equivalent.
11. Polypropylene SPE delivery needles – Agilent; Cat. No. 12234511.
12. Centrifuge – “Q-Sep 3000”; Restek Corp. Cat. No. 26230, or equivalent, capable of a minimum rotational speed of 3000 rpm.
13. Disposable polyethylene pipette – Fisher Scientific, Cat. No. S30467-1 or equivalent.
14. Auto Pipettes – Eppendorf; capable of accurately dispensing 10- to 1000-µL. FisherScientific cat # 14-287-150, or equivalent.
15. Polypropylene pipette tips: 0-200µl. Fisher; Cat. No. 02-681-135
16. Polypropylene pipette tips: 101-1000µl. Fisher, Cat. No. 02-707-508
17. Pipettes – Disposable transfer. FisherScientific, Cat. No. 13-711-7M
18. Vortex mixer, variable speed, Fisher Scientific or equivalent.
19. N-Evap sample extract concentrator with N₂ supply and water bath for temperature control. Organomation, Inc. Cat. #11250, or equivalent.
20. Reagent Water Purification System: Capable of producing ultrapure “Type 1/Milli-Q”-grade water from in-house deionized water system. Millipore SAS; Cat. No. FTPF08831.
21. Thermo Target PP Polyspring inserts, catalog number C4010-630P
22. Agilent 9mm vial kit pack, catalog number 5190-2278, or equivalent
23. Centrifuge tubes – 50-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 06-443-21 or equivalent
24. Polypropylene bottles for standard storage - 4 mL; Fisher Scientific, Cat. No. 2006-9125
25. Stainless steel spatula/scoop set. Bel-Art SP Scienceware; Product # 11-865-130.
26. pH paper, range 0-14, Whatman Panpeha or equivalent, 0.5 unit readability

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27. Syringe filter - Acrodisc, Syringe Filter, GHP, 13 mm, 0.2 µm, Aqueous, 100/pkg, Part # WAT097962.

28. Silanized glass wool (Sigma-Aldrich, Cat #20411 or equivalent)

29. Disposable syringe filter, 25-mm, 0.2µm Nylon membrane, PALL/Acrodisc or equivalent

30. Glass fiber filter, 47 mm, 1 µm, PALL A/E or equivalent

31. Variable speed mixing table (Fisherbrand™ Nutating mixer or equivalent)

32. Evaporation/concentrator tubes: 60 mL clear glass vial, 30x125 mm, without caps (Wheaton Cat # W226060 or equivalent).

33. Wooden Tongue Depressors - Fisher; Cat. # 11-700-555, or equivalent.

34. Wheaton Bottle, 15ml, Narrow mouth, HDPE, Leak resistant; DWK Life Sciences, Cat. No. 209044SP, or equivalent

B. Equipment

1. AB Sciex Triple Quad 4500/5500/5500 Plus Turbo V Ion Source

ExionLC Controller
 ExionLC AC Pump
 ExionLC AC Autosampler
 Exion AC Column Oven
 Data system –Analyst 1.6.3

2. HPLC columns

a. Analytical column: Gemini 3µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4439-YO or equivalent

b. Pre-column: Luna, 5µm C18, 50 x 3 mm, Phenomenex Cat# 00B-4252-YO, or equivalent

Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions.

A. Reagents:

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1. Methanol (MeOH) – Honeywell Burdick and Jackson "Chromasolv LC-MS" grade Cat. No. BJ34966-4L or equivalent
2. Acetonitrile (ACN) – Fisher Scientific, Optima Cat. No. A955-4 or equivalent
3. Ammonium acetate – Fisher Scientific, Cat. No. A637-500 or equivalent
4. Ammonium hydroxide, 30% in water, certified ACS+ grade or equivalent, store at room temperature
5. Methanolic ammonium hydroxide (0.3%) – add ammonium hydroxide (10 mL, 30%) to methanol (990 mL), store at room temperature, replace after 1 month
6. Methanolic ammonium hydroxide (1%) - add ammonium hydroxide (3.3 mL, 30%) to methanol (97 mL), store at room temperature, replace after 1 month
7. Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid - add ammonium hydroxide (3.3 mL, 30%), reagent water (1.7 mL) and acetic acid (0.625 mL) to methanol (92 mL), store at room temperature, replace after 1 month.
8. Acetic Acid – ACS grade or equivalent, store at room temperature
9. Acetic Acid (0.1%) – dissolve acetic acid (1 mL) in reagent water (1 L), store at room temperature, replace after 3 months.
10. Formic acid
 - a. Formic acid (aqueous, 0.1 M) - dissolve formic acid (4.6 g) in reagent water (1 L), store at room temperature, replace after 2 years
 - b. Formic acid (aqueous, 0.3 M) - dissolve formic acid (13.8 g) in reagent water (1 L), store at room temperature, replace after 2 years
 - c. Formic acid (aqueous, 5% v/v) - mix 5 mL formic acid with 95 mL reagent water, store at room temperature, replace after 2 years
 - d. Formic acid (aqueous, 50% v/v) - mix 50 mL formic acid with 50 mL reagent water, store at room temperature, replace after 2 years
 - e. Formic acid (methanolic 1:1, 0.1 M formic acid/methanol) - mix equal volumes of methanol and 0.1 M formic acid, store at room temperature, replace after 2 years
11. "Superclean Envi-Carb"; bulk sorbent. Millipore-Sigma; 50g; Product # 57210-U.
12. Solids reference matrix – Ottawa or reagent-grade sand

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13. 20 mM ammonium acetate solution in 95:5(v/v) Milli-Q water/acetonitrile-Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 1900 mL Milli-Q water and mix well to dissolve the ammonium acetate. Add 100 mL acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.

14. 20 mM ammonium acetate solution in 90:10 acetonitrile/Milli-Q water – Weigh 3.08 ± 0.01g ammonium acetate into a 2-L glass mobile phase bottle. Add 200 mL of Milli-Q water and mix well to dissolve the Ammonium Acetate. Add 1800 mL of acetonitrile and mix well. Store at room temperature for up to 2 months. Different volumes can be prepared as long as final concentrations are equivalent.

B. Standards:

Standards are prepared using calibrated pipettes, polypropylene microcentrifuge tubes, polypropylene bottles, and 10 ml Class A PP volumetric flasks to create solutions at desired concentrations. The concentrated solution is injected below the surface of the diluting solvent. After preparation is completed, standards should be vortexed to ensure complete mixing. Measurement of volumes less than 5 µl should be avoided in routine production operations.

All stock, intermediate and spiking solutions are prepared using Methanol.

All initial calibration, initial calibration verification, and linear branched working standard solutions are prepared using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid.

All diluted solutions must be stored in HDPE containers that have been thoroughly rinsed with methanol.

Stock standard and intermediate standard solutions are stored in the refrigerator in labeled polypropylene screw-top vials, PP bottles, or PP centrifuge tubes.

Expiration dates are managed through LIMS Reagent. Solutions transferred from sealed glass ampules to screw-capped vials are given expiration dates of 1 year from the date opened or the expiration date provided by the vendor, whichever occurs sooner. Intermediate solutions are given an expiration date of 6 months from the preparation date, or the expiration date from the ampule provided by the vendor, whichever occurs sooner. The ampules and transferred solutions are stored in the refrigerator.

Working native and labeled (extraction surrogate and internal standard) compound spiking solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working solution, whichever occurs sooner. The solutions are stored in labeled polypropylene screw-top vials in the refrigerator. When these solutions are prepared they must be tested prior to use in the PFAS extraction lab and verified monthly until they are consumed by operations or expire. Records of the standard verification are maintained by the laboratory. Prior to use, the working spiking solution should be evaluated against recovery windows of 85-115% for all compounds that will be analyzed using that solution. Should a standard fail to meet these criteria, the data must be reviewed by departmental management for acceptability and/or corrective action.

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Working initial calibration solutions are given an expiration date of 6 months, or the expiration date of the solutions used to prepare the working initial calibration solution, whichever occurs sooner.

The primary/preferred standard vendor is Wellington Laboratories, Inc. Ontario, Canada. Listed catalog numbers are taken from Wellington product lists. Equivalent standards may be substituted, if the listed standards are unavailable.

The solution concentration listed is as presented on the certificate of analysis and includes adjustment for purity and the salt form of the compound used.

Note: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of analysis (CofA). See [Attachment 4](#).

If the compound purity is assayed to be 96% or greater, weight can be used without correction to calculate concentrations.

Log purchased standards into LIMS Reagent. Select the solution category SOURCE for purchased mixes and/or single-compound ampules. LIMS Reagent system will assign formatted names to the purchased standard solutions. The automatically-generated name can be overwritten with a manually created name if desired. Use labels printed through the LIMS Reagent to identify and track standard solutions after transfer from original ampule to storage vial. The CofA for the ampulated stock standard is attached in LIMS Reagent for reference.

Standards are prepared by transferring a known quantity of Standard to a final volume of solvent.

Standard Preparation is documented in LIMS Reagent. Solutions are stored by Type in LIMS Reagent, i.e., INTERMEDIATE=working solutions and intermediate standards and SOURCE=stocks (ampulated solutions). Each Standard is given a unique name.

The following attachments provide examples of standard preparation and purchasing information. Refer to the documentation in LIMS Reagent for standards preparation information.

- [Attachment 5](#) - Native PFAS Intermediate A
- [Attachment 6](#) - Native PFAS Intermediate B
- [Attachment 7](#) - Working Labeled Extraction Standard Spike
- [Attachment 8](#) - Internal Standard Spike
- [Attachment 9](#) - Native 1633 Mid-Level Spike
- [Attachment 10](#) - Native 1633 Low-Level Spike
- [Attachment 11](#) - 1633 Initial Calibration Standards Preparation
- [Attachment 12](#) - 1633 Initial calibration Standards Concentrations
- [Attachment 13](#) - TDCA Stock Solution
- [Attachment 14](#) - TDCA Working Solution A
- [Attachment 15](#) - TDCA Working Solution B
- [Attachment 16](#) - 1633 Linear/Branched TDCA Intermediate
- [Attachment 17](#) - 1633 Linear/Branched TDCA Solution

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[Attachment 18](#) - PFAS 1633 ICV Working Standard

[Attachment 19](#) - 1633 Labeled Ampulated Standards

[Attachment 20](#) - 1633 Native Ampulated Standards

Calibration

A. Initial Calibration

1. A minimum of six calibration standards are required when using an average or linear curve fit. A minimum of seven calibration standards are required for a second-order curve fit (quadratic). In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA, and NEtFOSAA. S/N ratio must be greater than or equal to 3:1 for all ions used for quantification.
2. Analyze a Cal4 level standard that contains TDCA retention time marker and linear and branch chained isomers of PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE. The analysis of this standard is used to evaluate the interference from bile salts in tissue samples, as well as evaluate where the branch chained isomers elute and not included in the calibration curve. This will assist the chemist in identifying and properly integrating this compound in samples.

Example Initial Calibration Sequence:

1. Instrument Blank
 2. Instrument Blank
 3. Instrument Blank
 4. CAL 1
 5. CAL 2
 6. CAL 3
 7. CAL 4
 8. CAL 5
 9. CAL 6
 10. CAL 7
 11. ICB (Instrument Blank)
 12. ICV
 13. MDL
 14. WDM (Linear Branched/TDCA standard)
3. Isotopically-labeled compounds are not available for some compounds. See below for compounds and their referenced extraction standards. See [Attachment 2](#) for additional information about compound relationships.
 4. Analyze a standard at a concentration of 100 ppb containing Taurodeoxycholic Acid (TDCA). The analysis of this standard is used to evaluate the chromatographic program relative to the risk of an interference from bile salts in tissue samples. The analytical conditions must be set to allow a separation of at least 1 minute between the bile salts and PFOS.

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NOTE: For better accuracy, PFTrDA is quantitated using the average of the areas of labeled compounds 13C2-PFTeDA and 13C2-PFDoA.

| Compound | Extraction Standard |
|----------|--------------------------------|
| PFBA | 13C4-PFBA |
| PFPeA | 13C5-PFPeA |
| 3:3FTCA | |
| PFMPA | |
| PFMBA | |
| PFHxA | 13C5-PFHxA |
| NFDHA | |
| 5:3FTCA | |
| 7:3FTCA | |
| PFEESA | |
| PFHpA | 13C4-PFHpA |
| PFOA | 13C8-PFOA |
| PFNA | 13C9-PFNA |
| PFDA | 13C6-PFDA |
| PFUnA | 13C7-PFUnA |
| PFDoA | 13C2-PFDoA |
| PFTrDA | Avg 13C2-PFTeDA and 13C2-PFDoA |
| PFTeDA | 13C2-PFTeDA |
| PFBS | 13C3-PFBS |
| PFPeS | 13C3-PFHxS |
| PFHxS | |

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|--------------|--------------|
| PFHpS | 13C8-PFOS |
| PFOS | |
| PFNS | |
| PFDS | |
| PFDoS | |
| 4:2-FTS | 13C2-4:2-FTS |
| 6:2-FTS | 13C2-6:2-FTS |
| 8:2-FTS | 13C2-8:2-FTS |
| PFOSA | 13C8-PFOSA |
| NMeFOSA | D3-NMeFOSA |
| NEtFOSA | D5-NEtFOSA |
| NMeFOSAA | D3-NMeFOSAA |
| NEtFOSAA | D5-N-EtFOSAA |
| NMeFOSE | D7-NMeFOSE |
| NEtFOSE | D9-NEtFOSE |
| HFPO-DA | 13C3-HFPO-DA |
| DONA | |
| 9Cl-PF3ONS | |
| 11Cl-PF3OUdS | |

5. Fit the curve

- a. If the %RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data.
- b. If the %RSD is greater than 20%, a linear regression with a concentration weighing factor of 1/x is tried for the compounds not meeting the criteria in 5.a. The RSE for all method analytes must be less than or equal to 20%
- c. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration for each calibration point should be within $\pm 30\%$ of its true value.

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d. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

NOTE: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of Analysis (CofA). See [Attachment 4](#).

6. Initial Calibration Verification (ICV)

A check standard prepared from a second source (ICV) is injected to confirm the validity of the calibration curve/standard. If a second source is not available, a separate preparation from the same stock by a second analyst may be used. The calculated amount for each analyte must be within $\pm 30\%$ of the true value. If this criteria is not met, re-inject or remake the standard. If the criteria is still not met, recalibration is necessary. Instrument maintenance may be needed prior to recalibrating.

B. Continuing calibration

1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten samples and at the end of the analysis sequence. Subsequent CCV standards should use the Cal4 level standard.
2. Acceptance criteria
 - a. The calculated amount for each compound (native and extraction standard) in the CCV standard must be within $\pm 30\%$ of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment. If two consecutive CCVs fail criteria for target analytes, two passing CCVs must be analyzed or the source of the problem determined and the system recalibrated before continuing sample analysis.
 - b. The absolute areas of the injection internal standards should be greater than 30% of the average areas measured during the initial calibration.

Procedure

A. Sample Preparation

NOTE: Prior to weighing out samples, thoroughly mix each sample using a wooden tongue depressor or stainless steel spoon to ensure a homogeneous sample matrix. Stir from the bottom to the top in

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a circular motion along the sides of the jar, breaking particles to less than 1 mm by pressing against the side of the container. Remove rocks, invertebrates, and foreign objects. Vegetation can either be removed or cut into smaller pieces based on project requirements.

1. On a calibrated, top-loading balance, accurately weigh 5.0g ± 0.10g (0.5 g for biosolids) of solid sample into a tared, labeled 15-mL centrifuge tube using a disposable polypropylene spatula. Record sample weight in the prep entry system.
2. For each batch - maximum 20 samples - include the following quality control samples:
 - a. Method Blank: Weigh 5.0g ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 g (0.25 g for biosolids) of reagent water
 - b. LCS: Fortify 5.0g ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 g (0.25 g for biosolids) reagent water and spiked with 200 µL of Native 1633 Mid-Level Spike Solution (PFC_1633_MID_XXXXX).
 - c. LLCS: Fortify 5.0g ± 0.10g (0.5 g for biosolids) of sand wetted with 2.5 g (0.25 g for biosolids) reagent water and spiked with 400 µL of Native 1633 Low-Level Spike Solution (PFC_1633_LOW_XXXXX).
 - d. Matrix Spike/Matrix Spike Duplicate (MS/MSD): Fortify 5.0g ± 0.10 g (0.5 g for biosolids) of sample as specified in sample preparation log with 200 µL of Native 1633 Mid-Level Spike Solution (PFC_1633_MID_XXXXX).
3. Add 25 µl working labeled extraction standard spike solution (PFC_1633_SS_XXXXX) to each sample/QC tube.
4. Cap and vortex for approximately 30 seconds.
5. Allow samples/QC to equilibrate for at least 30 minutes.
6. Add 10 mL of 0.3% methanolic ammonium hydroxide to each centrifuge tube.
7. Cap and vortex
8. Shake for 30 minutes on a variable speed mixing table
9. Centrifuge for 10 minutes and transfer supernatant to a clean 50 mL polypropylene centrifuge tube.
10. Add 15 mL of 0.3% methanolic ammonium hydroxide to the remaining solid sample in each centrifuge tube. Cap and vortex.
11. Shake for 30 minutes on a variable speed mixing table
12. Centrifuge for 10 minutes and decant the supernatant from the second extraction into the centrifuge tube with the supernatant from the first extraction.
13. Add another 5 mL of 0.3% methanolic ammonium hydroxide to the remaining sample in each centrifuge tube.
14. Shake by hand to disperse.
15. Immediately decant the supernatant from the third extraction into the centrifuge tube with the supernatant from the first and second extraction.
16. Using a 10 mg scoop, add 10 mg of Superclean Envi-Carb to the combined extract, mix by occasionally hand shaking for no more than 5 minutes.
17. Centrifuge for 10 minutes.
18. Immediately decant the extract into a new labeled 50ml PP centrifuge tube.
19. Concentrate the extracts at no more than 40°C with an N₂ flow of approximately 1.2 L/min to a final volume of approximately 3-5 mL.
20. Allow extracts to concentrate for 25 minutes, then vortex to mix thoroughly.
21. Continue concentrating and mixing every 10 minutes until the extract has been reduced to the required volume.

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22. Add enough reagent water to the extract to reach the "40ml" mark on the centrifuge tube and vortex. Check that the pH is 6.5 ± 0.5 and adjust as necessary with 50% formic acid or 30% ammonium hydroxide (or with 5% formic acid and 4% aqueous ammonium hydroxide).

B. Solid Phase Extraction (SPE)

1. Pack clean silanized glass wool to half the height of the WAX SPE cartridge barrel.
2. Label each SPE cartridge to correspond with each associated sample/QC piece and attach to a rinsed SPE port. Record the SPE port # for each sample/QC piece on the batchlog.
3. Condition each SPE cartridge with the following reagents in the following order:
 - a. 15 mL 1% methanolic ammonium hydroxide
 - b. 5 mL 0.3M formic acid
 - c. Discard conditioning eluent(s)
4. Label each sample bottle, cap and reservoir with the same number to insure samples are not inadvertently switched during the extraction procedure (i.e.; 1,1,1; 2,2,2; 3,3,3; etc.).
5. Attach a 25-mL SPE reservoir to each cartridge. Load the QC and samples to their respective cartridges. Allow full volume to pass through each cartridge by gravity, if possible. Apply light vacuum if necessary. The drip rate should be approximately 1-2 drops per second.
6. After full volume has passed through the cartridges, dry the cartridges with vacuum - no more than 15" of Hg - for approximately five minutes. After five minutes, visually inspect the cartridge to determine if the sorbent is dry. This done by comparing the cartridge to a visual standard (an unused SPE cartridge). If the sorbent is not dry, continue to check at one minute intervals until the cartridge is dry.
7. Discard the waste and rinse the waste reservoir with DI water. Wipe each needle with a Kim-wipe/methanol.
8. Rinse the walls of the reservoir with 5mL reagent water (twice) followed by 5ml of **1:1 0.1M formic acid/methanol** and pass the rinses through the cartridge using vacuum. Dry the cartridge by pulling air through for 15 seconds. Discard the rinse solution.
9. Place labeled 15-mL polypropylene centrifuge collection tubes under each respective SPE cartridge.
10. Rinse the inside of the evaporation/concentrator tube using 5mL of 1% methanolic ammonium hydroxide.

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11. Using a glass pipette, transfer the rinse to the SPE reservoirs, washing the walls of the reservoirs.
12. Apply a slight vacuum to the manifold in order to reclaim as much solvent as possible from the SPE cartridges.
13. Disconnect the cartridge/adaptor from the manifold. Remove the collection tubes.
14. Add 25 uL of concentrated acetic acid to each collection tube and vortex to mix thoroughly.

Note: The instrument lab chemist performs the next steps.

15. Add 25 uL of Internal Standard Spike Solution (PFC_ST_XXXXX) to each sample extract.
16. Bring each sample extract to final volume 5mL with methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution.
17. Cap and vortex to mix.
18. Place a syringe filter (25-mm filter, 0.2-um nylon membrane) on a 3 mL polypropylene syringe. Take the plunger out and carefully decant ~1 mL the sample supernatant into the syringe barrel. Replace the plunger and filter ~1 mL of sample into the corresponding labeled auto-sampler vial. Cap the auto-sampler vial. Samples are now ready for analysis.
19. Cap the centrifuge tube. Store the remaining centrifuged extracts in the refrigerator for dilution or reinjection if needed.

C. LC/MS/MS Analysis

1. Mass Calibration and Tuning
 - a. At instrument set up and installation, after the performance of major maintenance, or annually calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.
 - b. When masses fall outside of the ± 0.5 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.5 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.
2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (4500/5500/5500 Plus) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions.

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Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.

3. Acquisition method: See [Attachment 3](#). Mass Transitions: See [Attachment 1](#).
4. Instrument Sensitivity Check (ISC) and Instrument Blanks
 - a. Prior to sample analysis, an instrument sensitivity check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the LOQ. The CAL1 standard will be analyzed. All analyte concentrations must be within $\pm 30\%$ of their true value. The signal-to-noise ratio must be greater than or equal to 3:1. If the criteria is not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.
 - b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily or at the start of a sequence. The concentration of all analytes must be less than or equal to 1/2 the LOQ. If acceptance criteria are not met the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.
5. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.
6. After the initial calibration and when analyzing samples within the same tune, inject an instrument blank, followed by the ICV, Linear branched (L/B) standard, instrument sensitivity check, CCV standard using the CAL4, qualitative identification standard (includes TDCA RT marker), Instrument blank, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard at the CAL4 level, followed by an instrument blank.

Example Sample Sequence:

1. Instrument blank
 2. Instrument blank
 3. Instrument blank
 4. Instrument Sensitivity Check (CCV0 _CAL1)
 5. CCV 2 _CAL4
 6. Linear Branched/TDCA marker (WDM)
 7. Instrument Blank (ICB)
 8. Method Blank (MB)
 9. Low Level LCS (LLCS)
 10. LCS
 11. Sample (10 or less)
 12. CCV 3 _CAL4
 13. Instrument Blank
7. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and

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retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

8. Quantitate results for the extraction blank. No target analytes at or above the reporting limit, at or greater than one-third the regulatory compliance limit, at or greater than one-tenth the concentration in a sample in the extraction batch, whichever is greatest, may be found in the extraction blank for acceptable batch results. If this criteria is not met, the samples must be re-extracted.
9. Calculate the recoveries of spiked analytes for the LLCS, LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.
 - a. LLCS, LCS, MS, extraction standard recoveries and RPDs are calculated and compared to the limits stored on the LIMS.
 - b. If LLCS and LCS recoveries are acceptable, proceed to sample quantitation.
 - c. If the LCS and LLCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, the samples associated with the LCS/LLCS must be reextracted.
 - d. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.
10. Isotopically-labeled extraction standards are added to all samples, extraction blank, LLCS/LCS, and MS/MSD prior to extraction. The recovery of the extraction standards should be within QC acceptance criteria. If the extraction standard recovery(ies) is(are) outside the QC limit(s), reextract using a reduced sample volume. If the extraction standard recovery(ies) is(are) again outside the QC limit(s), consult a supervisor to determine the appropriate course of action based on batch and sample results.
11. Isotopically-labeled injection standards are added to each QC and field sample extract prior to analysis. The absolute areas of the injection standards should be within 30-200% of the average areas measured during the initial calibration. If the internal standards are recovered outside 30-200%, consult a supervisor to determine the appropriate course of action based on batch and sample results.
12. Compare the retention times of all of the analytes, surrogates, and internal standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than
 - a. 0.4 minutes for isotopically-labeled compounds

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- b. 0.1 minutes from their analog for native compounds with an exact isotopically-labeled compound
- c. 0.4 minutes from their assigned analog for native compounds without an exact isotopically-labeled compound.

If the retention time is outside of the criteria, the compound is considered a false positive unless it is a compound with branched isomers. Compounds with branched isomers can vary in intensity of the individual isomers that are used for reporting and must be reviewed and compared to the preceding CCV to determine if it should be reported.

13. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of 13C4-PFBA, 13C5-PFPeA, 13C4-PFHpA, 13C8-PFOA, 13C9-PFNA, 13C6-PFDA, 13C7-PFUnA, 13C2-PFDA, 13C2-PFDoDA, 13C2-PFTeDA, 13C8-PFOSA, D3-NMePFOSA, D5-NEtFOSAA, D3-NMeFOSAA, D5-NEtPFOSA, D7-NMePFOSAE, D9-NEtPFOSAE, 13C3-PFBA, 13C4-PFOA, 13C5-PFNA, 13C2-PFOA, 18O2-PFHxS, PFBA, PFECA F(PFMPA), PFECA A(PFMBA), NMePFOSAE, and NEtPFOSAE. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards. When an ion ratio for a compound differs from the expected ion ratio by more than 50%, a qualifier is placed on the raw data and on the sample report. No corrective action is required.
14. The linear/branch chain standard is used when assessing the correctness of the computer generated peak integrations for PFOA, PFNA, PFOSA, NMeFOSA, NEtFOSA, NMeFOSE, and NEtFOSE.
15. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract using Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution and adjust the amount of Internal Standard Spike solution in the diluted extract. Select the dilution so that the expected EIS recoveries in the diluted extract are >5%. Extracts requiring greater than a 10x dilution should be reextracted using a reduced aliquot.

Dilution Example 1/10: Mix 895 µl of Methanol with 4% water, 1% ammonium hydroxide and 0.625% acetic acid solution with 100 µl of sample extract and 5 uL of Internal Standard Spike solution. Vortex to mix. Using an auto-pipette, transfer an aliquot of the mixed solution into a labeled auto-sampler vial. Cap and vortex thoroughly to mix.

Calculations

1. Peak Area Ratio

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$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

2. On-Column Analyte Concentration using average RRF

$$\text{On-column Concentration} = \text{peak area ratio} \div \text{AVE RRF}$$

3. On-Column Analyte Concentration using linear curve

$$\text{On-column Concentration} = (\text{peak area ratio} - \text{intercept}) \div \text{slope}$$

4. Sample Concentration

$$\text{Sample concentration (ng/g)} = (\text{On-column concentration} \times \text{Final Sample Volume} \times \text{DF}) \div \text{Initial Sample Volume}$$

5. Ion Ratio

$$\text{ion ratio} = (\text{peak area or height of quantifier}) / (\text{peak area or height of qualifier})$$

5. See [T-PEST-WI9847](#) for additional calculations used to evaluate the calibrations and quality control samples.

Statistical Information/Method Performance

The LCS should contain all compounds of interest. LCS, MS, and extraction standard recoveries are compared to the limits stored on the LIMS. These limits are statistically derived when sufficient data points are available. If sufficient data points are not available to generate statistical windows advisory limits will be used.

| QC parameter | Lower acceptance limit | High acceptance limit |
|---------------------------------------|--|-----------------------|
| Extracted Internal standard (EIS) | 20% | 150% |
| Non-extracted Internal Standard (NIS) | >30% of the average NIS from the initial calibration | 200% |
| Analyte recoveries | 40% | 150% |

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| LCS/LLCS/MS/MSD | | |

Note: lower acceptance limit for EIS cannot not be <20%, lower acceptance limit for analyte recovery cannot be <40%.

Historical data for MS/Ds, LCSs, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to [QA-SOP11892](#) for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

Quality Assurance/Quality Control

For each batch of samples extracted, a method blank(sand) and an LCS/LLCS (sand spiked with all compounds to be determined carried through the entire procedure) must be extracted and analyzed. MS/MSD is extracted only if submitted by the client. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared.

If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

Attachment:

- Attachment 1 - Mass Transitions (.doc)
- Attachment 10 - Native Low Level Spike (.pdf)
- Attachment 11 - 1633 Initial Calibration Standards Preparation (.pdf)
- Attachment 12 - 1633 Initial Calibration Standard Concentrations (.pdf)
- Attachment 13 - TDCA Stock Solution (.pdf)
- Attachment 14 - TDCA working Solution A (.pdf)
- Attachment 15 - TDCA Working Solution B (.pdf)
- Attachment 16 - 1633 Linear Branched and TDCA Intermediate (.pdf)
- Attachment 17 - 1633 Linear Branched and TDCA Solution (.pdf)
- Attachment 18 - PFAS ICV Working Standard (.pdf)
- Attachment 19 - 1633 Labeled Ampulated Standards (.pdf)
- Attachment 2 - Standards relationships (.docx)
- Attachment 20 - 1633 Native Ampulated Standards (.pdf)
- Attachment 3 - Acquisition Parameters (.pdf)
- Attachment 4 - Example Certificate of Analysis (.pdf)
- Attachment 5 - 1633 Native PFAS Intermediate A (.pdf)
- Attachment 6 - Native PFAS Intermediate B (.pdf)
- Attachment 7 - Working Labeled Extraction Standard Spike (.pdf)
- Attachment 8 - Internal Standard Spike (.pdf)
- Attachment 9 - Native Mid Level Spike (.pdf)

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- 11178 Demonstrations of Capability
- 11892 Determining Method Detection Limits and Limits of Quantitation
- 21568 Manifold and N-EVAP Cleaning for PFAS Extractions
- 9847 Common Equations Used During Chromatographic Analyses
- Attachment: Attachment 1 - Mass Transitions (doc)
- Attachment: Attachment 10 - Native Low Level Spike (pdf)
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- Attachment: Attachment 15 - TDCA Working Solution B (pdf)
- Attachment: Attachment 16 - 1633 Linear Branched and TDCA Intermediate (pdf)
- Attachment: Attachment 17 - 1633 Linear Branched and TDCA Solution (pdf)
- Attachment: Attachment 18 - PFAS ICV Working Standard (pdf)
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- Attachment: Attachment 20 - 1633 Native Ampulated Standards (pdf)
- Attachment: Attachment 3 - Acquisition Parameters (pdf)
- Attachment: Attachment 4 - Example Certificate of Analysis (pdf)
- Attachment: Attachment 5 - 1633 Native PFAS Intermediate A (pdf)
- Attachment: Attachment 6 - Native PFAS Intermediate B (pdf)
- Attachment: Attachment 7 - Working Labeled Extraction Standard Spike (pdf)
- Attachment: Attachment 8 - Internal Standard Spike (pdf)
- Attachment: Attachment 9 - Native Mid Level Spike (pdf)

End of document

Version history

| Version | Approval | Revision information |
|---------|-------------|----------------------|
| 1 | 26.MAY.2022 | |
| 2 | 05.OCT.2022 | |

Attachment 1

Mass Transitions AB Sciex 4500/5500/5500+

| Compound | Parent Ion | Daughter Ion |
|------------------|------------|--------------|
| 13C3-PFBA | 216.0 | 172.0 |
| 13C4-PFBA | 216.8 | 171.9 |
| PFBA | 212.8 | 168.9 |
| 13C5-PFPeA | 268.3 | 223 |
| PFPeA | 263.0 | 219.0 |
| PFPeA (2) | 263.0 | 68.9 |
| 13C3-PFBS | 302.1 | 79.9 |
| 13C3-PFBS (2) | 302.1 | 98.9 |
| PFBS | 298.7 | 79.9 |
| PFBS (2) | 298.7 | 98.8 |
| 13C2-4:2-FTS | 329.1 | 80.9 |
| 13C2-4:2-FTS (2) | 329.1 | 309.0 |
| 4:2-FTS | 327.1 | 307.0 |
| 4:2-FTS (2) | 327.1 | 80.9 |
| 13C2-PFHxA | 315.1 | 270.0 |
| 13C2-PFHxA (2) | 315.1 | 119.4 |
| 13C5-PFHxA | 318.0 | 273.0 |
| 13C5-PFHxA (2) | 318.0 | 120.3 |
| PFHxA | 313.0 | 269.0 |
| PFHxA (2) | 313.0 | 118.9 |
| PFPeS | 349.1 | 79.9 |
| PFPeS (2) | 349.1 | 98.9 |
| 18O2-PFHxS | 403.0 | 83.9 |
| 13C3-PFHxS | 402.1 | 79.9 |
| 13C3-PFHxS (2) | 402.1 | 98.8 |
| PFHxS | 398.7 | 79.9 |
| PFHxS (2) | 398.7 | 98.9 |
| 13C4-PFHpA | 367.1 | 322.0 |
| PFHpA | 363.1 | 319.0 |
| PFHpA (2) | 363.1 | 169.0 |
| 13C2-6:2-FTS | 429.1 | 80.9 |
| 13C2-6:2-FTS (2) | 429.1 | 409.0 |
| 6:2-FTS | 427.1 | 407.0 |
| 6:2-FTS (2) | 427.1 | 80.9 |
| PFHpS | 449.0 | 79.9 |
| PFHpS (2) | 449.0 | 98.8 |
| 13C4-PFOA | 417.1 | 172.0 |

Attachment 1

| Compound | Parent Ion | Daughter Ion |
|------------------|------------|--------------|
| 13C8-PFOA | 421.1 | 376.0 |
| PFOA | 413.0 | 369.0 |
| PFOA (2) | 413.0 | 169.0 |
| 13C4-PFOS | 502.8 | 79.9 |
| 13C4-PFOS (2) | 502.8 | 98.9 |
| 13C8-PFOS | 507.1 | 79.9 |
| 13C8-PFOS (2) | 507.1 | 98.9 |
| PFOS | 498.9 | 79.9 |
| PFOS (2) | 498.9 | 98.8 |
| 13C5-PFNA | 468.0 | 423.0 |
| 13C9-PFNA | 472.1 | 427.0 |
| PFNA | 463.0 | 419.0 |
| PFNA (2) | 463.0 | 219.0 |
| 13C8-PFOSA | 506.1 | 77.8 |
| PFOSA | 498.1 | 77.9 |
| PFOSA (2) | 498.1 | 478.0 |
| PFNS | 548.8 | 79.9 |
| PFNS (2) | 548.8 | 98.8 |
| 13C2-PFDA | 515.1 | 470.1 |
| 13C6-PFDA | 519.1 | 474.1 |
| PFDA | 512.9 | 469.0 |
| PFDA (2) | 512.9 | 219.0 |
| 13C2-8:2-FTS | 529.1 | 80.9 |
| 13C2-8:2-FTS (2) | 529.1 | 509.0 |
| 8:2-FTS | 527.1 | 507.0 |
| 8:2-FTS (2) | 527.1 | 80.8 |
| d7-NMePFOSAE | 623.2 | 58.9 |
| NMePFOSAE | 616.1 | 58.9 |
| d3-NMePFOSA | 515.0 | 219.0 |
| NMEPFOSA | 511.9 | 219.0 |
| NMEPFOSA (2) | 511.9 | 169.0 |
| d3-NMeFOSAA | 573.2 | 419.0 |
| NMeFOSAA | 570.1 | 419.0 |
| NMeFOSAA (2) | 570.1 | 483.0 |
| d9-NEtPFOSAE | 639.2 | 58.9 |
| NEtPFOSAE | 630.0 | 58.9 |
| d5-NETPFOSA | 531.1 | 219.0 |
| NEtPFOSA | 526.0 | 219.0 |
| NEtPFOSA (2) | 526.0 | 169.0 |
| PFDS | 599.0 | 79.9 |

Attachment 1

| Compound | Parent Ion | Daughter Ion |
|--------------------|------------|--------------|
| PFDS (2) | 599.0 | 98.8 |
| 13C7-PFUnDA | 570.0 | 525.1 |
| PFUnDA | 563.1 | 519.0 |
| PFUnDA (2) | 563.1 | 269.1 |
| d5-NEtFOSAA | 589.2 | 419.0 |
| NEtFOSAA | 584.2 | 419.1 |
| NEtFOSAA (2) | 584.2 | 526.0 |
| 13C2-PFDoDA | 615.1 | 570.0 |
| PFDoDA | 613.1 | 569.0 |
| PFDoDA (2) | 613.1 | 319.0 |
| PFDoS | 699.1 | 79.9 |
| PFDoS (2) | 699.1 | 98.8 |
| PFTrDA | 663.0 | 619.0 |
| PFTrDA (2) | 663.0 | 168.9 |
| 13C2-PFTeDA | 715.2 | 670.0 |
| PFTeDA | 713.1 | 669.0 |
| PFTeDA (2) | 713.1 | 168.9 |
| 13C3-HFPODA | 286.9 | 168.9 |
| 13C3-HFPODA (2) | 286.9 | 184.9 |
| HFPODA | 284.9 | 168.9 |
| HFPODA (2) | 284.9 | 184.9 |
| DONA | 376.9 | 250.9 |
| DONA (2) | 376.9 | 84.8 |
| 9Cl-PF3ONS | 530.8 | 351.0 |
| 9Cl-PF3ONS (2) | 532.8 | 353.0 |
| 11Cl-PF3OUdS | 630.9 | 450.9 |
| 11Cl-PF3OUdS (2) | 632.9 | 452.9 |
| PFECA B (NFDHA) | 295.0 | 201.0 |
| PFECA B(NFDHA) (2) | 295.0 | 84.9 |
| PFECA F (PFMPA) | 229.0 | 84.9 |
| 3:3 FTCA | 241.0 | 177.0 |
| 3:3 FTCA (2) | 241.0 | 117.0 |
| PFECA A (PFMBA) | 279.0 | 85.1 |
| PFEESA (PES) | 314.8 | 134.9 |
| PFEESA (PES) (2) | 314.8 | 82.9 |
| 5:3 FTCA | 341.0 | 237.1 |
| 5:3 FTCA (2) | 341.0 | 217.0 |
| 7:3 FTCA | 441.0 | 316.9 |
| 7:3 FTCA (2) | 441.0 | 336.9 |

| Native 1633 Low-Level Spike | | | | | | | | |
|-----------------------------|----------------|--|-------------|--------------|---------------|--------------|---------------|---|
| Vendor | Catalog Number | Analyte | CAS# | Acronym | Conc. (ng/mL) | Aliquot (mL) | Final Volume | Final Conc. Native 1633 Low-Level Spike (ng/ml) |
| Wellington | PFAC-MXF | 11-Chloroicosafuoro-3-oxaundecane-1-sulfonic acid | 763051-92-9 | 11CI-PF3OUdS | 1890 | 0.1 | 10mL Methanol | 18.9 |
| | | 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid | 756426-58-1 | 9CI-PF3ONS | 1870 | | | 18.7 |
| | | 4,8-dioxa-3H-Perfluorononanoic acid | 919005-14-4 | DONA | 1890 | | | 18.9 |
| | | Perfluoro(2-propoxypropanoic) acid | 13252-13-6 | HFPODA | 2000 | | | 10 |
| Wellington | PFAC-MXH | 1H,1H,2H,2H perfluorodecanesulfonic acid | 39108-34-4 | 8:2-FTS | 3840 | 0.05 | 10mL Methanol | 19.2 |
| | | 1H,1H,2H,2H perfluorohexanesulfonic acid | 757124-72-4 | 4:2-FTS | 3750 | | | 18.75 |
| | | 1H,1H,2H,2H perfluorooctanesulfonic acid | 27619-97-2 | 6:2-FTS | 3800 | | | 19 |
| | | N-ethylperfluorooctanesulfonamidoacetic acid | 2991-50-6 | NEIFOSAA | 1000 | | | 5 |
| | | N-methylperfluorooctanesulfonamidoacetic acid | 2355-31-9 | NMeFOSAA | 1000 | | | 5 |
| | | Perfluorobutanesulfonic acid | 375-73-5 | PFBS | 887 | | | 4.435 |
| | | Perfluorobutanoic acid | 375-22-4 | PFBA | 4000 | | | 20 |
| | | Perfluorodecanesulfonic acid | 335-77-3 | PFDS | 965 | | | 4.825 |
| | | Perfluorodecanoic acid | 335-76-2 | PFDA | 1000 | | | 5 |
| | | Perfluorododecanesulfonic acid | 79780-39-5 | PFDoDS | 970 | | | 4.850 |
| | | Perfluorododecanoic acid | 307-55-1 | PFDoDA | 1000 | | | 5 |
| | | Perfluoroheptanesulfonic acid | 375-92-8 | PFHpS | 953 | | | 4.765 |
| | | Perfluoroheptanoic acid | 375-85-9 | PFHpA | 1000 | | | 5 |
| | | Perfluorohexanesulfonic acid | 355-46-4 | PFHxS | 914 | | | 4.57 |
| | | Perfluorohexanoic acid | 307-24-4 | PFHxA | 1000 | | | 5 |
| | | Perfluorononanesulfonic acid | 68259-12-1 | PFNS | 962 | | | 4.81 |
| | | Perfluorononanoic acid | 375-95-1 | PFNA | 1000 | | | 5 |
| | | Perfluorooctanesulfonamide | 754-91-6 | PFOSA | 1000 | | | 5 |
| | | Perfluorooctanesulfonic acid | 1763-23-1 | PFOS | 928 | | | 4.64 |
| | | Perfluorooctanoic acid | 335-67-1 | PFOA | 1000 | | | 5 |
| | | Perfluoropentanesulfonic acid | 2706-91-4 | PFPeS | 941 | | | 4.705 |
| | | Perfluoropentanoic acid | 2706-90-3 | PFPeA | 2000 | | | 10 |
| | | Perfluorotetradecanoic acid | 376-06-7 | PFTeDA | 1000 | | | 5 |
| | | Perfluorotridecanoic acid | 72629-94-8 | PFTrDA | 1000 | | | 5 |
| Perfluoroundecanoic acid | 2058-94-8 | PFUnDA | 1000 | 5 | | | | |
| Wellington | PFAC-MXG | Perfluoro-3-methoxypropanoic acid | 377-73-1 | PFMPA | 2000 | 0.05 | 10mL Methanol | 10 |
| | | Perfluoro-4-methoxybutanoic acid | 863090-89-5 | PFMBA | 2000 | | | 10 |
| | | Nonafluoro-3,6-dioxaheptanoic acid | 151722-58-6 | NFDHA | 2000 | | | 10 |
| | | Perfluoro(2-ethoxyethane)sulfonic acid | 113507-82-7 | PFEESA | 1780 | | | 8.9 |
| Wellington | PFAC-MXI | 2-(N-methylperfluoro-1-octanesulfonamido)-ethanol | 24448-09-7 | NMePFOSAE | 10000 | 0.05 | 10mL Methanol | 50 |
| | | N-methylperfluoro-1-octanesulfonamide | 31506-32-8 | NMePFOSA | 1000 | | | 5 |
| | | 2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol | 1691-99-2 | NEIPFOSAE | 10000 | | | 50 |
| | | N-ethylperfluoro-1-octanesulfonamide | 4151-50-2 | NEIPFOSA | 1000 | | | 5 |
| Wellington | PFAC-MXJ | 3-Perfluoropropylpropanoic acid | 763051-92-9 | 3:3 FTCA | 4000 | 0.0626 | 10mL Methanol | 25.04 |
| | | 3-Perfluoropentylpropanoic acid | 756426-58-1 | 5:3 FTCA | 20000 | | | 125.2 |
| | | 3-Perfluoroheptylpropanoic acid | 919005-14-4 | 7:3 FTCA | 20000 | | | 125.2 |

| TDCA Stock Solution | | | | | | | | |
|---------------------|----------------|----------------------------------|-------------|---------|------------|-------------|----------------------------|---|
| Vendor | Catalog Number | Analyte | CAS# | Acronym | Conc. (mg) | Aliquot (g) | Final Volume (ml) Methanol | Final Conc. TDCA Stock Solution (ng/ml) |
| Sigma Alrich | T0557-500MG | Sodium Taurodeoxycholate hydrate | 207737-97-1 | TDCA | 1000000 | 0.05 | 50 | 1000000 |

| TDCA Working Solution A | | | | | | | |
|-------------------------|----------------------------------|-------------|---------|---------------|--------------|----------------------------|---|
| Solution Name | Analyte | CAS# | Acronym | Conc. (ng/ml) | Aliquot (mL) | Final Volume (mL) Methanol | Final Conc. TDCA Working Solution A (ng/ml) |
| TDCA Stock Solution | Sodium Taurodeoxycholate hydrate | 207737-97-1 | TDCA | 1000000 | 1 | 4 | 250000 |

| TDCA Working Solution B | | | | | | | |
|-------------------------|----------------------------------|-------------|---------|---------------|--------------|----------------------------|---|
| Solution Name | Analyte | CAS# | Acronym | Conc. (ng/ml) | Aliquot (mL) | Final Volume (ml) Methanol | Final Conc. TDCA Working Solution B (ng/ml) |
| TDCA Working Solution A | Sodium Taurodeoxycholate hydrate | 207737-97-1 | TDCA | 250000 | 0.10 | 5 | 5000 |

| 1633 Linear/Branched TDCA Intermediate | | | | | | | | |
|--|----------------|--|------------|-----------|---------------|--------------|----------------------------|---|
| Vendor | Catalog Number | Analyte | CAS# | Acronym | Conc. (ng/mL) | Aliquot (mL) | Final Volume (ml) Methanol | Final Conc. 1633 Linear/ Branched TDCA Intermediate (ng/ml) |
| Wellington | T-PFOA | Technical Ammonium, Perfluorooctanoate (Technical Grade) | 95328-99-7 | T-PFOA | 50000 | 0.02 | 2mL | 500 |
| | | Perfluorooctanoic acid | 335-67-1 | PFOA | 50000 | | | 500 |
| Cambridge Isotope Laboratories, Inc. | ULM-11036-S | 2-(N-ethylperfluoro-1-octanesulfonamido) ethanol | 1691-99-2 | NEtPFOSAE | 50000 | 0.02 | | 500 |
| Cambridge Isotope Laboratories, Inc. | ULM-11034-S | 2-(N-methylperfluoro-1-octanesulfonamido) ethanol | 24448-09-7 | NMePFOSAE | 50000 | 0.02 | | 500 |
| Cambridge Isotope Laboratories, Inc. | ULM-10780-S | N-ethylperfluoro-1-octanesulfonamide | 4151-50-2 | NEtPFOSA | 100000 | 0.01 | | 500 |
| Cambridge Isotope Laboratories, Inc. | ULM-10779-S | N-methylperfluoro-1-octanesulfonamide | 31506-32-8 | NMeFOSA | 100000 | 0.01 | | 500 |
| Cambridge Isotope Laboratories, Inc. | ULM-10977-S | Perfluorooctanesulfonamide | 754-91-6 | PFOSA | 50000 | 0.02 | | 500 |
| Wellington | ipPFNA | Perfluoro-7-methyloctanoic acid | 15899-31-7 | PF7MOA | 50000 | 0.02 | | 500 |
| Wellington | PFNA | Perfluorononanoic acid | 375-95-1 | PFNA | 50000 | 0.02 | | 500 |

| 1633 Linear/Branched TDCA Solution | | | | | | | |
|--|--|-------------|--------------------------|---------------|--------------|--------------------|--|
| Solution Name | Analyte | CAS# | Acronym | Conc. (ng/mL) | Aliquot (mL) | Final Volume* (ml) | Final Conc. 1633 Linear/Branched TDCA Solution (ng/ml) |
| TDCA Working Solution B | Sodium Taurodeoxycholate hydrate | 207737-97-1 | TDCA | 5000 | 0.01 | 2 | 25 |
| 1633 Linear/Branched TDCA Intermediate | 2-(N-ethylperfluoro-1-octanesulfonamido) ethanol | 1691-99-2 | NEIPFOSAE | 500 | 0.02 | | 5 |
| | 2-(N-methylperfluoro-1-octanesulfonamido) ethanol | 24448-09-7 | NMePFOSAE | 500 | | | 5 |
| | N-ethylperfluoro-1-octanesulfonamide | 4151-50-2 | NEIPFOSA | 500 | | | 5 |
| | N-methylperfluoro-1-octanesulfonamide | 31506-32-8 | NMeFOSA | 500 | | | 5 |
| | Perfluorooctanesulfonamide | 754-91-6 | PFOSA | 500 | | | 5 |
| | Perfluoro-7-methyloctanoic acid | 15899-31-7 | PF7MOA | 500 | | | 5 |
| | Perfluorononanoic acid | 375-95-1 | PFNA | 500 | | | 5 |
| | Technical Ammonium, Perfluorooctanoate (Technical Grade) | 95328-99-7 | T-PFOA | 500 | | | 5 |
| | Perfluorooctanoic acid | 335-67-1 | PFOA | 500 | | | 5 |
| Mass-Labelled PFAS Extraction Standard Solution/Mixture-ES | Perfluoro-n-[¹³ C4]butanoic acid | STL00992 | ¹³ C4-PFBA | 2000 | 0.01 | 10 | |
| | Perfluoro-n-[¹³ C5]pentanoic acid | STL01893 | ¹³ C5-PFPeA | 1000 | | 5 | |
| | Perfluoro-n-[1,2,3,4,6- ¹³ C5]hexanoic acid | STL02577 | ¹³ C5-PFHxA | 500 | | 2.5 | |
| | Perfluoro-n-[1,2,3,4- ¹³ C4]heptanoic acid | STL01892 | ¹³ C4-PFHpA | 500 | | 2.5 | |
| | Perfluoro-n-[¹³ C8]octanoic acid | STL01052 | ¹³ C8-PFOA | 500 | | 2.5 | |
| | Perfluoro-n-[¹³ C9]nonanoic acid | STL02578 | ¹³ C9-PFNA | 250 | | 1.25 | |
| | Perfluoro-n-[1,2,3,4,5,6- ¹³ C6]decanoic acid | STL02579 | ¹³ C6-PFDA | 250 | | 1.25 | |
| | Perfluoro-n-[1,2,3,4,5,6,7- ¹³ C7]undecanoic acid | STL02580 | ¹³ C7-PFUaA | 250 | | 1.25 | |
| | Perfluoro-n-[1,2- ¹³ C2]dodecanoic acid | STL02703 | ¹³ C2-PFDaA | 250 | | 1.25 | |
| | Perfluoro-n-[1,2- ¹³ C2]tetradecanoic acid | STL02116 | ¹³ C2-PFTeDA | 250 | | 1.25 | |
| | Perfluoro-1-[2,3,4- ¹³ C3]butanesulfonic acid | STL02337 | ¹³ C3-PFBS | 466 | | 2.33 | |
| | Perfluoro-1-[1,2,3- ¹³ C3]hexanesulfonic acid | STL02581 | ¹³ C3-PFHxA | 474 | | 2.37 | |
| | Perfluoro-1-[¹³ C8]octanesulfonic acid | STL01054 | ¹³ C8-PFOS | 479 | | 2.4 | |
| | Perfluoro-1-[¹³ C8]octanesulfonamide | STL01056 | ¹³ C8-PFOSA | 500 | | 2.5 | |
| | N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid | STL02118 | D3-NMeFOSAA | 1000 | | 5 | |
| | N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid | STL02117 | D5-NEIFOSAA | 1000 | | 5 | |
| | 1H,1H,2H,2H-Perfluoro-1-[1,2- ¹³ C2]hexanesulfonic acid | STL02395 | ¹³ C2-4:2FTS | 938 | | 4.69 | |
| | 1H,1H,2H,2H-Perfluoro-1-[1,2- ¹³ C2]octanesulfonic acid | STL02279 | ¹³ C2-6:2FTS | 951 | | 4.76 | |
| | 1H,1H,2H,2H-Perfluoro-1-[1,2- ¹³ C2]decanesulfonic acid | STL02280 | ¹³ C2-8:2FTS | 960 | | 4.8 | |
| | Tetrafluoro-2-heptafluoropropoxy- ¹³ C3-propanoic acid | STL02255 | ¹³ C3-HFPO-DA | 2000 | | 10 | |
| | N-methyl-d7-perfluorooctanesulfonamidoethanol | STL02277 | D7-NMeFOSE | 5000 | | 25 | |
| | N-ethyl-d9-perfluorooctanesulfonamidoethanol | STL02278 | D9-NEIFOSE | 5000 | | 25 | |
| | N-ethyl-d5-perfluoro-1-octanesulfonamide | STL02704 | D5-NEIFOSA | 500 | | 2.5 | |
| N-methyl-d3-perfluoro-1-octanesulfonamide | STL02705 | D3-NMeFOSA | 500 | 2.5 | | | |
| Mass-Labelled PFAS Injection Standard Solution/Mixture | Perfluoro-n-[2,3,4- ¹³ C3]butanoic acid | STL02680 | ¹³ C3-PFBA | 1000 | 0.01 | 5 | |
| | Perfluoro-n-[1,2,3,4- ¹³ C4]octanoic acid | STL00990 | ¹³ C4-PFOA | 500 | | 2.5 | |
| | Perfluoro-n-[1,2- ¹³ C2]decanoic acid | STL00996 | ¹³ C2-PFDA | 250 | | 1.25 | |
| | Perfluoro-n-[1,2,3,4- ¹³ C4]octanesulfonic acid | STL00991 | ¹³ C4-PFOS | 479 | | 2.4 | |
| | Perfluoro-n-[1,2,3,4,5- ¹³ C5]nonanoic acid | STL00995 | ¹³ C5-PFNA | 250 | | 1.25 | |
| | Perfluoro-n-[1,2- ¹³ C2]hexanoic acid | STL00993 | ¹³ C2-PFHxA | 500 | | 2.5 | |
| | Perfluoro-1-hexane[¹⁸ O2]sulfonic acid | STL00994 | ¹⁸ O2-PFHxA | 474 | | 2.37 | |

* Bring to final volume using methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid

| PFAS 1633 ICV Working Standard | | | | | | | |
|---|---|-------------|--------------------------------------|---------------|--------------|--------------------|--|
| Solution Name | Analyte | CAS# | Acronym | Conc. (ng/ml) | Aliquot (ml) | Final Volume* (ml) | Final Conc. PFAS 1633 ICV Working Standard (ng/ml) |
| Native PFAS Intermediate A | 11-Chloroicosulfuro-3-oxaundecane-1-sulfonic acid | 763051-92-9 | 11Cl-PF3OUdS | 94.5 | 0.5 | 5 | 9.45 |
| | 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid | 756426-58-1 | 9Cl-PF3ONS | 93.5 | | | 9.35 |
| | 4,8-dioxo-3H-Perfluorononanoic acid | 919005-14-4 | DONA | 94.5 | | | 9.45 |
| | Perfluoro(2-propylpropanoic) acid | 13252-13-6 | HFPODA | 100 | | | 10 |
| | 1H,1H,2H,2H perfluorodecanesulfonic acid | 39108-34-4 | 8:2-FTS | 96 | | | 4.69 |
| | 1H,1H,2H,2H perfluorohexanesulfonic acid | 757124-72-4 | 4:2-FTS | 93.8 | | | 4.76 |
| | 1H,1H,2H,2H perfluorooctanesulfonic acid | 27819-97-2 | 6:2-FTS | 95 | | | 4.8 |
| | N-ethylperfluorooctanesulfonamidoacetic acid | 2991-50-6 | NEtFOSAA | 25 | | | 2.5 |
| | N-methylperfluorooctanesulfonamidoacetic acid | 2355-31-9 | NMeFOSAA | 25 | | | 2.5 |
| | Perfluorobutanesulfonic acid | 375-73-5 | PFBS | 22.2 | | | 2.22 |
| | Perfluorobutanoic acid | 375-22-4 | PFBA | 100 | | | 10 |
| | Perfluorodecanesulfonic acid | 335-77-3 | PFDS | 24.1 | | | 2.41 |
| | Perfluorodecanoic acid | 335-76-2 | PFDA | 25 | | | 2.5 |
| | Perfluorododecanesulfonic acid | 79780-39-5 | PFDDoS | 24.3 | | | 2.43 |
| | Perfluorododecanoic acid | 307-55-1 | PFDDoA | 25 | | | 2.5 |
| | Perfluoroheptanesulfonic acid | 375-92-8 | PFHpS | 23.8 | | | 2.38 |
| | Perfluoroheptanoic acid | 375-85-9 | PFHpA | 25 | | | 2.5 |
| | Perfluorohexanesulfonic acid | 355-46-4 | PFHxS | 22.9 | | | 2.29 |
| | Perfluorohexanoic acid | 307-24-4 | PFHxA | 25 | | | 2.5 |
| | Perfluorononanesulfonic acid | 68259-12-1 | PFNS | 24.1 | | | 2.41 |
| | Perfluorononanoic acid | 375-95-1 | PFNA | 25 | | | 2.5 |
| | Perfluorooctanesulfonamide | 754-91-6 | PFOSA | 25 | | | 2.5 |
| | Perfluorooctanesulfonic acid | 1763-23-1 | PFOS | 23.2 | | | 2.32 |
| | Perfluorooctanoic acid | 335-67-1 | PFOA | 25 | | | 2.5 |
| | Perfluoropentanesulfonic acid | 2706-91-4 | PFPeS | 23.5 | | | 2.35 |
| | Perfluoropentanoic acid | 2706-90-3 | PFPeA | 50 | | | 5 |
| | Perfluorotetradecanoic acid | 376-06-7 | PFTeDA | 25 | | | 2.5 |
| | Perfluorotridecanoic acid | 72629-94-8 | PFTIDA | 25 | | | 2.5 |
| | Perfluoroundecanoic acid | 2058-94-8 | PFUnDA | 25 | | | 2.5 |
| | Perfluoro-3-methoxypropanoic acid | 377-73-1 | PFMPA | 50 | | | 5 |
| | Perfluoro-4-methoxybutanoic acid | 863090-89-5 | PFMBA | 50 | | | 5 |
| | Nonafluoro-3,6-dioxaheptanoic acid | 151722-58-6 | NFDHA | 50 | | | 5 |
| | Perfluoro(2-ethoxyethane)sulfonic acid | 113507-82-7 | PFEESA | 44.5 | | | 4.45 |
| 2-(N-methylperfluoro-1-octanesulfonamido)- ethanol | 24448-09-7 | NMePFOSAE | 250 | 25 | | | |
| N-methylperfluoro-1-octanesulfonamide | 31506-32-8 | NMePFOSA | 25 | 2.5 | | | |
| 2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol | 1691-99-2 | NEtPFOSAE | 250 | 25 | | | |
| N-ethylperfluoro-1-octanesulfonamide | 4151-50-2 | NEtPFOSA | 25 | 2.5 | | | |
| Native PFAS Intermediate B | 3-Perfluoropropylpropanoic acid | 763051-92-9 | 3:3 FTCA | 100 | 0.625 | 5 | 12.5 |
| | 3-Perfluoropentylpropanoic acid | 756426-58-1 | 5:3 FTCA | 500 | | | 62.5 |
| | 3-Perfluoroheptylpropanoic acid | 919005-14-4 | 7:3 FTCA | 500 | | | 62.5 |
| Mass-Labelled PFAS Extraction Standard Solution/ Mixture-ES | Perfluoro-n-[¹³ C ₄]butanoic acid | STL00992 | ¹³ C ₄ -PFBA | 2000 | 0.025 | 5 | 10 |
| | Perfluoro-n-[¹³ C ₅]pentanoic acid | STL01893 | ¹³ C ₅ -PFPeA | 1000 | | | 5 |
| | Perfluoro-n-[1,2,3,4,6- ¹³ C ₅]hexanoic acid | STL02577 | ¹³ C ₅ -PFHxA | 500 | | | 2.5 |
| | Perfluoro-n-[1,2,3,4- ¹³ C ₄]heptanoic acid | STL01892 | ¹³ C ₄ -PFHpA | 500 | | | 2.5 |
| | Perfluoro-n-[¹³ C ₈]octanoic acid | STL01052 | ¹³ C ₈ -PFOA | 500 | | | 2.5 |
| | Perfluoro-n-[¹³ C ₉]nonanoic acid | STL02578 | ¹³ C ₉ -PFNA | 250 | | | 1.25 |
| | Perfluoro-n-[1,2,3,4,5,6- ¹³ C ₆]decanoic acid | STL02579 | ¹³ C ₆ -PFDA | 250 | | | 1.25 |
| | Perfluoro-n-[1,2,3,4,5,6,7- ¹³ C ₇]undecanoic acid | STL02580 | ¹³ C ₇ -PFUnA | 250 | | | 1.25 |
| | Perfluoro-n-[1,2- ¹³ C ₂]dodecanoic acid | STL02703 | ¹³ C ₂ -PFDDoA | 250 | | | 1.25 |
| | Perfluoro-n-[1,2- ¹³ C ₂]tetradecanoic acid | STL02116 | ¹³ C ₂ -PFTeDA | 250 | | | 1.25 |
| | Perfluoro-1-[2,3,4- ¹³ C ₃]butanesulfonic acid | STL02337 | ¹³ C ₃ -PFBS | 466 | | | 2.33 |
| | Perfluoro-1-[1,2,3- ¹³ C ₃]hexanesulfonic acid | STL02581 | ¹³ C ₃ -PFHxS | 474 | | | 2.37 |
| | Perfluoro-1-[¹³ C ₈]octanesulfonic acid | STL01054 | ¹³ C ₈ -PFOS | 479 | | | 2.4 |
| | Perfluoro-1-[¹³ C ₈]octanesulfonamide | STL01056 | ¹³ C ₈ -PFOSA | 500 | | | 2.5 |
| | N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid | STL02118 | D3-NMeFOSAA | 1000 | | | 5 |
| | N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid | STL02117 | D5-NEtFOSAA | 1000 | | | 5 |
| | 1H,1H,2H,2H-Perfluoro-1-[1,2- ¹³ C ₂]hexan sulfonic acid | STL02395 | ¹³ C ₂ -4:2FTS | 938 | | | 4.69 |
| | 1H,1H,2H,2H-Perfluoro-1-[1,2- ¹³ C ₂]octanesulfonic acid | STL02279 | ¹³ C ₂ -6:2FTS | 951 | | | 4.76 |
| | 1H,1H,2H,2H-Perfluoro-1-[1,2- ¹³ C ₂]decanesulfonic acid | STL02280 | ¹³ C ₂ -8:2FTS | 960 | | | 4.8 |
| | Tetrafluoro-2-heptafluoropropoxy- ¹³ C ₃ -propanoic acid | STL02255 | ¹³ C ₃ -HFPODA | 2000 | | | 10 |
| | N-methyl-d7-perfluorooctanesulfonamidoethanol | STL02277 | D7-NMeFOSE | 5000 | | | 25 |
| | N-ethyl-d9-perfluorooctanesulfonamidoethanol | STL02278 | D9-NEtFOSE | 5000 | | | 25 |
| | N-ethyl-d5-perfluoro-1-octanesulfonamide | STL02704 | D5-NEtFOSA | 500 | | | 5 |
| N-methyl-d3-perfluoro-1-octanesulfonamide | STL02705 | D3-NMeFOSA | 500 | 5 | | | |
| Mass-Labelled PFAS Injection Standard Solution/ Mixture-IS | Perfluoro-n-[2,3,4- ¹³ C ₃]butanoic acid | STL02680 | ¹³ C ₃ -PFBA | 1000 | 0.025 | 5 | 5 |
| | Perfluoro-n-[1,2,3,4- ¹³ C ₄]octanoic acid | STL00990 | ¹³ C ₄ -PFOA | 500 | | | 2.5 |
| | Perfluoro-n-[1,2- ¹³ C ₂]decanoic acid | STL00996 | ¹³ C ₂ -PFDA | 250 | | | 1.25 |
| | Perfluoro-n-[1,2,3,4- ¹³ C ₄]octanesulfonic acid | STL00991 | ¹³ C ₄ -PFOS | 479 | | | 2.4 |
| | Perfluoro-n-[1,2,3,4,5- ¹³ C ₅]nonanoic acid | STL00995 | ¹³ C ₅ -PFNA | 250 | | | 1.25 |
| | Perfluoro-n-[1,2- ¹³ C ₂]hexanoic acid | STL00993 | ¹³ C ₂ -PFHxA | 500 | | | 2.5 |
| | Perfluoro-1-hexane[¹⁸ O ₂]sulfonic acid | STL00994 | ¹⁸ O ₂ -PFHxS | 474 | | | 2.37 |

* Bring to final volume using methanol with 4% water, 1% ammonium hydroxide, and 0.625% acetic acid

| 1633 Labeled Ampulated Standards | | | | | | |
|--|------------|----------------|--|----------|--------------------------|---------------|
| Ampulated Solution Name | Vendor | Catalog Number | Analyte | CAS# | Acronym | Conc. (ng/mL) |
| Mass-Labelled PFAS Extraction Standard Solution/Mixture - ES | Wellington | MPFACHIFES | Perfluoro-n-[¹³ C4]butanoic acid | STL00992 | ¹³ C4-PFBA | 2000 |
| | | | Perfluoro-n-[¹³ C5]pentanoic acid | STL01893 | ¹³ C5-PFPeA | 1000 |
| | | | Perfluoro-n-[1,2,3,4,6- ¹³ C5]hexanoic acid | STL02577 | ¹³ C5 -PFHxA | 500 |
| | | | Perfluoro-n-[1,2,3,4- ¹³ C4]heptanoic acid | STL01892 | ¹³ C4-PFHpA | 500 |
| | | | Perfluoro-n-[¹³ C8]octanoic acid | STL01052 | ¹³ C8-PFOA | 500 |
| | | | Perfluoro-n-[¹³ C9]nonanoic acid | STL02578 | ¹³ C9-PFNA | 250 |
| | | | Perfluoro-n-[1,2,3,4,5,6- ¹³ C6]decanoic acid | STL02579 | ¹³ C6-PFDA | 250 |
| | | | Perfluoro-n-[1,2,3,4,5,6,7- ¹³ C7]undecanoic acid | STL02580 | ¹³ C7-PFUxA | 250 |
| | | | Perfluoro-n-[1,2- ¹³ C2]dodecanoic acid | STL02703 | ¹³ C2-PFDoA | 250 |
| | | | Perfluoro-n-[1,2- ¹³ C2]tetradecanoic acid | STL02116 | ¹³ C2-PFTeDA | 250 |
| | | | Perfluoro-1-[2,3,4- ¹³ C3]butanesulfonic acid | STL02337 | ¹³ C3-PFBS | 466 |
| | | | Perfluoro-1-[1,2,3- ¹³ C3]hexanesulfonic acid | STL02581 | ¹³ C3-PFHxS | 474 |
| | | | Perfluoro-1-[¹³ C8]octanesulfonic acid | STL01054 | ¹³ C8-PFOS | 479 |
| | | | Perfluoro-1-[¹³ C8]octanesulfonamide | STL01056 | ¹³ C8 -PFOSA | 500 |
| | | | N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid | STL02118 | D3-NMeFOSAA | 1000 |
| | | | N-ethyl-d5-perfluoro-1-octanesulfonamidoacetic acid | STL02117 | D5-NEIFOSAA | 1000 |
| | | | 1H,1H,2H,2H-Perfluoro-1-[1,2- ¹³ C2]hexan sulfonic acid | STL02395 | ¹³ C2-4:2FTS | 938 |
| | | | 1H,1H,2H,2H-Perfluoro-1-[1,2- ¹³ C2]octanesulfonic acid | STL02279 | ¹³ C2-6:2FTS | 951 |
| | | | 1H,1H,2H,2H-Perfluoro-1-[1,2- ¹³ C2]decanesulfonic acid | STL02280 | ¹³ C2-8:2FTS | 960 |
| | | | Tetrafluoro-2-heptafluoropropoxy- ¹³ C3-propanoic acid | STL02255 | ¹³ C3-HFPO-DA | 2000 |
| | | | N-methyl-d7-perfluorooctanesulfonamidoethanol | STL02277 | D7-NMeFOSE | 5000 |
| | | | N-ethyl-d9-perfluorooctanesulfonamidoethanol | STL02278 | D9-NEIFOSE | 5000 |
| | | | N-ethyl-d5-perfluoro-1-octanesulfonamide | STL02704 | D5-NEIFOSA | 500 |
| N-methyl-d3-perfluoro-1-octanesulfonamide | STL02705 | D3-NMeFOSA | 500 | | | |
| Mass-Labelled PFAS Injection Standard Solution/Mixture - IS | Wellington | MPFACHIFIS | Perfluoro-n-[2,3,4- ¹³ C3]butanoic acid | STL02680 | ¹³ C3-PFBA | 1000 |
| | | | Perfluoro-n-[1,2,3,4- ¹³ C4]octanoic acid | STL00990 | ¹³ C4-PFOA | 500 |
| | | | Perfluoro-n-[1,2- ¹³ C2]decanoic acid | STL00996 | ¹³ C2-PFDA | 250 |
| | | | Perfluoro-n-[1,2,3,4- ¹³ C4]octanesulfonic acid | STL00991 | ¹³ C4-PFOS | 479 |
| | | | Perfluoro-n-[1,2,3,4,5- ¹³ C5] nonanoic acid | STL00995 | ¹³ C5-PFNA | 250 |
| | | | Perfluoro-n-[1,2- ¹³ C2]hexanoic acid | STL00993 | ¹³ C2-PFHxA | 500 |
| | | | Perfluoro-1-hexane[¹⁸ O2]sulfonic acid | STL00994 | ¹⁸ O2-PFHxS | 474 |

Attachment 2

| |
|--|
| PFAS Injection Standards/Extraction Standards/Native Compounds |
|--|

Injection Standards

| Inj Std | Internal Standard/Injection Standard |
|-------------|--------------------------------------|
| I13C3-PFBA | 13C3-PFBA |
| I13C2-PFHxA | 13C2-PFHxA |
| I13C4-PFOA | 13C4-PFOA |
| I13C5-PFNA | 13C5-PFNA |
| I13C2-PFDA | 13C2-PFDA |
| I18O2-PFHxS | 18O2-PFHxS |
| I13C4-PFOS | 13C4-PFOS |

Extraction Standards

| Extraction Standard | Internal Standard |
|---------------------|-------------------|
| E13C4-PFBA | 13C3-PFBA |
| E13C5-PFPeA | 13C2-PFHxA |
| E13C5-PFHxA | |
| E13C4-PFHpA | |
| E13C3-HFPO-DA | |
| E13C8-PFOA | 13C4-PFOA |
| E13C9-PFNA | 13C5-PFNA |
| E13C6-PFDA | 13C2-PFDA |
| E13C7-PFUnA | |
| E13C2-PFDoA | |
| E13C2-PFTeDA | |
| E13C3-PFBS | 18O2-PFHxS |
| E13C3-PFHxS | |
| E13C2-4:2-FTS | |
| E13C2-6:2-FTS | |
| E13C2-8:2-FTS | |

| Extraction Standard | Internal Standard |
|---------------------|-------------------|
| E13C8-PFOS | 13C4-PFOS |
| E13C8-PFOA | |
| Ed3-NMeFOA | |
| Ed5-NEtFOA | |
| Ed3-NMeFOAA | |
| Ed7-NMeFOSE | |
| Ed9-NEtFOSE | |

Native PFAS Compounds

| Native | Extraction Standard |
|---------|-----------------------------------|
| PFBA | 13C4-PFBA |
| PFPeA | 13C5-PFPeA |
| 3:3FTCA | |
| PFMPA | |
| PFMBA | |
| PFHxA | 13C5-PFHxA |
| NFDHA | |
| 5:3FTCA | |
| 7:3FTCA | |
| PFEESA | |
| PFHpA | 13C4-PFHpA |
| PFOA | 13C8-PFOA |
| PFNA | 13C9-PFNA |
| PFDA | 13C6-PFDA |
| PFUnA | 13C7-PFUnA |
| PFDoA | 13C2-PFDoA |
| PFTTrDA | Avg 13C2-PFTeDA and 13C2-PFDoA |
| PFTeDA | 13C2-PFTeDA |
| PFBS | 13C3-PFBS |
| PFPeS | 13C3-PFHxS |
| PFHxS | |
| PFHpS | 13C8-PFOS |
| PFOS | |
| PFNS | |
| PFDS | |
| PFDoS | |

| Native | Extraction Standard |
|---------------|----------------------------|
| 4:2-FTS | 13C2-4:2-FTS |
| 6:2-FTS | 13C2-6:2-FTS |
| 8:2-FTS | 13C2-8:2-FTS |
| PFOSA | 13C8-PFOSA |
| NMeFOSA | D3-NMeFOSA |
| NEtFOSA | D5-NEtFOSA |
| NMeFOSAA | D3-NMeFOSAA |
| NEtFOSAA | D5-N-EtFOSAA |
| NMeFOSE | D7-NMeFOSE |
| NEtFOSE | D9-NEtFOSE |
| HFPO-DA | 13C3-HFPO-DA |
| DONA | |
| 9Cl-PF3ONS | |
| 11Cl-PF3OUdS | |

| 1633 Native Ampulated Standards | | | | | | |
|---|------------|----------------|--|-------------|--------------|---------------|
| Ampulated Solution Name | Vendor | Catalog Number | Analyte | CAS# | Acronym | Conc. (ng/mL) |
| Native Replacement PFAS Solution/Mixture | Wellington | PFAC-MXF | 11-Chloroicosafuoro-3-oxaundecane-1-sulfonic acid | 763051-92-9 | 11Cl-PF3OUdS | 1890 |
| | | | 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid | 756426-58-1 | 9Cl-PF3ONS | 1870 |
| | | | 4,8-dioxa-3H-Perfluorononanoic acid | 919005-14-4 | DONA | 1890 |
| | | | Perfluoro(2-propoxypropanoic) acid | 13252-13-6 | HFPODA | 2000 |
| Native PFAS Solution/Mixture | Wellington | PFAC-MXH | 1H,1H,2H,2H perfluorodecanesulfonic acid | 39108-34-4 | 8:2-FTS | 3840 |
| | | | 1H,1H,2H,2H perfluorohexanesulfonic acid | 757124-72-4 | 4:2-FTS | 3750 |
| | | | 1H,1H,2H,2H perfluorooctanesulfonic acid | 27619-97-2 | 6:2-FTS | 3800 |
| | | | N-ethylperfluorooctanesulfonamidoacetic acid | 2991-50-6 | NEtFOSAA | 1000 |
| | | | N-methylperfluorooctanesulfonamidoacetic acid | 2355-31-9 | NMeFOSAA | 1000 |
| | | | Perfluorobutanesulfonic acid | 375-73-5 | PFBS | 887 |
| | | | Perfluorobutanoic acid | 375-22-4 | PFBA | 4000 |
| | | | Perfluorodecanesulfonic acid | 335-77-3 | PFDS | 965 |
| | | | Perfluorodecanoic acid | 335-76-2 | PFDA | 1000 |
| | | | Perfluorododecanesulfonic acid | 79780-39-5 | PFDoDS | 970 |
| | | | Perfluorododecanoic acid | 307-55-1 | PFDoDA | 1000 |
| | | | Perfluoroheptanesulfonic acid | 375-92-8 | PFHpS | 953 |
| | | | Perfluoroheptanoic acid | 375-85-9 | PFHpA | 1000 |
| | | | Perfluorohexanesulfonic acid | 355-46-4 | PFHxS | 914 |
| | | | Perfluorohexanoic acid | 307-24-4 | PFHxA | 1000 |
| | | | Perfluorononanesulfonic acid | 68259-12-1 | PFNS | 962 |
| | | | Perfluorononanoic acid | 375-95-1 | PFNA | 1000 |
| | | | Perfluorooctanesulfonamide | 754-91-6 | PFOSA | 1000 |
| | | | Perfluorooctanesulfonic acid | 1763-23-1 | PFOS | 928 |
| | | | Perfluorooctanoic acid | 335-67-1 | PFOA | 1000 |
| | | | Perfluoropentanesulfonic acid | 2706-91-4 | PFPeS | 941 |
| | | | Perfluoropentanoic acid | 2706-90-3 | PFPeA | 2000 |
| | | | Perfluorotetradecanoic acid | 376-06-7 | PFTeDA | 1000 |
| | | | Perfluorotridecanoic acid | 72629-94-8 | PFTrDA | 1000 |
| Perfluoroundecanoic acid | 2058-94-8 | PFUnDA | 1000 | | | |
| Native Perfluoroalkyl Ether Carboxylic Acids and Sulfonate Solution/Mixture | Wellington | PFAC-MXG | Perfluoro-3-methoxypropanoic acid | 377-73-1 | PFMPA | 2000 |
| | | | Perfluoro-4-methoxybutanoic acid | 863090-89-5 | PFMBA | 2000 |
| | | | Nonafluoro-3,6-dioxaheptanoic acid | 151722-58-6 | NFDHA | 2000 |
| | | | Perfluoro(2-ethoxyethane)sulfonic acid | 113507-82-7 | PFEESA | 1780 |
| Native N-NMe/EtFOSA & N-Nme/EtFOSE Solution/Mixture | Wellington | PFAC-MXI | 2-(N-methylperfluoro-1-octanesulfonamido)- ethanol | 24448-09-7 | NMePFOSAE | 10000 |
| | | | N-methylperfluoro-1-octanesulfonamide | 31506-32-8 | NMePFOSA | 1000 |
| | | | 2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol | 1691-99-2 | NEtPFOSAE | 10000 |
| | | | N-ethylperfluoro-1-octanesulfonamide | 4151-50-2 | NEtPFOSA | 1000 |
| Native X:3 Fluorotelomer Carboxylic Acid Solution/Mixture | Wellington | PFAC-MXJ | 3-Perfluoropropylpropanoic acid | 763051-92-9 | 3:3 FTCA | 4000 |
| | | | 3-Perfluoropentylpropanoic acid | 756426-58-1 | 5:3 FTCA | 20000 |
| | | | 3-Perfluoroheptylpropanoic acid | 919005-14-4 | 7:3 FTCA | 20000 |
| | Wellington | T-PFOA | Technical Ammonium, Perfluorooctanoate (Technical Grade) | 95328-99-7 | T-PFOA | 50000 |
| | | | Perfluorooctanoic acid | 335-67-1 | PFOA | 50000 |
| | Cambridge | ULM-11036-S | 2-(N-ethylperfluoro-1-octanesulfonamido) ethanol | 1691-99-2 | NEtPFOSAE | 50000 |
| | Cambridge | ULM-11034-S | 2-(N-methylperfluoro-1-octanesulfonamido) ethanol | 24448-09-7 | NMePFOSAE | 50000 |
| | Cambridge | ULM-10780-S | N-ethylperfluoro-1-octanesulfonamide | 4151-50-2 | NEtPFOSA | 100000 |
| | Cambridge | ULM-10779-S | N-methylperfluoro-1-octanesulfonamide | 31506-32-8 | NMePFOSA | 100000 |
| | Cambridge | ULM-10977-S | Perfluorooctanesulfonamide | 754-91-6 | PFOSA | 50000 |
| | Wellington | ipPFNA | Perfluoro-7-methyloctanoic acid | 15899-31-7 | PF7MOA | 50000 |
| | Wellington | PFNA | Perfluorononanoic acid | 375-95-1 | PFNA | 50000 |

| Acquisition Method | Mass Spectrometer Method Properties | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|---|--|--------------|--------------|----------|--------|-----------|------|----|---------|---------|------|----|--------|--------|-----------|--|--|--|--|----|--------|--------|--|--------------|----------|-------|-------|------|----|---------|---------|------|----|--------|--------|-----------|--|--|--|--|----|--------|--------|--|--------------|----------|-------|-------|------|----|---------|---------|------|----|--------|--------|-----------|--|--|--|--|----|--------|--------|
| EPAL633_DOD Mass Spec 10.500 min Period 10.500 min -MRM Integrated Valve Sciex IC System Equilibrate Injection | <p>Period 1: -----</p> <p>Scans in Period: 1050 Min. Dwell Time: 3 ms Max. Dwell Time: 250 ms Relative Start Time: 0.00 msec Scheduled Ionization: Off Experiments in Period: 1 Use target Cycle Time: No Target Cycle Time: N/A</p> <p>Period 1 Experiment 1: -----</p> <p>MRM (MRM) Scan Type: Yes Scheduled MRM: Yes Polarity: Negative Scan Mode: N/A Ion Source: Turbo Spray sMRM Q1/Q3 Resolution: No MRM detection window: 60 sec Target Scan Time: 0.6000 sec Resolution Q1: Unit Resolution Q3: Unit Intensity Thres.: 0.00 cps Settling Time: 0.0000 msec MR Pause: 5.0070 msec MCA: No Step Size: 0.00 Da</p> <table border="1"> <thead> <tr> <th>Q1 Mass (Da)</th> <th>Q3 Mass (Da)</th> <th>RT (min)</th> <th>Parar</th> <th>Start</th> <th>Stop</th> <th>ID</th> </tr> </thead> <tbody> <tr> <td>216.000</td> <td>172.000</td> <td>3.88</td> <td>DF</td> <td>-40.00</td> <td>-40.00</td> <td>13C3-PFEA</td> </tr> <tr> <td></td> <td></td> <td></td> <td></td> <td>CE</td> <td>-14.00</td> <td>-14.00</td> </tr> <tr> <td>Q1 Mass (Da) <th>Q3 Mass (Da)</th> <th>RT (min)</th> <th>Parar</th> <th>Start</th> <th>Stop</th> <th>ID</th> </td></tr> <tr> <td>217.000</td> <td>172.000</td> <td>3.88</td> <td>DF</td> <td>-40.00</td> <td>-40.00</td> <td>13C4-PFEA</td> </tr> <tr> <td></td> <td></td> <td></td> <td></td> <td>CE</td> <td>-14.00</td> <td>-14.00</td> </tr> <tr> <td>Q1 Mass (Da) <th>Q3 Mass (Da)</th> <th>RT (min)</th> <th>Parar</th> <th>Start</th> <th>Stop</th> <th>ID</th> </td></tr> <tr> <td>268.000</td> <td>223.000</td> <td>4.44</td> <td>DF</td> <td>-40.00</td> <td>-40.00</td> <td>13C5-PFEa</td> </tr> <tr> <td></td> <td></td> <td></td> <td></td> <td>CE</td> <td>-14.00</td> <td>-14.00</td> </tr> </tbody> </table> | Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | 216.000 | 172.000 | 3.88 | DF | -40.00 | -40.00 | 13C3-PFEA | | | | | CE | -14.00 | -14.00 | Q1 Mass (Da) <th>Q3 Mass (Da)</th> <th>RT (min)</th> <th>Parar</th> <th>Start</th> <th>Stop</th> <th>ID</th> | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | 217.000 | 172.000 | 3.88 | DF | -40.00 | -40.00 | 13C4-PFEA | | | | | CE | -14.00 | -14.00 | Q1 Mass (Da) <th>Q3 Mass (Da)</th> <th>RT (min)</th> <th>Parar</th> <th>Start</th> <th>Stop</th> <th>ID</th> | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | 268.000 | 223.000 | 4.44 | DF | -40.00 | -40.00 | 13C5-PFEa | | | | | CE | -14.00 | -14.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 216.000 | 172.000 | 3.88 | DF | -40.00 | -40.00 | 13C3-PFEA | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | CE | -14.00 | -14.00 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Q1 Mass (Da) <th>Q3 Mass (Da)</th> <th>RT (min)</th> <th>Parar</th> <th>Start</th> <th>Stop</th> <th>ID</th> | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 217.000 | 172.000 | 3.88 | DF | -40.00 | -40.00 | 13C4-PFEA | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | CE | -14.00 | -14.00 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Q1 Mass (Da) <th>Q3 Mass (Da)</th> <th>RT (min)</th> <th>Parar</th> <th>Start</th> <th>Stop</th> <th>ID</th> | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 268.000 | 223.000 | 4.44 | DF | -40.00 | -40.00 | 13C5-PFEa | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | CE | -14.00 | -14.00 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |

Attachment 3

| | | | | | | |
|--------------|--------------|----------|-------|---------|---------|----------------|
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 302.000 | 80.000 | 4.49 | DE | -120.00 | -120.00 | 13C3-PRES |
| | | | | CE | -65.00 | -65.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 329.000 | 81.000 | 4.83 | DE | -100.00 | -100.00 | 13C2-4:2-FTS |
| | | | | CE | -28.00 | -28.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 315.000 | 270.000 | 4.86 | DE | -30.00 | -30.00 | 13C2-PFHxA |
| | | | | CE | -15.00 | -15.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 318.000 | 273.000 | 4.86 | DE | -30.00 | -30.00 | 13C5-PFHxA |
| | | | | CE | -15.00 | -15.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 287.000 | 169.000 | 5.00 | DE | -20.00 | -20.00 | 13C3-HFFODA |
| | | | | CE | -10.00 | -10.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 367.000 | 322.000 | 5.27 | DE | -40.00 | -40.00 | 13C4-PFHxA |
| | | | | CE | -15.00 | -15.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 402.000 | 80.000 | 5.27 | DE | -100.00 | -100.00 | 13C3-PFHxS |
| | | | | CE | -80.00 | -80.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 359.000 | 294.000 | 5.42 | DE | -40.00 | -40.00 | 13C2-6:2 FTUCA |
| | | | | CE | -25.00 | -25.00 |

Attachment 3

| | | | | | | |
|--------------|--------------|----------|-------|---------|--------------|---------------|
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 379.000 | 294.000 | 5.43 | DF | -30.00 | -30.00 | 13C2-6:2 FTCA |
| | | | | CE | -30.00-30.00 | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 429.000 | 81.000 | 5.63 | DF | -100.00 | -100.00 | 13C2-6:2-FTS |
| | | | | CE | -35.00-35.00 | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 415.000 | 370.000 | 5.65 | DF | -50.00 | -50.00 | 13C2-PROA |
| | | | | CE | -16.00-16.00 | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 417.000 | 172.000 | 5.65 | DF | -50.00 | -50.00 | 13C4-PROA |
| | | | | CE | -16.00-16.00 | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 421.000 | 376.000 | 5.65 | DF | -50.00 | -50.00 | 13C8-PROA |
| | | | | CE | -16.00-16.00 | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 503.000 | 99.000 | 5.98 | DF | -100.00 | -100.00 | 13C4-PROS |
| | | | | CE | -100.00 | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 507.000 | 99.000 | 5.98 | DF | -100.00 | -100.00 | 13C8-PROS |
| | | | | CE | -100.00 | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| | | | | | | |

Attachment 3

| | | | | | | |
|--------------|--------------|----------|-------|---------------|---------------------|--------------------------------|
| 472.000 | 427.000 | 5.99 | DE | -50.00 CE | 13C9-PFNA -18.00 | -18.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 459.000 | 394.000 | 6.13 | DF | -50.00 CE | -50.00 | 13C2-8:2 FTUCA -25.00-25.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 479.000 | 394.000 | 6.13 | DF | -35.00 CE | -35.00 | 13C2-8:2 FTCA -25.00-25.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 519.000 | 474.000 | 6.30 | DF | -50.00 CE | -50.00 | 13C6-PFDA -18.00-18.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 515.000 | 470.000 | 6.30 | DF | -50.00 CE | -50.00 | 13C2-PFDA -18.00-18.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 529.000 | 81.000 | 6.31 | DF | -100.00 CE | -100.00 | 13C2-8:2-FTS -42.00-42.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 506.000 | 78.000 | 6.40 | DF | -100.00 CE | -100.00 | 13C8-PFOXA -80.00-80.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 573.000 | 419.000 | 6.40 | DF | -80.00 CE | -80.00 | d3-NMeFOSMA -30.00-30.00 |

Attachment 3

| | | | | | | |
|--------------|--------------|----------|-------|---------|---------|-----------------|
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 565.000 | 520.000 | 6.58 | DF | -70.00 | -70.00 | 13C2-PFUnDA |
| | | | | CE | -19.00 | -19.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 570.000 | 525.000 | 6.58 | DF | -70.00 | -70.00 | 13C7-PFUnDA |
| | | | | CE | -19.00 | -19.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 589.000 | 419.000 | 6.50 | DF | -90.00 | -90.00 | d5-NEtFOSSAA |
| | | | | CE | -30.00 | -30.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 559.000 | 494.000 | 6.70 | DF | -60.00 | -60.00 | 13C2-10:2 FTUCA |
| | | | | CE | -30.00 | -30.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 579.000 | 494.000 | 6.72 | DF | -50.00 | -50.00 | 13C2-10:2 FTCA |
| | | | | CE | -30.00 | -30.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 615.000 | 570.000 | 6.81 | DF | -60.00 | -60.00 | 13C2-PFDoDA |
| | | | | CE | -20.00 | -20.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 623.000 | 59.000 | 6.85 | DF | -50.00 | -50.00 | d7-ANMePFOSAE |
| | | | | CE | -70.00 | -70.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stoç | ID |
| 515.000 | 219.000 | 6.86 | DF | -100.00 | -100.00 | d3-ANMePFOSA |
| | | | | | | |

Attachment 3

CE -37.00 -37.00

Q1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stof ID
 639.000 59.000 7.01 DE -45.00 -45.00 d9-NETPFOSAE
 CE -70.00-70.00

Q1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stof ID
 531.000 219.000 7.03 DE -100.00 -100.00 d5-NETPFOSA
 CE -38.00-38.00

Q1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stof ID
 715.000 670.000 7.21 DE -60.00 -60.00 13C2-PFTeDA
 CE -22.00-22.00

Q1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stof ID
 163.000 119.000 1.83 DE -30.00 -30.00 PPF Acid
 CE -15.00-15.00

Q1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stof ID
 213.000 169.000 3.89 DE -40.00 -40.00 PFPA
 CE -14.00-14.00

Q1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stof ID
 249.000 99.000 4.12 DE -60.00 -60.00 PPFrS
 CE -40.00-40.00

Q1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stof ID
 229.000 85.000 4.17 DE -40.00 -40.00 PFPCA F
 CE -25.00-25.00

Attachment 3

| | | | | | | |
|--------------|--------------|----------|-------|---------|---------|----------|
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 241.000 | 177.000 | 4.49 | DE | -60.00 | -60.00 | 3:3 FTCA |
| | | | | CE | -12.00 | -12.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 263.000 | 219.000 | 4.43 | DE | -40.00 | -40.00 | PFPeA |
| | | | | CE | -14.00 | -14.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 299.000 | 80.000 | 4.49 | DE | -120.00 | -120.00 | PFES |
| | | | | CE | -65.00 | -65.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 279.000 | 85.000 | 4.62 | DE | -40.00 | -40.00 | PFCA A |
| | | | | CE | -20.00 | -20.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 315.000 | 135.000 | 4.71 | DE | -60.00 | -60.00 | PFESA |
| | | | | CE | -30.00 | -30.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 295.000 | 201.000 | 4.84 | DE | -70.00 | -70.00 | PFCA B |
| | | | | CE | -25.00 | -25.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 327.000 | 307.000 | 4.83 | DE | -100.00 | -100.00 | 4:2-FTS |
| | | | | CE | -28.00 | -28.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 313.000 | 269.000 | 4.86 | DE | -30.00 | -30.00 | PFHxA |
| | | | | CE | -15.00 | -15.00 |

Attachment 3

| | | | | | | |
|--------------|--------------|----------|-------|---------|---------|-----------|
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 349.000 | 80.000 | 4.89 | DF | -90.00 | -90.00 | PFPeS |
| | | | | CE | -70.00 | -70.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 285.000 | 169.000 | 5.00 | DF | -20.00 | -20.00 | HFPODA |
| | | | | CE | -10.00 | -10.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 363.000 | 319.000 | 5.27 | DF | -40.00 | -40.00 | PFHpA |
| | | | | CE | -15.00 | -15.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 399.000 | 80.000 | 5.27 | DF | -100.00 | -100.00 | PFHxS |
| | | | | CE | -80.00 | -80.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 377.000 | 251.000 | 5.32 | DF | -40.00 | -40.00 | DONA |
| | | | | CE | -20.00 | -20.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 341.000 | 237.000 | 5.40 | DF | -70.00 | -70.00 | 5:3 FTCA |
| | | | | CE | -20.00 | -20.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 357.000 | 293.000 | 5.42 | DF | -45.00 | -45.00 | 6:2 FTUCA |
| | | | | CE | -25.00 | -25.00 |

Attachment 3

| | | | | | | |
|--------------|--------------|----------|-------|---------|---------|----------|
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 377.000 | 293.000 | 5.44 | DF | -45.00 | -30.00 | 6:2 FTCA |
| | | | | CE | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 461.000 | 381.000 | 5.63 | DF | -70.00 | -40.00 | PFCHS |
| | | | | CE | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 427.000 | 407.000 | 5.62 | DF | -100.00 | -35.00 | 6:2-FTS |
| | | | | CE | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 449.000 | 80.000 | 5.63 | DF | -100.00 | -90.00 | PFHps |
| | | | | CE | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 413.000 | 369.000 | 5.65 | DF | -50.00 | -16.00 | PFCA |
| | | | | CE | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 499.000 | 80.000 | 5.90 | DF | -100.00 | -100.00 | PFCS |
| | | | | CE | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 463.000 | 419.000 | 5.99 | DF | -50.00 | -18.00 | PFNA |
| | | | | CE | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 441.000 | 317.000 | 6.13 | DF | -80.00 | -20.00 | 7:3 FTCA |
| | | | | CE | | |

Attachment 3

| | | | | | | |
|--------------|--------------|----------|-------|---------|---------|------------|
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 457.000 | 393.000 | 6.13 | DE | -50.00 | -25.00 | 8:2 FTUCA |
| | | | | CE | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 477.000 | 393.000 | 6.15 | DE | -45.00 | -30.00 | 8:2 FTCA |
| | | | | CE | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 531.000 | 351.000 | 6.12 | DE | -100.00 | -100.00 | 9Cl-PF3ONS |
| | | | | CE | -38.00 | -38.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 549.000 | 80.000 | 6.28 | DE | -100.00 | -100.00 | PENS |
| | | | | CE | -110.00 | -110.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 513.000 | 469.000 | 6.30 | DE | -50.00 | -50.00 | PFDA |
| | | | | CE | -18.00 | -18.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 527.000 | 507.000 | 6.30 | DE | -100.00 | -100.00 | 8:2-FTS |
| | | | | CE | -42.00 | -42.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 498.000 | 78.000 | 6.40 | DE | -100.00 | -100.00 | PFOGA |
| | | | | CE | -80.00 | -80.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| | | | | | | |

Attachment 3

| | | | | | | | | |
|--------------|--------------|----------|-------|---------|---------|--------------|--------|--------|
| 570.000 | 419.000 | 6.40 | DE | -80.00 | CE | NM6FOSAA | -30.00 | -30.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | |
| 599.000 | 80.000 | 6.54 | DE | -100.00 | -100.00 | PFDS | | |
| | | | | CE | -120.00 | -120.00 | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | |
| 563.000 | 519.000 | 6.58 | DE | -70.00 | -70.00 | PFUnDA | | |
| | | | | CE | -19.00 | -19.00 | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | |
| 584.000 | 419.000 | 6.50 | DE | -90.00 | -90.00 | NEFOSAA | | |
| | | | | CE | -30.00 | -30.00 | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | |
| 557.000 | 493.000 | 6.70 | DE | -70.00 | -70.00 | 10:2 FTUCA | | |
| | | | | CE | -25.00 | -25.00 | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | |
| 631.000 | 451.000 | 6.68 | DE | -100.00 | -100.00 | 11Cl-PF300ds | | |
| | | | | CE | -43.00 | -43.00 | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | |
| 577.000 | 493.000 | 6.72 | DE | -60.00 | -60.00 | 10:2 FTUCA | | |
| | | | | CE | -30.00 | -30.00 | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | |
| 613.000 | 569.000 | 6.99 | DE | -60.00 | -60.00 | PFDoDA | | |
| | | | | CE | -20.00 | -20.00 | | |

Attachment 3

| | | | | | | |
|--------------|--------------|----------|-------|---------|---------|-----------|
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 627.000 | 607.000 | 6.84 | DE | -100.00 | -100.00 | 10:2-FTIS |
| | | | | CE | -47.00 | -47.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 616.000 | 59.000 | 6.85 | DE | -50.00 | -50.00 | NMePFOSAE |
| | | | | CE | -70.00 | -70.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 512.000 | 219.000 | 6.86 | DE | -100.00 | -100.00 | NMePFOSA |
| | | | | CE | -37.00 | -37.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 699.000 | 80.000 | 6.99 | DE | -100.00 | -100.00 | PFDoS |
| | | | | CE | -150.00 | -150.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 630.000 | 59.000 | 7.01 | DE | -45.00 | -45.00 | NEHFOSAE |
| | | | | CE | -70.00 | -70.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 526.000 | 219.000 | 7.03 | DE | -100.00 | -100.00 | NEHFOSA |
| | | | | CE | -38.00 | -38.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 663.000 | 619.000 | 7.03 | DE | -60.00 | -60.00 | PFTrDA |
| | | | | CE | -21.00 | -21.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 713.000 | 669.000 | 7.21 | DE | -60.00 | -60.00 | PFTeDA |
| | | | | | | |

Attachment 3

CE -22.00 -22.00

Q1 Mass (Da) 813.000 Q3 Mass (Da) 769.000 RT (min) 7.51 Parar DE Start Stof ID -100.00 -100.00 PFFxDA CE -25.00 -25.00

Q1 Mass (Da) 913.000 Q3 Mass (Da) 869.000 RT (min) 7.74 Parar DE Start Stof ID -100.00 -100.00 PFODA CE -27.00 -27.00

Q1 Mass (Da) 299.000 Q3 Mass (Da) 99.000 RT (min) 4.50 Parar DE Start Stof ID -100.00 -100.00 PFEES_2 CE -45.00 -45.00

Q1 Mass (Da) 295.000 Q3 Mass (Da) 85.000 RT (min) 4.45 Parar DE Start Stof ID -25.00 -25.00 PFECA B_2 CE -15.00 -15.00

Q1 Mass (Da) 327.000 Q3 Mass (Da) 81.000 RT (min) 4.83 Parar DE Start Stof ID -100.00 -100.00 4:2 FTS_2 CE -50.00 -50.00

Q1 Mass (Da) 313.000 Q3 Mass (Da) 119.000 RT (min) 4.86 Parar DE Start Stof ID -50.00 -50.00 PFFxA_2 CE -31.00 -31.00

Q1 Mass (Da) 349.000 Q3 Mass (Da) 99.000 RT (min) 4.89 Parar DE Start Stof ID -100.00 -100.00 PFFeS_2 CE -50.00 -50.00

Attachment 3

| | | | | | | |
|--------------|--------------|----------|-------|---------|---------|------------|
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 285.000 | 185.000 | 5.00 | DE | -75.00 | -75.00 | HFPODA_2 |
| | | | | CE | -10.00 | -10.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 385.000 | 185.000 | 5.00 | DE | -75.00 | -75.00 | HFPODA_3 |
| | | | | CE | -10.00 | -10.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 363.000 | 169.000 | 5.27 | DE | -60.00 | -60.00 | PFHpA_2 |
| | | | | CE | -25.00 | -25.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 399.000 | 99.000 | 5.27 | DE | -100.00 | -100.00 | PFHxS_2 |
| | | | | CE | -70.00 | -70.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 341.000 | 217.000 | 5.40 | DE | -80.00 | -80.00 | 5:3 FTCA_2 |
| | | | | CE | -20.00 | -20.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 461.000 | 99.000 | 5.63 | DE | -60.00 | -60.00 | PFCHS_2 |
| | | | | CE | -60.00 | -60.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 427.000 | 81.000 | 5.62 | DE | -120.00 | -120.00 | 6:2 FTS_2 |
| | | | | CE | -70.00 | -70.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stof | ID |
| 449.000 | 99.000 | 5.63 | DE | -100.00 | -100.00 | PFHPS_2 |
| | | | | CE | -80.00 | -80.00 |

Attachment 3

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|--------------|--------------|----------|-------|---------|---------|------------|
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 413.000 | 169.000 | 5.65 | DF | -60.00 | -60.00 | PROA_2 |
| | | | | CE | -26.00 | -26.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 499.000 | 99.000 | 5.97 | DF | -100.00 | -100.00 | PFOS_2 |
| | | | | CE | -80.00 | -80.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 463.000 | 219.000 | 5.99 | DF | -60.00 | -60.00 | PFNA_2 |
| | | | | CE | -30.00 | -30.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 549.000 | 99.000 | 6.28 | DF | -100.00 | -100.00 | PFNS_2 |
| | | | | CE | -90.00 | -90.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 513.000 | 219.000 | 6.30 | DF | -50.00 | -50.00 | PFDA_2 |
| | | | | CE | -31.00 | -31.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 527.000 | 81.000 | 6.30 | DF | -100.00 | -100.00 | 8:2 FTS_2 |
| | | | | CE | -80.00 | -80.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 570.000 | 483.000 | 6.40 | DF | -80.00 | -80.00 | NM#FOSAA_2 |
| | | | | CE | -24.00 | -24.00 |

Attachment 3

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|--------------|---------|--------------|---------|----------|------|-------|----|-------|---------|------|---------|----|------------|
| Q1 Mass (Da) | 599.000 | Q3 Mass (Da) | 99.000 | RT (min) | 6.54 | Parar | DF | Start | -100.00 | Stop | -100.00 | ID | PFDS_2 |
| | | | | | | | | CE | -100.00 | | -100.00 | | |
| Q1 Mass (Da) | 563.000 | Q3 Mass (Da) | 269.000 | RT (min) | 6.58 | Parar | DF | Start | -80.00 | Stop | -80.00 | ID | PFHxDA_2 |
| | | | | | | | | CE | -35.00 | | -35.00 | | |
| Q1 Mass (Da) | 584.000 | Q3 Mass (Da) | 526.000 | RT (min) | 6.50 | Parar | DF | Start | -100.00 | Stop | -100.00 | ID | NEHFOGAA_2 |
| | | | | | | | | CE | -30.00 | | -30.00 | | |
| Q1 Mass (Da) | 613.000 | Q3 Mass (Da) | 319.000 | RT (min) | 6.81 | Parar | DF | Start | -60.00 | Stop | -60.00 | ID | PFDoDA_2 |
| | | | | | | | | CE | -38.00 | | -38.00 | | |
| Q1 Mass (Da) | 627.000 | Q3 Mass (Da) | 81.000 | RT (min) | 6.84 | Parar | DF | Start | -120.00 | Stop | -120.00 | ID | 10:2 FTS_2 |
| | | | | | | | | CE | -100.00 | | -100.00 | | |
| Q1 Mass (Da) | 663.000 | Q3 Mass (Da) | 169.000 | RT (min) | 7.03 | Parar | DF | Start | -60.00 | Stop | -60.00 | ID | PFHxDA_2 |
| | | | | | | | | CE | -40.00 | | -40.00 | | |
| Q1 Mass (Da) | 713.000 | Q3 Mass (Da) | 169.000 | RT (min) | 7.21 | Parar | DF | Start | -60.00 | Stop | -60.00 | ID | PFHxDA_2 |
| | | | | | | | | CE | -40.00 | | -40.00 | | |
| Q1 Mass (Da) | 813.000 | Q3 Mass (Da) | 169.000 | RT (min) | 7.51 | Parar | DF | Start | -80.00 | Stop | -80.00 | ID | PFHxDA_2 |
| | | | | | | | | CE | -45.00 | | -45.00 | | |

Attachment 3

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|--------------|---------|--------------|---------|----------|------|----------|--------|-----------------|--|
| Q1 Mass (Da) | 913.000 | Q3 Mass (Da) | 169.000 | RT (min) | 7.74 | Parar DF | Start | Stof ID | |
| | | | | | | | -80.00 | PFODA_2 | |
| | | | | | | | CE | -50.00-50.00 | |
| Q1 Mass (Da) | 179.000 | Q3 Mass (Da) | 85.000 | RT (min) | 2.90 | Parar DF | Start | Stof ID | |
| | | | | | | | -15.00 | PFMOAA | |
| | | | | | | | CE | -15.00-15.00 | |
| Q1 Mass (Da) | 441.000 | Q3 Mass (Da) | 241.000 | RT (min) | 3.92 | Parar DF | Start | Stof ID | |
| | | | | | | | -80.00 | R-PSDA | |
| | | | | | | | CE | -32.00-32.00 | |
| Q1 Mass (Da) | 405.000 | Q3 Mass (Da) | 217.000 | RT (min) | 3.92 | Parar DF | Start | Stof ID | |
| | | | | | | | -60.00 | R-EVE | |
| | | | | | | | CE | -25.00-25.00 | |
| Q1 Mass (Da) | 439.000 | Q3 Mass (Da) | 343.000 | RT (min) | 3.94 | Parar DF | Start | Stof ID | |
| | | | | | | | -80.00 | Hydrolyzed FSDA | |
| | | | | | | | CE | -35.00-35.00 | |
| Q1 Mass (Da) | 229.000 | Q3 Mass (Da) | 185.000 | RT (min) | 4.06 | Parar DF | Start | Stof ID | |
| | | | | | | | -20.00 | PMFA | |
| | | | | | | | CE | -12.00-12.00 | |
| Q1 Mass (Da) | 297.000 | Q3 Mass (Da) | 135.000 | RT (min) | 4.17 | Parar DF | Start | Stof ID | |
| | | | | | | | -80.00 | NVHOS | |
| | | | | | | | CE | -35.00-35.00 | |
| Q1 Mass (Da) | | Q3 Mass (Da) | | RT (min) | | Parar | Start | Stof ID | |

Attachment 3

| | | | | | | | | | |
|--------------|--------------|----------|-------|--------|--------|----------------|--|--|--|
| 245.000 | 85.000 | 4.37 | DE | -10.00 | -10.00 | PFO2HxA | | | |
| | | | | CE | -15.00 | -15.00 | | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | | |
| 279.000 | 235.000 | 4.59 | DF | -10.00 | -10.00 | PEFA | | | |
| | | | | CE | -20.00 | -20.00 | | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | | |
| 311.000 | 85.000 | 4.97 | DF | -20.00 | -20.00 | PFO3OA | | | |
| | | | | CE | -15.00 | -15.00 | | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | | |
| 427.000 | 283.000 | 5.27 | DF | -40.00 | -40.00 | Hydro-EVE Acid | | | |
| | | | | CE | -18.00 | -18.00 | | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | | |
| 397.000 | 217.000 | 5.27 | DF | -80.00 | -80.00 | R-PSDOA | | | |
| | | | | CE | -35.00 | -35.00 | | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | | |
| 463.000 | 263.000 | 5.26 | DF | -80.00 | -80.00 | Hydro-FS Acid | | | |
| | | | | CE | -38.00 | -38.00 | | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | | |
| 379.000 | 185.000 | 5.38 | DF | -35.00 | -35.00 | PFECA-G | | | |
| | | | | CE | -20.00 | -20.00 | | | |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID | | | |
| 377.000 | 84.000 | 5.48 | DF | -20.00 | -20.00 | PFO4DA | | | |
| | | | | CE | -40.00 | -40.00 | | | |

Attachment 3

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|--------------|---------|--------------|---------|----------|------|-------|----|---------|---------|------------|
| Q1 Mass (Da) | 443.000 | Q3 Mass (Da) | 147.000 | RT (min) | 5.53 | Parar | DF | Start | Stoç | ID |
| | | | | | | | | -70.00 | -70.00 | FS Acid |
| | | | | | | | | CE | -32.00 | -32.00 |
| Q1 Mass (Da) | 407.000 | Q3 Mass (Da) | 263.000 | RT (min) | 5.55 | Parar | DF | Start | Stoç | ID |
| | | | | | | | | -40.00 | -40.00 | EVE Acid |
| | | | | | | | | CE | -14.00 | -14.00 |
| Q1 Mass (Da) | 443.000 | Q3 Mass (Da) | 85.000 | RT (min) | 5.93 | Parar | DF | Start | Stoç | ID |
| | | | | | | | | -7.00 | -7.00 | PFO5DA |
| | | | | | | | | CE | -37.00 | -37.00 |
| Q1 Mass (Da) | 175.000 | Q3 Mass (Da) | 97.000 | RT (min) | 1.46 | Parar | DF | Start | Stoç | ID |
| | | | | | | | | -45.00 | -45.00 | MTP |
| | | | | | | | | CE | -22.00 | -22.00 |
| Q1 Mass (Da) | 468.000 | Q3 Mass (Da) | 423.000 | RT (min) | 5.99 | Parar | DF | Start | Stoç | ID |
| | | | | | | | | -50.00 | -50.00 | 13C5-PFNA |
| | | | | | | | | CE | -18.00 | -18.00 |
| Q1 Mass (Da) | 403.000 | Q3 Mass (Da) | 84.000 | RT (min) | 5.27 | Parar | DF | Start | Stoç | ID |
| | | | | | | | | -100.00 | -100.00 | 1802-PFHxS |
| | | | | | | | | CE | -80.00 | -80.00 |
| Q1 Mass (Da) | 263.000 | Q3 Mass (Da) | 69.000 | RT (min) | 4.43 | Parar | DF | Start | Stoç | ID |
| | | | | | | | | -40.00 | -40.00 | PFFeA_2 |
| | | | | | | | | CE | -14.00 | -14.00 |
| Q1 Mass (Da) | 498.000 | Q3 Mass (Da) | 478.000 | RT (min) | 6.40 | Parar | DF | Start | Stoç | ID |
| | | | | | | | | -100.00 | -100.00 | PFO5A_2 |

Attachment 3

CE -80.00 -80.00

Q1 Mass (Da) 512.000 Q3 Mass (Da) 169.000 RT (min) 6.86 Start Stof ID -100.0C -100.00 NMEPFOSA_2
 DE -37.00 -37.00

Q1 Mass (Da) 526.000 Q3 Mass (Da) 169.000 RT (min) 7.03 Start Stof ID -180.0C -180.00 NEHPFOGA_2
 DE -40.00 -40.00

Q1 Mass (Da) 377.000 Q3 Mass (Da) 85.000 RT (min) 5.32 Start Stof ID -40.0C -40.00 DONA_2
 DE -20.00 -20.00

Q1 Mass (Da) 533.000 Q3 Mass (Da) 353.000 RT (min) 6.12 Start Stof ID -100.0C -100.00 9CI-PF3ONS_2
 DE -38.00 -38.00

Q1 Mass (Da) 633.000 Q3 Mass (Da) 453.000 RT (min) 6.68 Start Stof ID -180.0C -180.00 11CI-PF300dS_2
 DE -40.00 -40.00

Q1 Mass (Da) 241.000 Q3 Mass (Da) 117.000 RT (min) 4.49 Start Stof ID -60.0C -60.00 3:3 FTCA_2
 DE -12.00 -12.00

Q1 Mass (Da) 441.000 Q3 Mass (Da) 337.000 RT (min) 6.13 Start Stof ID -80.0C -80.00 7:3 FTCA_2
 DE -20.00 -20.00

Attachment 3

| | | | | | | |
|--------------|--------------|----------|-------|---------|---------|----------------|
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 315.000 | 83.000 | 4.71 | DE | -60.00 | -60.00 | PFESA_2 |
| | | | | CE | -30.00 | -30.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 699.000 | 99.000 | 6.99 | DE | -100.00 | -100.00 | PFDoS_2 |
| | | | | CE | -150.00 | -150.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 318.000 | 120.000 | 4.86 | DE | -180.00 | -180.00 | 13C5-PFHxA_2 |
| | | | | CE | -40.00 | -40.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 302.000 | 99.000 | 4.49 | DE | -120.00 | -120.00 | 13C3-PFES_2 |
| | | | | CE | -65.00 | -65.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 402.000 | 99.000 | 5.27 | DE | -100.00 | -100.00 | 13C3-PFHxS_2 |
| | | | | CE | -80.00 | -80.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 507.000 | 80.000 | 5.98 | DE | -100.00 | -100.00 | 13C8-PFOS_2 |
| | | | | CE | -100.00 | -100.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 329.000 | 309.000 | 4.83 | DE | -100.00 | -100.00 | 13C2-4:2-FTS_2 |
| | | | | CE | -28.00 | -28.00 |
| Q1 Mass (Da) | Q3 Mass (Da) | RT (min) | Parar | Start | Stop | ID |
| 429.000 | 409.000 | 5.63 | DE | -100.00 | -100.00 | 13C2-6:2-FTS_2 |
| | | | | CE | -35.00 | -35.00 |

Attachment 3

Q1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID
 529.000 509.000 6.31 DF -100.00 -100.00 13C2-8:2-FTS_2
 CE -42.00 -42.00

Q1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID
 287.000 185.000 5.00 DF -20.00 -20.00 13C3-HFFODA_2
 CE -10.00 -10.00

Q1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID
 315.000 119.000 4.86 DF -30.00 -30.00 13C2-PFHxA_2
 CE -18.00 -18.00

Q1 Mass (Da) Q3 Mass (Da) RT (min) Parar Start Stop ID
 503.000 80.000 5.98 DF -100.00 -100.00 13C4-PFOS_2
 CE -100.00 -100.00

Parameter Table(Period 1 Experiment 1):

CUR: 35.00
 CAD: 10.00
 IS: -3000.00
 TEM: 350.00
 GS1: 40.00
 GS2: 50.00
 EF -10.00
 CXE -14.00

**WELLINGTON**
LABORATORIESCERTIFICATE OF ANALYSIS
DOCUMENTATION**PFAC-MXC****Native Perfluorinated
Compound Solution/Mixture**

PRODUCT CODE: PFAC-MXC
LOT NUMBER: PFACMXC0617
SOLVENT(S): Methanol / Water (<1%)
DATE PREPARED: (mm/dd/yyyy) 06/14/2017
LAST TESTED: (mm/dd/yyyy) 03/19/2019
EXPIRY DATE: (mm/dd/yyyy) 03/19/2024
RECOMMENDED STORAGE: Store ampoule in a cool, dark place

DESCRIPTION:

PFAC-MXC is a solution/mixture of thirteen native perfluoroalkylcarboxylic acids (C₄-C₁₄, C₁₆, and C₁₈) and eight native perfluoroalkylsulfonates (C₄-C₁₀ and C₁₂). The full name, abbreviation and concentration for each of the components are given in Table A.

The individual perfluoroalkylcarboxylic acids and perfluoroalkylsulfonates all have chemical purities of >98%.

DOCUMENTATION/ DATA ATTACHED:

Table A: Components and Concentrations of the Solution/Mixture
Figure 1: LC/MS Data (SIR)
Figure 2: LC/MS/MS Data (Selected MRM Transitions)

ADDITIONAL INFORMATION:

- See page 2 for further details.
- Contains 4 mole eq. of NaOH to prevent conversion of the carboxylic acids to their respective methyl esters.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

Wellington Laboratories Inc., 345 Southgate Dr. Guelph ON N1G 3M5 CANADA
519-822-2436 • Fax: 519-822-2849 • info@well-labs.com

ATTACHMENT 4

INTENDED USE:

The products prepared by Wellington Laboratories Inc. are for laboratory use only. This certified reference material (CRM) was designed to be used as a standard for the identification and/or quantification of the specific chemical compounds it contains.

HANDLING:

This product should only be used by qualified personnel familiar with its potential hazards and trained in the handling of hazardous chemicals. Due care should be exercised to prevent unnecessary human contact or ingestion. All procedures should be carried out in a well-functioning fume hood and suitable gloves, eye protection, and clothing should be worn at all times. Waste should be disposed of according to national and regional regulations. Safety Data Sheets (SDSs) are available upon request.

SYNTHESIS / CHARACTERIZATION:

Our products are synthesized using single-product unambiguous routes whenever possible. They are then characterized, and their structures and purities confirmed, using a combination of the most relevant techniques, such as NMR, GC/MS, LC/MS/MS, SFC/UV/MS/MS, x-ray crystallography, and melting point. Isotopic purities of mass-labelled compounds are also confirmed using HRGC/HRMS and/or LC/MS/MS.

HOMOGENEITY:

Prior to solution preparation, crystalline material is tested for homogeneity using a variety of techniques (as stated above) and its solubility in a given diluent is taken into consideration. Duplicate solutions of a new product are prepared from the same crystalline lot and, after the addition of an appropriate internal standard, they are compared by GC/MS, LC/MS/MS, and/or SFC/UV/MS/MS. The relative response factors of the analyte of interest in each solution are required to be <5% RSD. New solution lots of existing products, as well as mixtures and calibration solutions, are compared to older lots in a similar manner. This further confirms the homogeneity of the crystalline material as well as the stability and homogeneity of the solutions in the storage containers. In order to maintain the integrity of the assigned value(s), and associated uncertainty, the dilution or injection of a subsample of this product should be performed using calibrated measuring equipment.

UNCERTAINTY:

The maximum combined relative standard uncertainty of our reference standard solutions is calculated using the following equation:

The combined relative standard uncertainty, $u_c(y)$, of a value y and the uncertainty of the independent parameters

x_1, x_2, \dots, x_n on which it depends is:

$$u_c(y(x_1, x_2, \dots, x_n)) = \sqrt{\sum_{i=1}^n u(y, x_i)^2}$$

where x is expressed as a relative standard uncertainty of the individual parameter.

The individual uncertainties taken into account include those associated with weights (calibration of the balance) and volumes (calibration of the volumetric glassware). An expanded maximum combined percent relative uncertainty of $\pm 5\%$ (calculated with a coverage factor of 2 and a level of confidence of 95%) is stated on the Certificate of Analysis for all of our products.

TRACEABILITY:

All reference standard solutions are traceable to specific crystalline lots. The microbalances used for solution preparation are regularly calibrated by an external ISO/IEC 17025 accredited laboratory. In addition, their calibration is verified prior to each weighing using calibrated external weights traceable to an ISO/IEC 17025 accredited laboratory. All volumetric glassware used is calibrated, of Class A tolerance, and traceable to an ISO/IEC 17025 accredited laboratory. For certain products, traceability to international interlaboratory studies has also been established.

EXPIRY DATE / PERIOD OF VALIDITY:

Ongoing stability studies of this product have demonstrated stability in its composition and concentration, until the specified expiry date, in the unopened ampoule. Monitoring for any degradation or change in concentration of the listed analyte(s) is performed on a routine basis.

LIMITED WARRANTY:

At the time of shipment, all products are warranted to be free of defects in material and workmanship and to conform to the stated technical and purity specifications.

QUALITY MANAGEMENT:

This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAI Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A 1226), and ISO 17034 by ANSI-ASQ National Accreditation Board (ANAB; AR-1523).



For additional information or assistance concerning this or any other products from Wellington Laboratories Inc., please visit our website at www.well-labs.com or contact us directly at info@well-labs.com

ATTACHMENT 4

Table A: PFAC-MXC; Components and Concentrations (ng/ml, ± 5% in Methanol / Water (<1%))

| Compound | Abbreviation | Concentration (ng/ml)* | | Peak Assignment in Figure 1 |
|---|--------------|------------------------|--------------|-----------------------------|
| | | As the salt | As the anion | |
| Perfluoro-n-butanoic acid | PFBA | 2000 | | A |
| Perfluoro-n-pentanoic acid | PFPeA | 2000 | | B |
| Perfluoro-n-hexanoic acid | PFHxA | 2000 | | D |
| Perfluoro-n-heptanoic acid | PFHpA | 2000 | | F |
| Perfluoro-n-octanoic acid | PFOA | 2000 | | H |
| Perfluoro-n-nonanoic acid | PFNA | 2000 | | J |
| Perfluoro-n-decanoic acid | PFDA | 2000 | | L |
| Perfluoro-n-undecanoic acid | PFUDA | 2000 | | N |
| Perfluoro-n-dodecanoic acid | PFDoA | 2000 | | P |
| Perfluoro-n-tridecanoic acid | PFTDA | 2000 | | Q |
| Perfluoro-n-tetradecanoic acid | PFTeDA | 2000 | | S |
| Perfluoro-n-hexadecanoic acid | PFHxDA | 2000 | | T |
| Perfluoro-n-octadecanoic acid | PFODA | 2000 | | U |
| Compound | Abbreviation | Concentration (ng/ml)* | | Peak Assignment in Figure 1 |
| | | As the salt | As the anion | |
| Potassium perfluoro-1-butanefluorobutanesulfonate | L-PFBS | 2000 | 1770 | C |
| Sodium perfluoro-1-pentanesulfonate | L-PFPeS | 2000 | 1880 | E |
| Sodium perfluoro-1-hexanesulfonate | L-PFHxS | 2000 | 1890 | G |
| Sodium perfluoro-1-heptanesulfonate | L-PFHpS | 2000 | 1900 | I |
| Sodium perfluoro-1-octanesulfonate | L-PFOS | 2000 | 1910 | K |
| Sodium perfluoro-1-nonanesulfonate | L-PFNS | 2000 | 1920 | M |
| Sodium perfluoro-1-decanesulfonate | L-PFDS | 2000 | 1930 | O |
| Sodium perfluoro-1-dodecanesulfonate | L-PFDoS | 2000 | 1940 | R |

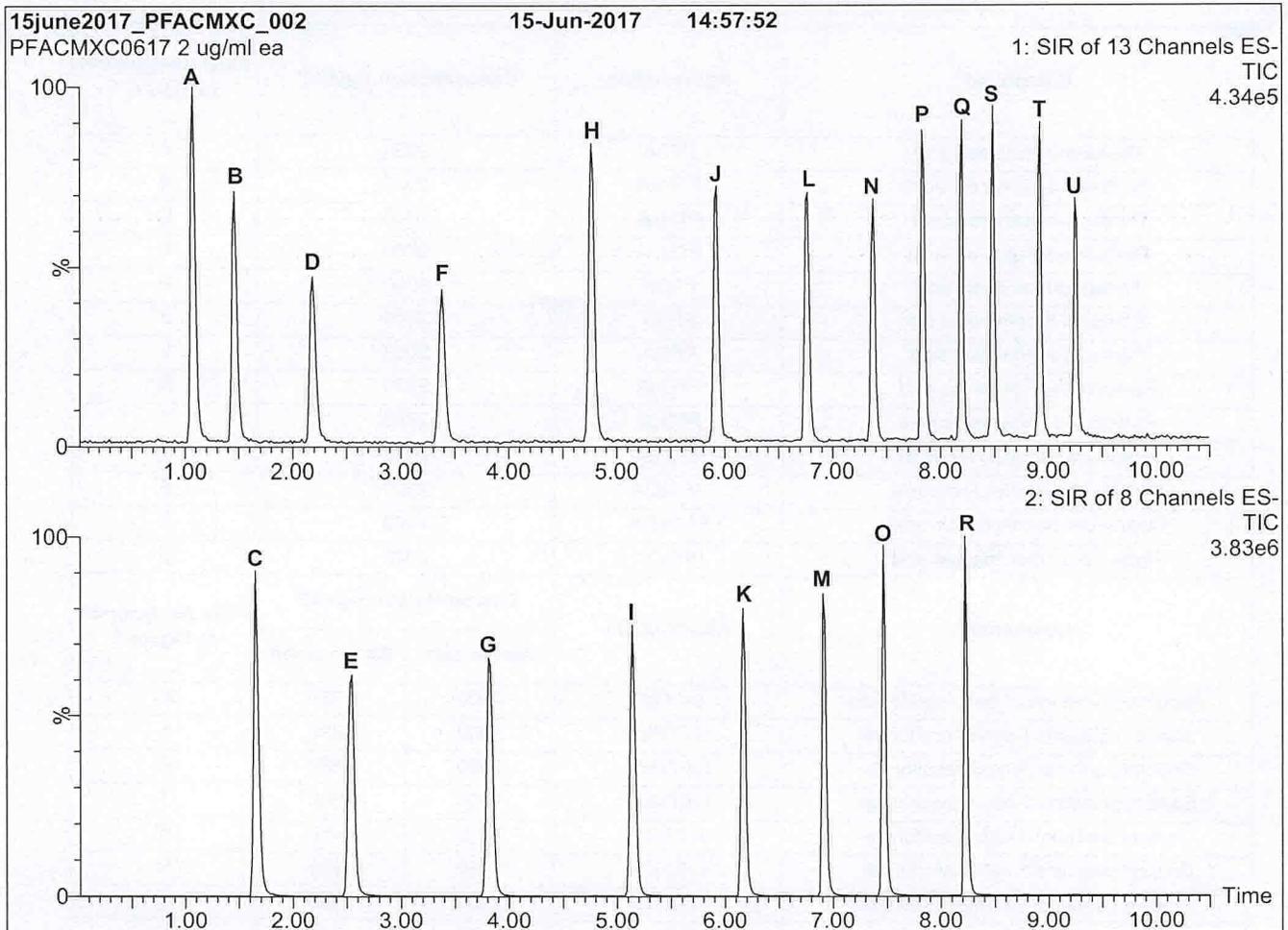
* Concentrations have been rounded to three significant figures.

Certified By: 
 B.G. Chittim, General Manager

Date: 06/06/2019
(mm/dd/yyyy)

ATTACHMENT 4

Figure 1: PFAC-MXC; LC/MS Data (Total Ion Current Chromatogram; SIR)



Conditions for Figure 1:

LC: Waters Acquity Ultra Performance LC
MS: Micromass Quattro *micro* API MS

Chromatographic Conditions

Column: Acquity UPLC BEH Shield RP₁₈
 1.7 μ m, 2.1 x 100 mm

Mobile phase: Gradient
 Start: 50% H₂O / 50% (80:20 MeOH:ACN)
 (both with 10 mM NH₄OAc buffer)
 Ramp to 90% organic over 8 min and hold for 2 min
 before returning to initial conditions in 1 min.

Time: 12 min

Flow: 300 μ l/min

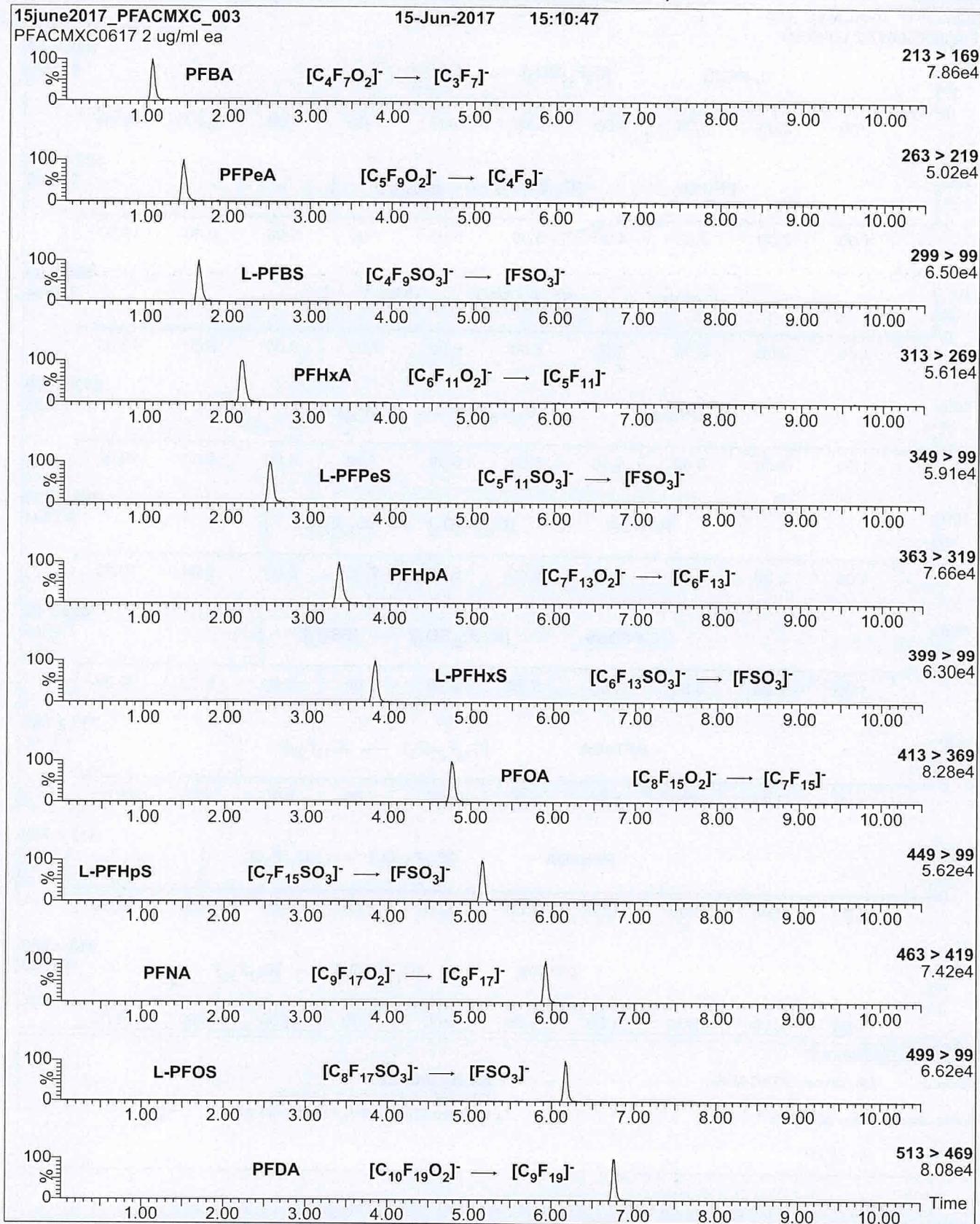
MS Parameters

Experiment: SIR of 21 Channels

Source: Electrospray (negative)
 Capillary Voltage (kV) = 3.00
 Cone Voltage (V) = variable (10-80)
 Cone Gas Flow (l/hr) = 50
 Desolvation Gas Flow (l/hr) = 750

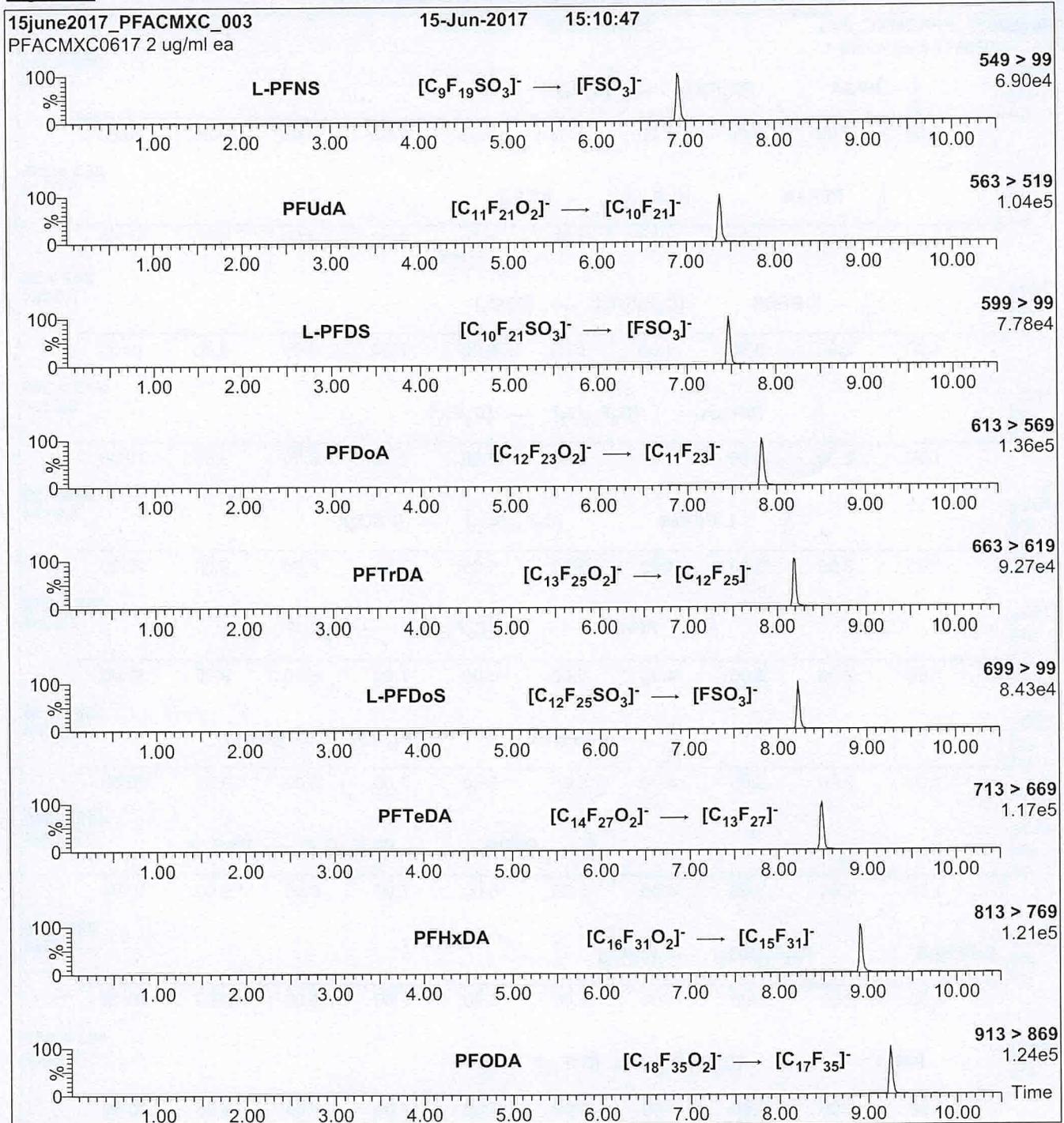
ATTACHMENT 4

Figure 2: PFAC-MXC; LC/MS/MS Data (Selected MRM Transitions)



ATTACHMENT 4

Figure 2: PFAC-MXC; LC/MS/MS Data (Selected MRM Transitions)



Conditions for Figure 2:

Injection: On-column (PFAC-MXC)
 Mobile phase: Same as Figure 1
 Flow: 300 μ l/min

MS Parameters

Collision Gas (mbar) = 3.46e-3
 Collision Energy (eV) = 8-50 (variable)

| Native PFAS Intermediate A | | | | | | | | |
|----------------------------|----------------|--|-------------|--------------|---------------|--------------|----------------------------|--|
| Vendor | Catalog Number | Analyte | CAS# | Acronym | Conc. (ng/mL) | Aliquot (mL) | Final Volume (ml) Methanol | Final Conc. Native PFAS Intermediate A (ng/ml) |
| Wellington | PFAC-MXF | 11-Chloroicosafuoro-3-oxaundecane-1-sulfonic acid | 763051-92-9 | 11Cl-PF3OUdS | 1890 | 0.10 | | 94.5 |
| | | 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid | 756426-58-1 | 9Cl-PF3ONS | 1870 | | | 93.5 |
| | | 4,8-dioxa-3H-Perfluorononanoic acid | 919005-14-4 | DONA | 1890 | | | 94.5 |
| | | Perfluoro(2-propoxypropanoic) acid | 13252-13-6 | HFPODA | 2000 | | | 100 |
| Wellington | PFAC-MXH | 1H,1H,2H,2H perfluorodecanesulfonic acid | 39108-34-4 | 8:2-FTS | 3840 | 0.05 | 2 | 96 |
| | | 1H,1H,2H,2H perfluorohexanesulfonic acid | 757124-72-4 | 4:2-FTS | 3750 | | | 93.8 |
| | | 1H,1H,2H,2H perfluorooctanesulfonic acid | 27619-97-2 | 6:2-FTS | 3800 | | | 95 |
| | | N-ethylperfluorooctanesulfonamidoacetic acid | 2991-50-6 | NEIFOSAA | 1000 | | | 25 |
| | | N-methylperfluorooctanesulfonamidoacetic acid | 2355-31-9 | NMeFOSAA | 1000 | | | 25 |
| | | Perfluorobutanesulfonic acid | 375-73-5 | PFBS | 887 | | | 22.2 |
| | | Perfluorobutanoic acid | 375-22-4 | PFBA | 4000 | | | 100 |
| | | Perfluorodecanesulfonic acid | 335-77-3 | PFDS | 965 | | | 24.1 |
| | | Perfluorodecanoic acid | 335-76-2 | PFDA | 1000 | | | 25 |
| | | Perfluorododecanesulfonic acid | 79780-39-5 | PFDoDS | 970 | | | 24.3 |
| | | Perfluorododecanoic acid | 307-55-1 | PFDoDA | 1000 | | | 25 |
| | | Perfluoroheptanesulfonic acid | 375-92-8 | PFHpS | 953 | | | 23.8 |
| | | Perfluoroheptanoic acid | 375-85-9 | PFHpA | 1000 | | | 25 |
| | | Perfluorohexanesulfonic acid | 355-46-4 | PFHxS | 914 | | | 22.9 |
| | | Perfluorohexanoic acid | 307-24-4 | PFHxA | 1000 | | | 25 |
| | | Perfluoronanesulfonic acid | 68259-12-1 | PFNS | 962 | | | 24.1 |
| | | Perfluoronanoic acid | 375-95-1 | PFNA | 1000 | | | 25 |
| | | Perfluorooctanesulfonamide | 754-91-6 | PFOSA | 1000 | | | 25 |
| | | Perfluorooctanesulfonic acid | 1763-23-1 | PFOS | 928 | | | 23.2 |
| | | Perfluorooctanoic acid | 335-67-1 | PFOA | 1000 | | | 25 |
| | | Perfluoropentanesulfonic acid | 2706-91-4 | PFPeS | 941 | | | 23.5 |
| | | Perfluoropentanoic acid | 2706-90-3 | PFPeA | 2000 | | | 50 |
| | | Perfluorotradecanoic acid | 376-06-7 | PFTeDA | 1000 | | | 25 |
| | | Perfluorotridecanoic acid | 72629-94-8 | PFTrDA | 1000 | | | 25 |
| Perfluoroundecanoic acid | 2058-94-8 | PFUnDA | 1000 | 25 | | | | |
| Wellington | PFAC-MXG | Perfluoro-3-methoxypropanoic acid | 377-73-1 | PFMPA | 2000 | 0.05 | | 50 |
| | | Perfluoro-4-methoxybutanoic acid | 863090-89-5 | PFMBA | 2000 | | | 50 |
| | | Nonafluoro-3,6-dioxaheptanoic acid | 151722-58-6 | NFDHA | 2000 | | | 50 |
| | | Perfluoro(2-ethoxyethane)sulfonic acid | 113507-82-7 | PFEESA | 1780 | | | 44.5 |
| Wellington | PFAC-MXI | 2-(N-methylperfluoro-1-octanesulfonamido)-ethanol | 24448-09-7 | NMePFOSAE | 10000 | 0.05 | | 250 |
| | | N-methylperfluoro-1-octanesulfonamide | 31506-32-8 | NMePFOSA | 1000 | | | 25 |
| | | 2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol | 1691-99-2 | NEiPFOSAE | 10000 | | | 250 |
| | | N-ethylperfluoro-1-octanesulfonamide | 4151-50-2 | NEiPFOSA | 1000 | | | 25 |

| Native PFAS Intermediate B | | | | | | | | |
|----------------------------|----------------|---------------------------------|-------------|----------|---------------|--------------|----------------------------|--|
| Vendor | Catalog Number | Analyte | CAS# | Acronym | Conc. (ug/mL) | Aliquot (mL) | Final Volume (ml) Methanol | Final Conc. Native PFAS Intermediate B (ug/ml) |
| Wellington | PFAC-MXJ | 3-Perfluoropropylpropanoic acid | 763051-92-9 | 3:3 FTCA | 4 | 0.125 | 5 | 100 |
| | | 3-Perfluoropentylpropanoic acid | 756426-58-1 | 5:3 FTCA | 20 | | | 500 |
| | | 3-Perfluoroheptylpropanoic acid | 919005-14-4 | 7:3 FTCA | 20 | | | 500 |

| Working Labeled Extraction Standard Spike* | | | | | | | |
|---|--|------------|--------------------------|---------------|--------------|--------------|---|
| Solution Name | Analyte | CAS# | Acronym | Conc. (ng/mL) | Aliquot (mL) | Final Volume | Final Conc. Working Labeled Extraction Standard Spike (ng/ml) |
| Mass-Labelled PFAS Extraction Standard Solution/Mixture-ES* | Perfluoro-n-[¹³ C4]butanoic acid | STL00992 | ¹³ C4-PFBA | 2000 | 1 | 1 | 2000 |
| | Perfluoro-n-[¹³ C5]pentanoic acid | STL01893 | ¹³ C5-PFPeA | 1000 | | | 1000 |
| | Perfluoro-n-[1,2,3,4,6- ¹³ C5]hexanoic acid | STL02577 | ¹³ C5 -PFHxA | 500 | | | 500 |
| | Perfluoro-n-[1,2,3,4- ¹³ C4]heptanoic acid | STL01892 | ¹³ C4-PFHpA | 500 | | | 500 |
| | Perfluoro-n-[¹³ C8]octanoic acid | STL01052 | ¹³ C8-PFOA | 500 | | | 500 |
| | Perfluoro-n-[¹³ C9]nonanoic acid | STL02578 | ¹³ C9-PFNA | 250 | | | 250 |
| | Perfluoro-n-[1,2,3,4,5,6- ¹³ C6]decanoic acid | STL02579 | ¹³ C6-PFDA | 250 | | | 250 |
| | Perfluoro-n-[1,2,3,4,5,6,7- ¹³ C7]undecanoic acid | STL02580 | ¹³ C7-PFUhA | 250 | | | 250 |
| | Perfluoro-n-[1,2- ¹³ C2]dodecanoic acid | STL02703 | ¹³ C2-PFDoA | 250 | | | 250 |
| | Perfluoro-n-[1,2- ¹³ C2]tetradecanoic acid | STL02116 | ¹³ C2-PFTeDA | 250 | | | 250 |
| | Perfluoro-1-[2,3,4- ¹³ C3]butanesulfonic acid | STL02337 | ¹³ C3-PFBFS | 466 | | | 466 |
| | Perfluoro-1-[1,2,3- ¹³ C3]hexanesulfonic acid | STL02581 | ¹³ C3-PFHxS | 474 | | | 474 |
| | Perfluoro-1-[¹³ C8]octanesulfonic acid | STL01054 | ¹³ C8-PFOS | 479 | | | 479 |
| | Perfluoro-1-[¹³ C8]octanesulfonamide | STL01056 | ¹³ C8 -PFOSA | 500 | | | 500 |
| | N-methyl-d3-perfluoro-1-octanesulfonamido acetic acid | STL02118 | D3-NMeFOSAA | 1000 | | | 1000 |
| | N-ethyl-d5-perfluoro-1-octanesulfonamido acetic acid | STL02117 | D5-NEFOSAA | 1000 | | | 1000 |
| | 1H,1H,2H,2H-Perfluoro-1-[1,2- ¹³ C2]hexan sulfonic acid | STL02395 | ¹³ C2-4:2FTS | 938 | | | 938 |
| | 1H,1H,2H,2H-Perfluoro-1-[1,2- ¹³ C2]octanesulfonic acid | STL02279 | ¹³ C2-6:2FTS | 951 | | | 951 |
| | 1H,1H,2H,2H-Perfluoro-1-[1,2- ¹³ C2]decanesulfonic acid | STL02280 | ¹³ C2-8:2FTS | 960 | | | 960 |
| | Tetrafluoro-2-heptafluoropropoxy- ¹³ C3-propanoic acid | STL02255 | ¹³ C3-HFPO-DA | 2000 | | | 2000 |
| | N-methyl-d7-perfluorooctanesulfonamidoethanol | STL02277 | D7-NMeFOSE | 5000 | | | 5000 |
| | N-ethyl-d9-perfluorooctanesulfonamidoethanol | STL02278 | D9-NEFOSE | 5000 | | | 5000 |
| | N-ethyl-d5-perfluoro-1-octanesulfonamide | STL02704 | D5-NEFOSA | 500 | | | 500 |
| N-methyl-d3-perfluoro-1-octanesulfonamide | STL02705 | D3-NMeFOSA | 500 | 500 | | | |

* Solution used without dilution for spiking. Entered into LIMS as a 1:1 dilution to utilize the standardized naming convention for a working standard. (PFC_1633_SS_XXXXX)

Attachment 8

| Internal Standard Spike | | | | |
|--|--|----------|------------------------|---------------|
| Solution Name | Analyte | CAS# | Acronym | Conc. (ng/mL) |
| Mass-Labelled PFAS Injection Standard Solution/Mixture | Perfluoro-n-[2,3,4- ¹³ C3]butanoic acid | STL02680 | ¹³ C3-PFBA | 1000 |
| | Perfluoro-n-[1,2,3,4- ¹³ C4]octanoic acid | STL00990 | ¹³ C4-PFOA | 500 |
| | Perfluoro-n-[1,2- ¹³ C2]decanoic acid | STL00996 | ¹³ C2-PFDA | 250 |
| | Perfluoro-n-[1,2,3,4- ¹³ C4]octanesulfonic acid | STL00991 | ¹³ C4-PFOS | 479 |
| | Perfluoro-n-[1,2,3,4,5- ¹³ C5] nonanoic acid | STL00995 | ¹³ C5-PFNA | 250 |
| | Perfluoro-n-[1,2- ¹³ C2]hexanoic acid | STL00993 | ¹³ C2-PFHxA | 500 |
| | Perfluoro-1-hexane[¹⁸ O2]sulfonic acid | STL00994 | ¹⁸ O2-PFHxS | 474 |

| Native 1633 Mid-Level Spike | | | | | | | | |
|-----------------------------|----------------|--|-------------|--------------|---------------|--------------|--------------|---|
| Vendor | Catalog Number | Analyte | CAS# | Acronym | Conc. (ng/mL) | Aliquot (mL) | Final Volume | Final Conc. Native 1633 Mid-Level Spike (ng/ml) |
| Wellington | PFAC-MXF | 11-Chloroeicosafuoro-3-oxaundecane-1-sulfonic acid | 763051-92-9 | 11Cl-PF3OUdS | 1890 | 0.625 | | 236.25 |
| | | 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid | 756426-58-1 | 9Cl-PF3ONS | 1870 | | | 233.75 |
| | | 4,8-dioxo-3H-Perfluorononanoic acid | 919005-14-4 | DONA | 1890 | | | 236.25 |
| | | Perfluoro(2-propoxypropanoic) acid | 13252-13-6 | HFPODA | 2000 | | | 250 |
| Wellington | PFAC-MXH | 1H,1H,2H,2H perfluorodecanesulfonic acid | 39108-34-4 | 8:2-FTS | 3840 | 0.313 | 5mL Methanol | 240.38 |
| | | 1H,1H,2H,2H perfluorohexanesulfonic acid | 757124-72-4 | 4:2-FTS | 3750 | | | 234.75 |
| | | 1H,1H,2H,2H perfluorooctanesulfonic acid | 27619-97-2 | 6:2-FTS | 3800 | | | 237.88 |
| | | N-ethylperfluorooctanesulfonamidoacetic acid | 2991-50-6 | NEIFOSAA | 1000 | | | 62.6 |
| | | N-methylperfluorooctanesulfonamidoacetic acid | 2355-31-9 | NMeFOSAA | 1000 | | | 62.6 |
| | | Perfluorobutanesulfonic acid | 375-73-5 | PFBS | 887 | | | 55.53 |
| | | Perfluorobutanoic acid | 375-22-4 | PFBA | 4000 | | | 250.4 |
| | | Perfluorodecanesulfonic acid | 335-77-3 | PFDS | 965 | | | 60.41 |
| | | Perfluorodecanoic acid | 335-76-2 | PFDA | 1000 | | | 62.6 |
| | | Perfluorododecanesulfonic acid | 79780-39-5 | PFDoDS | 970 | | | 60.72 |
| | | Perfluorododecanoic acid | 307-55-1 | PFDoDA | 1000 | | | 62.6 |
| | | Perfluoroheptanesulfonic acid | 375-92-8 | PFHpS | 953 | | | 59.66 |
| | | Perfluoroheptanoic acid | 375-85-9 | PFHpA | 1000 | | | 62.6 |
| | | Perfluorohexanesulfonic acid | 355-46-4 | PFHxS | 914 | | | 57.22 |
| | | Perfluorohexanoic acid | 307-24-4 | PFHxA | 1000 | | | 62.6 |
| | | Perfluorononanesulfonic acid | 68259-12-1 | PFNS | 962 | | | 60.22 |
| | | Perfluorononanoic acid | 375-95-1 | PFNA | 1000 | | | 62.6 |
| | | Perfluorooctanesulfonamide | 754-91-6 | PFOSA | 1000 | | | 62.6 |
| | | Perfluorooctanesulfonic acid | 1763-23-1 | PFOS | 928 | | | 58.09 |
| | | Perfluorooctanoic acid | 335-67-1 | PFOA | 1000 | | | 62.6 |
| | | Perfluoropentanesulfonic acid | 2706-91-4 | PFPeS | 941 | | | 58.91 |
| | | Perfluoropentanoic acid | 2706-90-3 | PFPeA | 2000 | | | 125.2 |
| | | Perfluorotetradecanoic acid | 376-06-7 | PFTeDA | 1000 | | | 62.6 |
| | | Perfluorotridecanoic acid | 72629-94-8 | PFTrDA | 1000 | | | 62.6 |
| Perfluoroundecanoic acid | 2058-94-8 | PFUnDA | 1000 | 62.6 | | | | |
| Wellington | PFAC-MXG | Perfluoro-3-methoxypropanoic acid | 377-73-1 | PFMPA | 2000 | 0.313 | | 125.2 |
| | | Perfluoro-4-methoxybutanoic acid | 863090-89-5 | PFMBA | 2000 | | | 125.2 |
| | | Nonafluoro-3,6-dioxiheptanoic acid | 151722-58-6 | NFDHA | 2000 | | | 125.2 |
| | | Perfluoro(2-ethoxyethane)sulfonic acid | 113507-82-7 | PFEESA | 1780 | | | 111.4 |
| Wellington | PFAC-MXI | 2-(N-methylperfluoro-1-octanesulfonamido)-ethanol | 24448-09-7 | NMePFOSAE | 10000 | 0.313 | | 626 |
| | | N-methylperfluoro-1-octanesulfonamide | 31506-32-8 | NMePFOSA | 1000 | | | 62.6 |
| | | 2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol | 1691-99-2 | NEIPFOSAE | 10000 | | | 626 |
| | | N-ethylperfluoro-1-octanesulfonamide | 4151-50-2 | NEIPFOSA | 1000 | | | 62.6 |
| Wellington | PFAC-MXJ | 3-Perfluoropropylpropanoic acid | 763051-92-9 | 3:3 FTCA | 4000 | 0.391 | | 312.8 |
| | | 3-Perfluoropentylpropanoic acid | 756426-58-1 | 5:3 FTCA | 20000 | | | 1564 |
| | | 3-Perfluoroheptylpropanoic acid | 919005-14-4 | 7:3 FTCA | 20000 | | | 1564 |

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|  | Always check on-line for validity. | Level:  |
| | Document number: T-PFAS-WI36458 | Work Instruction |
| | Old Reference: | |
| | Version: 3 | Organisation level: 5-Sub-BU |
| Approved by: XL3S Effective Date 25-MAY-2022 | Document users: 5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep | Responsible: 5_EUUSLA_PFAS_Manager |

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Revision Log

| | | |
|--------------------------------------|--|--|
| Revision: | 03 | Effective date: This version |
| Section | Justification | Changes |
| Revision log | Required section | Removed revision logs up to previous version |
| Header | Enhancement | Updated Company name to Eurofins Lancaster Laboratories Environment Testing, LLC |
| Title and references | Enhancement | Update to QSM5.4 |
| Throughout document and attachments. | Enhancement/reflect current procedure. | Added references to PES, PFECA A, PFECA B, PFECA F, 3:3 FTCA, 5:3 FTCA, 7:3 FTCA as needed, updated SPE cartridge and procedure added in envi-carb cleanup |

| | | |
|---|---|--------|
|  | Always check on-line for validity. | Level: |
| | Polyfluorinated Alkyl Substances (PFAS) in | |

| | | | |
|---|--|--|--|
| Document number: T-PFAS-WI36458 | Aqueous Samples by Method 537 Version 1.1 Modified QSM5.4 Table B-15 Using LC/MS/MS | Work Instruction |  |
| Old Reference: | | Organisation level: 5-Sub-BU | |
| Version: 3 | | Document users: 5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep | Responsible: 5_EUUSLA_PFAS_Manager |
| Approved by: XL3S Effective Date 25-MAY-2022 | | | |

| Revision: | 03 | Effective date: <u>This version</u> |
|---------------------|---------------------------|--|
| Section | Justification | Changes |
| <i>Attachment 1</i> | Reflect current procedure | Updated as needed to reflect current practice. |
| <i>Attachment 5</i> | Correction | Added attachment 5 which was omitted in the previous version |

| Revision: | 02 | Effective date: <u>14-FEB-2022</u> |
|------------------|---------------------------|---|
| Section | Justification | Changes |
| Revision log | Required section | Add revision log for version 2 |
| Procedure | Reflect current procedure | Procedure B.10,12,13 volumes updated to 7 ml from 5ml |
| Cross reference | enhancement | Add G-DC-FRM23907 |

Reference

1. Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LCMSMS), EPA 537 Version 1.1, September 2009. Department of Defense Quality System Manual Version 5.4, Table B-15.
2. US EPA Method 537 Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LCMSMS), Version 1.1, September 2009.
3. Standard Test Method for Determination of Perfluorinated Compounds in Soil by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS), ASTM Method D7968, 2014.
4. ISO 25101:2009(E) Water quality - Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) - Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry, March 2009.
5. Method for Trace Level Analysis of C8, C9, C10, C11, and C13 Perfluorocarbon Carboxylic Acids in Water. Karen Risha, John Flaherty, Roice Wille, Warren Buck, Francesco Morandi, and Tsuguhide Isemura. Anal. Chem. 2005, 77, 1503-1508.
6. *Chemical Hygiene Plan*, current version.

Cross Reference

| Document | Document Title | Level: |
|---|---|--------|
|  | Always check on-line for validity. Polyfluorinated Alkyl Substances (PFAS) in | |

| | | | |
|---|--|--|--|
| Document number: T-PFAS-WI36458 | Aqueous Samples by Method 537 Version 1.1 Modified QSM5.4 Table B-15 Using LC/MS/MS | Work Instruction |  |
| Old Reference: | | Organisation level: 5-Sub-BU | |
| Version: 3 | | Document users: 5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep | Responsible: 5_EUUSLA_PFAS_Manager |
| Approved by: XL3S Effective Date 25-MAY-2022 | | | |

| Document | Document Title |
|--------------------------------|--|
| T-PEST-WI9847 | Common Equations Used During Chromatographic Analyses |
| T-PFAS-WI13881 | Standards Management in the PFAS Laboratory |
| QA-SOP11178 | Demonstrations of Capability |
| QA-SOP11892 | Determining Method Detection Limits and Limits of Quantitation |
| G-DC-FRM23907 | Redacted SOPs |

Scope

This method is applicable for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in aqueous samples to include non-potable waters and non-regulatory potable water when directed by the client. The compounds analyzed in this method are listed in the table below. The most current MDLs and LOQs are listed in the LIMS.

| Analyte | Acronym | CAS# |
|--|----------|------------|
| Perfluorobutanesulfonic acid | PFBS | 375-73-5 |
| Perfluorodecanoic acid | PFDA | 335-76-2 |
| Perfluorododecanoic acid | PFDoDA | 307-55-1 |
| Perfluoroheptanoic acid | PFHpA | 375-85-9 |
| Perfluorohexanesulfonic acid | PFHxS | 355-46-4 |
| Perfluorohexanoic acid | PFHxA | 307-24-4 |
| Perfluorononanoic acid | PFNA | 375-95-1 |
| Perfluorooctanesulfonic acid | PFOS | 1763-23-1 |
| Perfluorooctanoic acid | PFOA | 335-67-1 |
| Perfluorotetradecanoic acid | PFTeDA | 376-06-7 |
| Perfluorotridecanoic acid | PFTrDA | 72629-94-8 |
| Perfluoroundecanoic acid | PFUnDA | 2058-94-8 |
| Perfluoro-n-butanoic acid | PFBA | 375-22-4 |
| Perfluoro-n-pentanoic acid | PFPeA | 2706-90-3 |
| 8:2 - Fluorotelomersulfonic acid | 8:2FTS | 39108-34-4 |
| N-methylperfluoro-1-octanesulfonamidoacetic acid | NMeFOSAA | 2355-31-9 |

| | | |
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| Polyfluorinated Alkyl Substances (PFAS) in | | |

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| Document number: T-PFAS-WI36458 |
| Old Reference: |
| Version: 3 |

**Aqueous Samples by Method 537 Version 1.1
Modified QSM5.4 Table B-15 Using LC/MS/MS**

Work Instruction



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| Approved by: XL3S Effective Date 25-MAY-2022 |
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Document users:
**5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst,
6_EUUSLA_PFAS_Data_Reviewers,
6_EUUSLA_PFAS_Management_Team,
6_EUUSLA_PFAS_Sample_Prep**

Organisation level:
5-Sub-BU
Responsible:
5_EUUSLA_PFAS_Manager

| | | |
|---|---------------|---------------|
| N-ethylperfluoro-1-octanesulfonamidoacetic acid | NEtFOSAA | 2991-50-6 |
| 4:2-Fluorotelomersulfonic acid | 4:2-FTS | 757124-72-4 |
| Perfluoropentanesulfonic acid | PFPeS | 2706-91-4 |
| 6:2-Fluorotelomersulfonic acid | 6:2-FTS | 27619-97-2 |
| Perfluoroheptanesulfonic acid | PFHpS | 375-92-8 |
| Perfluorononanesulfonic acid | PFNS | 68259-12-1 |
| Perfluorodecanesulfonic acid | PFDS | 335-77-3 |
| 10:2-Fluorotelomersulfonic acid | 10:2-FTS | 120226-60-0 |
| Perfluorododecanesulfonic acid | PFDoDS | 79780-39-5 |
| Perfluorohexadecanoic acid | PFHxDA | 67905-19-5 |
| Perfluorooctadecanoic acid | PFODA | 16517-11-6 |
| Perfluorooctanesulfonamide | PFOSA | 754-91-6 |
| 2-(N-methylperfluoro-1-octanesulfonamido)-ethanol | NMePFOSAE | 24448-09-7 |
| N-methylperfluoro-1-octanesulfonamide | NMePFOSA | 31506-32-8 |
| 2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol | NEtPFOSAE | 1691-99-2 |
| N-ethylperfluoro-1-octanesulfonamide | NEtPFOSA | 4151-50-2 |
| 2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid | HFPODA | 13252-13-6 |
| 4,8-Dioxa-3H-perfluorononanoic acid | DONA ** | 919005-14-4 * |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid | 9Cl-PF3ONS | 756426-58-1 * |
| 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid | 11Cl-PF3OUdS | 763051-92-9 * |
| 3-Perfluoropropylpropanoic acid | 3:3 FTCA | 356-02-5 |
| 3-Perfluoropentylpropanoic acid | 5:3 FTCA | 914637-49-3 |
| 3-Perfluoroheptylpropanoic acid | 7:3 FTCA | 812-70-4 |
| Perfluoro-3-methoxypropanoic acid | PFMPA/PFECA F | 377-73-1 |
| Perfluoro-4-methoxybutanoic acid | PFMBA/PFECA A | 863090-89-5 |
| Nonfluoro-3,6-dioxahexanoic acid | NFDHA/PFECA B | 151772-58-6 |
| Perfluoro(2-ethoxyethane)sulfonic acid | PFEESA/PES | 113507-82-7 |

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* = CAS number for the free acid form of the analyte.

** = The acronym for the free acid form of the analyte.

Basic Principles

A 250-mL aqueous sample is fortified with isotopically-labeled extraction standards and is passed through a solid phase extraction (SPE) cartridge to extract the analytes. The compounds are eluted from the solid phase with a combination of solvents. The extract is concentrated to ~400-500µl with nitrogen in a heated water bath, and then reconstituted to 1ml with methanol. The sample extract is analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.

Reference Modifications

EPA Method 537 is written specifically for the analysis of drinking water samples. The following modifications to the method have been made to accommodate all aqueous samples.

1. A labeled isotopic analog is spiked into samples for all compounds where an isotopic analog is commercially available. These isotopic compounds are referred to as extraction standards. For those compounds, an isotope dilution calibration model is used. Where labeled isotopes are not available, an internal standard calibration model using the extraction standards is used.
2. Field reagent blanks are not processed as listed in EPA 537 Version 1.1 section 8.3.
3. Trizma is not used for waters except in the cases where the water comes from a chlorinated water source.
4. Peak asymmetry factors are not calculated.
5. MRL confirmation is not performed.
6. Spike concentrations are not rotated between low, medium and high levels.
7. SPE is used for sample preparation. Cartridge types and elution profiles differ from EPA 537 Version 1.1.

MDL studies and IDOCs have been performed to validate method performance.

Interferences

Compounds which have similar structures to the compounds of interest and similar molecular weights would potentially interfere. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene)

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products, LC solvent lines, methanol, aluminum foil, etc. A laboratory blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Precaution to Minimize Method Interference

1. Proprietary Content
2. Proprietary Content
3. PFAS standards, extracts and samples should not come in contact with any glass containers as these analytes can potentially adsorb to glass surfaces. PFAS analytes and labeled extraction standards commercially purchased in glass ampules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be stored in polypropylene containers.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. PFOA has been described as "likely to be carcinogenic to humans". Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal into the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is

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capable of independent preparation. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

Each LC/MS/MS analyst must work with an experienced employee for a period of time until they can independently calibrate the LC/MS/MS, review and process data, and perform maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC and DOC consist of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

See [QA-SOP11178](#) for additional information on IDOC and DOC.

Sample Collection, Preservation, and Handling

A. Sample Collection

The samples are collected in 250-mL polyethylene bottles. Keep the sample sealed from time of collection until extraction.

NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

B. Sample Storage and Shipment

1. Samples must be chilled during shipment and must not exceed 10°C during the first 48 hours after collection. Sample temperature must be confirmed to be at or below 10°C when the samples are received at the laboratory.
2. Samples stored in the lab must be held at a temperature of 0° to 6°C, not frozen, until extraction.
3. Water samples must be extracted within 14 days. Extracts must be analyzed within 28 days after extraction. Extracts are stored at room temperature.

Apparatus and Equipment

A. Apparatus

1. 250mL HDPE bottles: Scientific Specialties; # 334008-blk-1, or equivalent.
2. Centrifuge tubes – 15-mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 05-539-5 or equivalent
3. 10-mL polypropylene volumetric flask, class A – Fisher Scientific, Inc., Cat. No. S02288 or equivalent.
4. HPDE bottles for extraction fluid storage: L; Environmental Sampling Supply, Cat. No. 1000-1902-PC

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5. Analytical Balance – Capable of weighing to 0.0001 g
6. Top-Loading Balance – Capable of weighing to 0.01 g
7. Proprietary Content
8. Proprietary Content
9. Proprietary Content
10. SPE vacuum extraction manifold –“Resprep” 24-port manifold; Restek Corp catalogue # 26080, or equivalent.
11. Proprietary Content
12. Centrifuge – “Q-Sep 3000”; Restek Corp. Cat. No. 26230, or equivalent, capable of a minimum rotational speed of 3000 rpm.
13. Disposable polyethylene pipette – Fisher Scientific, Cat. No. S30467-1 or equivalent
14. Auto Pipettes – Eppendorf; capable of accurately dispensing 10- to 1000-µL. Fisher Scientific cat # 14-287-150, or equivalent.
15. Polypropylene pipette tips: 0-200µl. Fisher; Cat. No. 02-681-135
16. Polypropylene pipette tips: 101-1000µl. Fisher, Cat. No. 02-707-508
17. Pipettes – Disposable transfer. Fisher Scientific, Cat. No. 13-711-7M
18. Vortex mixer, variable speed, Fisher Scientific or equivalent
19. N-Evap sample extract concentrator with N₂ supply and water bath for temperature control. Organomation, Inc. Cat. #11250, or equivalent.
20. Reagent Water Purification System: Capable of producing ultrapure “Type 1/Milli-Q”-grade water from in-house deionized water system. Millipore SAS; Cat. No. FTF08831.
21. Thermo Target PP Polyspring inserts, catalog number C4010-630P
22. Agilent 9mm vial kit pack, catalog number 5190-2278, or equivalent
23. Centrifuge tubes – 50 mL conical polypropylene with polypropylene screw caps; Fisher Scientific, Cat. No. 06-443-21 or equivalent
24. Polypropylene bottles for standard storage - 4 mL; Fisher Scientific, Cat. No. 2006-9125
25. Stainless steel spatula/scoop set. Bel-Art SP Scienceware; Product # 11-865-130

B. Equipment

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1. AB Sciex Triple Quad 4500/5500/5500 Plus Turbo V Ion Source
ExionLC Controller
ExionLC AC Pump
ExionLC AC Autosampler
Exion AC Column Oven
Data system –Analyst 1.7
2. HPLC columns
 - a. Proprietary Content
 - b. Proprietary Content

Reagents and Standards

All solvents, acids, and bases are stored in glass bottles in flammable proof cabinets or pressure resistant steel drums. Solvents, acids, and bases are stored at ambient temperature for up to 1 year. All non-solvents are stored according to manufacturer's storage conditions.

A. Reagents:

1. Methanol (MeOH) – Honeywell Burdick and Jackson "Chromasolv LC-MS" grade Cat. No. BJ34966-4L or equivalent
2. Proprietary Content
3. Proprietary Content
4. Proprietary Content
5. Proprietary Content
6. Proprietary Content
7. Proprietary Content
8. Proprietary Content
9. Graphitized Non-Porous Carbon - Supelco/Millipore Sigma Superclean ENVI-Carb SPE Bulk Packing; Cat. No. 57210-U, or equivalent.

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B. Standards: See SOP [T-PFAS-WI13881](#).

Calibration

A. Initial Calibration

1. A minimum of five calibration standards are required. In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal 7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA and NEtFOSAA. S/N ratio must be greater than or equal to 10:1 for all ions used for quantification.
2. Analyze a Cal3 level standard that contains linear and branch chained isomers of PFOA. The analysis of this standard is used to demonstrate where the branch chained isomers elute and not included in the calibration curve. This will assist the chemist in identifying and properly integrating this compound in samples.
3. Isotopically-labeled compounds are not available for PFPeS, PFHpS, PFNS, PFDS, PFDoS, 10:2-FTS, PFTrA, PFHxDA, PFODA, DONA, 9Cl-PF3ONS, 11Cl-PF3OUdS, 3:3FTCA, 5:3FTCA, 7:3FTCA, PFEESA, PFMPA, PFMBA, and NFDHA. See below for referenced extraction standards. See [Attachment 2](#) for additional information about compound relationships.

| Compound | Extraction standard |
|--------------|---------------------|
| 10:2-FTS | 13C2-8:2-FTS |
| PFTrDA | 13C2-PFDoDA |
| PFHxDA | 13C2-PFTeDA |
| PFODA | |
| PFPeS | 13C3-PFBS |
| PFHpS | 13C3-PFHxS |
| DONA | 13C4-PFHpA |
| PFNS | 13C8-PFOS |
| PFDS | |
| PFDoS | |
| 9Cl-PF3ONS | |
| 11Cl-PF3OUdS | |
| 3:3FTCA | 13C5-PFPeA |
| PFMPA | |
| PFMBA | |
| 5:3FTCA | 13C5-PFHxA |

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|---------|--|
| 7:3FTCA | |
| PFEESA | |
| NFDHA | |

4. Fit the curve

- a. If the % RSD for the response factors is less than or equal to 20%, the average response factor (Ave RRF) can be used to quantitate the data.
- b. If the %RSD is greater than 20%, then a linear regression with a concentration weighing factor of 1/x forced through zero is tried for the compounds not meeting the criteria in 4.a. R² for each analyte using the linear regression must be greater than or equal to 0.99.
- c. If the linear regression curve fails, then a quadratic regression with a concentration weighing factor 1/x² is tried for the compounds not meeting 4.a or 4.b. R² for each analyte using the quadratic regression must be greater than or equal to 0.99. A minimum of six standards must be analyzed to use a quadratic fit.
- d. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration should be within ±30% of its true value.
- e. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

NOTE: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of Analysis (CofA). See [Attachment 4](#).

5. Initial Calibration Verification (ICV)

A check standard prepared from a second source (ICV) is injected to confirm the validity of the calibration curve/standard. If a second source is not available, a separate preparation from the same stock may be used. The calculated amount for each analyte must be within ± 30% of the true value. If this criteria is not met, re-inject or remake the standard. If the criteria is still not met, recalibration is necessary. Instrument maintenance may be needed prior to recalibrating.

B. Continuing Calibration

1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten field samples and at the end of the analysis sequence.

- a. The CCV run after the initial calibration must be at the CAL3 level.
- b. The CCV standards must alternate between the CAL2, CAL3, and CAL4 levels.

2. Acceptance criteria

- a. The calculated amount for each native compound in the CCV standard must be within ±30% of the true value. Samples that are not bracketed by acceptable CCV analyses must be

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reanalyzed. The exception to this would be if the CCV recoveries are high, indicating increased sensitivity, and there are no positive detections in the associated samples, the data may be reported with a qualifying comment.

- b. The absolute areas of the extraction standards for the opening CCV on the day following the ICAL should be within 50-150% of the areas measured during the initial calibration CAL3 standard. On subsequent days where an ICAL is not performed, the absolute areas of the extraction standards from the opening CCV are compared to the absolute areas of the extraction standards from the previous day's opening CCV and should be within 50-150%.
- c. If acceptance criteria are not met, immediately analyze two additional consecutive CCVs. If both pass acceptance criteria, samples may be reported without reanalysis. If either fails, or two consecutive CCVs cannot be run, repeat CCV and reanalyze all samples since last successful CCV.

Procedure

A. Sample Preparation

1. Weigh sample container with contents on a calibrated top loading balance and record the first reading in the automated prep entry system.
 - a. For all samples, the full bottle must be extracted.
 - b. If the sample matrix is such that SPE extraction cannot be performed, the client must be contacted to determine if using a reduced volume is an acceptable alternative. If the client directive is to use a reduced sample volume, see Procedure C.
 - c. If the sample has dissolved and/or settleable solid content (i.e; is cloudy or has a layer of sediment/solids at the bottom of the bottle), the sample must be centrifuged in order to minimize the difficulty of passing through the SPE sorbent bed. In order to preserve the integrity of the sample and ensure the full volume of the container is used, see Procedure D.
2. Use a 250-mL HDPE bottle for the method blank and the laboratory control sample (LCS) and LCSD if needed. Fill each bottle with 250 ml of Milli-Q water. Record 250 mL as the volume for the batch QC samples on the batchlog.

B. Solid Phase Extraction (SPE)

1. Proprietary Content
2. Proprietary Content

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3. Proprietary Content
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14. Proprietary Content
15. Proprietary Content

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16. Proprietary Content

17. Place each empty sample bottle on the top-loading balance and weigh. Record the second reading in the automated prep entry system. The prep entry system will calculate the sample weight. Record the calculated weight as the sample volume on the batchlog.

Note: The instrument lab chemist performs the next steps.

18. Extract Clean Up: DoD requires all sample/QC extracts be treated with a carbon sorbent to remove potential interferences. Once the SPE extracts have been brought to final volume 1 mL, (see step 17 above), 25 mg loose/bulk "ENVI-Carb" carbon sorbent is added to the extract in its centrifuge tube.

19. The sorbent is added with a stainless steel scoop such that, when a level scoop is measured, a 25 mg amount of sorbent is drawn. The 25 mg of sorbent is added directly to the extracts in the 15 mL centrifuge tube. The scoop is rinsed with MeOH and dried between samples to avoid cross-contamination.

20. After addition of the ENVI-Carb the extracts are centrifuged for a complete cycle and are ready for vialing into LC vials for analysis.

21. Transfer 400 µL of the final extract to the corresponding labeled auto-sampler vial. Cap and vortex the auto-sampler vial. Samples are now ready for analysis.

22. Cap the centrifuge tube. Store the remaining centrifuged extracts at room temperature for dilution or reinjection if needed.

C. Reduced Sample Volume

1. Determine the aliquot to be used for extraction (i.e.; 50 mL, 100 mL).
2. Label a clean 250-mL HDPE bottle with the associated ELLE sample number.
3. Label the appropriate number of 50-mL centrifuge tubes with the associated ELLE sample number. The number required will be determined by the volume to be used for extraction.
 1. 4.Shake/invert the sample bottle to thoroughly mix the sample before pouring aliquot(s).
4. Pour sample from original bottle into centrifuge tubes. Cap tubes and centrifuge for 5 minutes at full speed (one full cycle).
5. On a calibrated, top-loading balance, place labeled empty 250 mL PP wide-mouthed bottle.
6. Decant centrifuged sample aliquot(s) from centrifuge tube(s) to the 250 mL bottle until desired volume (weight in grams) is reached. 100 g = 100 mL, 50 g = 50 mL, etc. If the weight is exceeded, remove excess volume with a disposable pipette and discard to a waste container.
7. Add Milli-Q water to the bottle until a weight of 250 g (total of 250 mL) is reached.
8. Shake/invert several times to mix thoroughly.
9. Record the aliquot taken from the original bottle (50 mL, 100 mL) as the sample volume.

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| Approved by: XL3S Effective Date 25-MAY-2022 | Document users: 5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep | Responsible: 5_EUUSLA_PFAS_Manager |

10. Extract sample beginning with Procedure B.

D. Samples Containing Dissolved and/or Settleable Solids

1. Spike sample with appropriate spikes as in Procedure B.5.
2. Centrifuge the full bottle.
3. DO NOT SHAKE BOTTLE FOLLOWING THE CENTRIFUGE STEP.
4. Follow steps in Procedure B, 1 through 3.
5. Attach a 25-mL SPE reservoir to each cartridge. Decant centrifuged sample onto its respective SPE cartridge. Allow full volume to pass through each cartridge by gravity, if possible. Apply light vacuum if necessary. The drip rate should be approximately 1-2 drops per second.
6. Rinse the sample bottle with 5 mL of Milli-Q water, add the rinseate to the cartridge, and repeat.
7. Continue extraction process with Procedure B. 7.

E. LC/MS/MS Analysis

1. Mass Calibration and Tuning
 - a. At instrument set up and installation and after the performance of major maintenance, calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.
 - b. When masses fall outside of the ± 0.5 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.5 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.
2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak. See the AB Sciex (4500/5500/5500 plus) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.
3. Example Acquisition method: See *Proprietary Content*.
4. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of PFBA, PFPeA, PFOSA, NMePFOSAE, NMEPFOSA, NEtPFOSAE, NEtPFOSA, PFDoS, DONA, 9Cl-PF3ONS, 11Cl-PF3OUdS, PFMPA, 3:3 FTCA, PFMBA, PFEESA, and 7:3 FTCA. See *Attachment 1*.
5. Instrument Sensitivity Check (ISC) and Instrument Blanks

| | | | |
|---|--|--|--|
| | Always check on-line for validity. | Level: |  |
| Document number: T-PFAS-WI36458 | Polyfluorinated Alkyl Substances (PFAS) in Aqueous Samples by Method 537 Version 1.1 Modified QSM5.4 Table B-15 Using LC/MS/MS | Work Instruction | |
| Old Reference: | | | |
| Version: 3 | | Organisation level: 5-Sub-BU | |
| Approved by: XL3S Effective Date 25-MAY-2022 | Document users: 5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep | Responsible: 5_EUUSLA_PFAS_Manager | |

- a. Prior to sample analysis and at least every 12 hours, an instrument sensitivity check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL2 standard's concentration is at the LOQ. The CAL2 standard will be analyzed. All analyte concentrations must be within $\pm 30\%$ of their true values. If the criteria is not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.
 - b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily or at the start of a sequence. The concentration of all analytes must be less than or equal to 1/2 the LOQ. If acceptance criteria are not met the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.
6. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks to clean the instrument prior to sample acquisition.
 7. After the initial calibration, inject an instrument blank, followed by the ICV, Linear Branched (L/B) standard, closing Cal3 level CCV, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard, alternating between the Cal2, Cal3, and Cal4 levels.
 8. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.
 9. Quantitate results for the extraction blank. No target analytes detected greater than 1/2 the LOQ or greater than 1/10 the regulatory limit, whichever is greater. If criteria is exceeded, reextract all samples with positive detections associated with the method blank. If the target analyte in the sample is detected at a concentration greater than 10 times the amount detected in the method blank, the data is reported.
 10. Calculate the recoveries of spiked analytes for the LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.
 - a. Method defined limits are used for the evaluation of the LCS and MS/MSD recoveries. Where there are no limits stated an advisory window of 70-130% will be used until sufficient data points are available to generate statistical windows. The QC acceptance limit for the relative percent difference (%RPD) between LCS/LCSD and MS/MSD is less than or equal to 30%.
 - b. If LCS and/or LCSD recoveries are acceptable, proceed to sample quantitation.
 - c. If the LCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, the samples associated with the LCS must be reextracted.
 - d. If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.

| | | | |
|---|--|--|--|
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| Old Reference: | | | |
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| Approved by: XL3S Effective Date 25-MAY-2022 | Document users: 5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep | Responsible: 5_EUUSLA_PFAS_Manager | |

11. Isotopically-labeled extraction standards are added to all samples, extraction blank, LCS/LCSD, and MS/MSD prior to extraction. The absolute areas of the extraction standards must be within $\pm 50\%$ of the areas measured in the ICAL mid-point standard (CAL3 standard). On days when an ICAL is not performed, the absolute areas must be within $\pm 50\%$ of the absolute areas measured in the daily opening initial CCV. If the extraction standards fall outside the acceptance window, analyze a second aliquot of the extract. If none remains, reanalyze the first aliquot.

Refer to [Attachment 5](#) for the Eurofins DOD variances for isotopically labeled recoveries. If these variances are accepted by the client, the laboratory will follow these guidelines in determining when to re-extract samples. If the client does not accept these variances, samples with recoveries that are outside the $\pm 50\%$ of the true value acceptance criteria must be re-extracted.

12. Compare the retention times of all of the analytes and extraction standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than:
- 0.4 minutes for isotopically-labeled compounds
 - 0.1 minutes from their analog for native compounds with an exact isotopically-labeled compound
 - 0.4 minutes from their assigned analog for native compounds without an exact isotopically-labeled compound.

If the retention time is outside of the criteria, the compound is considered a false positive unless it is a compound with branched isomers. Compounds with branched isomers can vary in intensity of the individual isomers that are used for reporting and must be reviewed and compared to the preceding CCV to determine if it should be reported.

13. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of PFBA, PFPeA, PFOSA, NMePFOSAE, NMEPFOSA, NEtPFOSAE, NEtPFOSA, PFDoS, DONA, 9Cl-PF3ONS, 11Cl-PF3OUdS, PFMPA, 3:3 FTCA, PFMB, PFEESA, and 7:3 FTCA. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards. When an ion ratio for a compound differs from the expected ion ratio by more than 50%, a qualifier is placed on the raw data and on the sample report. No corrective action is required.
14. The linear/branch chain standard is used when assessing the correctness of the computer generated peak integrations for PFOA.
15. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract with MeOH. If the sample dilution required exceeds 100 fold, the client must be contacted to determine if the data can be reported with result(s) that exceed the calibration range or if the sample should be re-prepped at a reduced volume.

Dilution Example 1/10: Mix 900 μL of MeOH with 100 μL of sample extract. Vortex to mix. Using an auto-pipette, transfer 400 μL of the mixed solution into a labeled auto-sampler vial. Cap and vortex thoroughly to mix.

Calculations

| | | |
|---|--|---|
| | Always check on-line for validity. | Level:  |
| Document number: T-PFAS-WI36458 | Polyfluorinated Alkyl Substances (PFAS) in Aqueous Samples by Method 537 Version 1.1 Modified QSM5.4 Table B-15 Using LC/MS/MS | Work Instruction |
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A. Peak Area Ratio

$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

B. On-Column Analyte Concentration using linear through zero curves

On-column Concentration = (peak area ratio x Isotope Dilution Analyte concentration) ÷ slope

C. Sample Concentration

Sample concentration (ng/l) = (On-column concentration x Final Sample volume x DF) ÷ Sample weight

D. Ion Ratio

ion ratio = (peak area or height of quantifier)/(peak area or height of qualifier)

E. See [T-PEST-WI9847](#) for additional calculations used to evaluate the calibrations and quality control samples.

Statistical Information/Method Performance

The LCS should contain all compounds of interest. LCS, MS, extraction standard recoveries and RPD are compared to the limits stored on the LIMS. These limits are defined by the method. For compounds not defined by the method, these limits are statistically derived when sufficient data points are available. If sufficient data points are not available to generate statistical windows, an advisory window of 70% to 130% will be used. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to [QA-SOP11892](#) for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

Quality Assurance/Quality Control

For each batch of 20 samples extracted, a method blank and an LCS/LCSD (Milli Q water spiked with all compounds to be determined carried through the entire procedure) must be extracted and analyzed. If an MS/MSD is submitted then an LCSD would not be extracted. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

Attachment:
[Attachment 1 - Mass Transitions \(.doc\)](#)

| | | | |
|---|--|--|--|
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- Attachment 2 - Compound Relationships (.docx)
Attachment 3 - Proprietary Content
Attachment 4 - Example Certificate of Analysis (.pdf)
Attachment 5 - Eurofins DoD variances (.doc)

G-DC-FRM23907 Redacted SOPs
QA-SOP11178 Demonstrations of Capability
QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
T-PEST-WI9847 Common Equations Used During Chromatographic Analyses
T-PFAS-WI13881 Standards Management in the PFAS Laboratory
Attachment: Attachment 1 - Mass Transitions (doc)
Attachment: Attachment 2 - Compound Relationships (docx)
Attachment: Attachment 3 - Proprietary Content
Attachment: Attachment 4 - Example Certificate of Analysis (pdf)
Attachment: Attachment 5 - Eurofins DoD variances (doc)

End of document

Version history

| Version | Approval | Revision information | |
|---------|-------------|----------------------|--|
| 1 | 28.JAN.2021 | | |
| 2 | 14.FEB.2022 | | |
| 3 | 25.MAY.2022 | | |

Attachment 1

Mass Transitions AB Sciex 4500/5500/5500+

| Compound | Parent Ion | Daughter Ion |
|--------------|------------|--------------|
| 13C4-PFBA | 217 | 172 |
| PFBA | 213 | 169 |
| 13C5-PFPeA | 268 | 223 |
| PFPeA | 263 | 219 |
| 13C3-PFBS | 302 | 80 |
| PFBS | 299 | 80 |
| PFBS (2) | 299 | 99 |
| 13C2-4:2-FTS | 329 | 81 |
| 4:2-FTS | 327 | 307 |
| 4:2-FTS (2) | 327 | 81 |
| 13C5-PFHxA | 318 | 273 |
| PFHxA | 313 | 269 |
| PFHxA (2) | 313 | 119 |
| PFPeS | 349 | 80 |
| PFPeS (2) | 349 | 99 |
| 13C3-PFHxS | 402 | 80 |
| PFHxS | 399 | 80 |
| PFHxS (2) | 399 | 99 |
| 13C4-PFHpA | 367 | 322 |
| PFHpA | 363 | 319 |
| PFHpA (2) | 363 | 169 |
| 13C2-6:2-FTS | 429 | 81 |
| 6:2-FTS | 427 | 407 |
| 6:2-FTS (2) | 427 | 81 |
| PFHpS | 449 | 80 |
| PFHpS (2) | 449 | 99 |
| 13C8-PFOA | 421 | 376 |
| PFOA | 413 | 369 |
| PFOA (2) | 413 | 169 |
| 13C8-PFOS | 507 | 80 |
| PFOS | 499 | 80 |
| PFOS (2) | 499 | 99 |
| 13C9-PFNA | 472 | 427 |
| PFNA | 463 | 419 |
| PFNA (2) | 463 | 169 |
| 13C8-PFOSA | 506 | 78 |
| PFOSA | 498 | 78 |

Attachment 1

| Compound | Parent Ion | Daughter Ion |
|--------------|------------|--------------|
| PFNS | 549 | 80 |
| PFNS (2) | 549 | 99 |
| 13C6-PFDA | 519 | 474 |
| PFDA | 513 | 469 |
| PFDA (2) | 513 | 169 |
| 13C2-8:2-FTS | 529 | 81 |
| 8:2-FTS | 527 | 507 |
| 8:2-FTS (2) | 527 | 81 |
| d7-NMePFOSAE | 623 | 59 |
| NMePFOSAE | 616 | 59 |
| d3-NMePFOSA | 515 | 169 |
| NMEPFOSA | 512 | 169 |
| d3-NMeFOSAA | 573 | 419 |
| NMeFOSAA | 570 | 419 |
| NMeFOSAA (2) | 570 | 483 |
| d9-NEtPFOSAE | 639 | 59 |
| NEtPFOSAE | 630 | 59 |
| d5-NETPFOSA | 531 | 169 |
| NEtPFOSA | 526 | 169 |
| PFDS | 599 | 80 |
| PFDS (2) | 599 | 99 |
| 13C7-PFUnDA | 570 | 525 |
| PFUnDA | 563 | 519 |
| PFUnDA (2) | 563 | 169 |
| d5-NEtFOSAA | 589 | 419 |
| NEtFOSAA | 584 | 419 |
| NEtFOSAA (2) | 584 | 526 |
| 13C2-PFDoDA | 615 | 570 |
| PFDoDA | 613 | 569 |
| PFDoDA (2) | 613 | 169 |
| 10:2-FTS | 627 | 607 |
| 10:2-FTS (2) | 627 | 81 |
| PFDoS | 699 | 80 |
| PFTrDA | 663 | 619 |
| PFTrDA (2) | 663 | 169 |
| 13C2-PFTeDA | 715 | 670 |
| PFTeDA | 713 | 669 |
| PFTeDA (2) | 713 | 169 |
| PFHxDA | 813 | 769 |
| PFHxDA (2) | 813 | 169 |

Attachment 1

| Compound | Parent Ion | Daughter Ion |
|--------------------|------------|--------------|
| PFODA | 913 | 869 |
| PFODA (2) | 913 | 169 |
| 13C3-HFPODA | 332 | 287 |
| HFPODA | 329 | 285 |
| HFPODA (2) | 285 | 169 |
| DONA | 377 | 251 |
| 9Cl-PF3ONS | 531 | 351 |
| 11Cl-PF3OUdS | 631 | 451 |
| PFECA B (NFDHA) | 201 | 85 |
| PFECA B(NFDHA) (2) | 295 | 201 |
| PFECA F (PFMPA) | 229 | 85 |
| 3:3 FTCA | 241 | 177 |
| PFECA A (PFMBA) | 279 | 85 |
| PFEESA (PES) | 315 | 135 |
| 5:3 FTCA | 341 | 237 |
| 5:3 FTCA (2) | 339 | 217 |
| 7:3 FTCA | 441 | 317 |

Confidential

Attachment 2

| |
|--|
| PFAS Native Compounds/Extraction Standards |
|--|

Native PFAS Compounds

| Native | Extraction Standard |
|--------------|---------------------|
| PFBA | 13C4-PFBA |
| PFPeA | 13C5-PFPeA |
| 3:3FTCA | |
| PFMPA | |
| PFMBA | |
| PFBS | 13C3-PFBS |
| PFPeS | |
| 4:2-FTS | 13C2-4:2-FTS |
| PFHxA | 13C5-PFHxA |
| NFDHA | |
| 5:3FTCA | |
| 7:3FTCA | |
| PFEESA | |
| PFHxS | 13C3-PFHxS |
| PFHpS | |
| PFHpA | 13C4-PFHpA |
| DONA | |
| 6:2-FTS | 13C2-6:2-FTS |
| PFOA | 13C8-PFOA |
| PFOS | 13C8-PFOS |
| PFNS | |
| PFDS | |
| 9Cl-PF3ONS | |
| 11Cl-pf3OUdS | |
| PFDoS | |
| PFNA | 13C9-PFNA |
| PFOSA | 13C8-PFOSA |
| PFDA | 13C6-PFDA |

| Native | Extraction Standard |
|---------------------|----------------------------|
| 8:2-FTS | 13C2-8:2-FTS |
| 10:2-FTS | |
| NMePFOSAE | d7-NMePFOSAE |
| NMePFOSA | d3-NMePFOSA |
| NMeFOSAA | d3-NMeFOSAA |
| NEtPFOSAE | d9-NEtPFOSAE |
| NEtPFOSA | d5-NEtPFOSA |
| PFUnDA | 13C7-PFUnDA |
| NEtFOSAA | d5-NEtFOSAA |
| PFD _o DA | 13C2-PFD _o DA |
| PFT _r DA | |
| PFT _e DA | 13C2-PFT _e DA |
| PFH _x DA | |
| PFODA | |
| HFPODA | 13C3-HFPODA |

Confidential

PFAC-MXC

Native Perfluorinated Compound Solution/Mixture

| | |
|---|-------------------------------------|
| <u>PRODUCT CODE:</u> | PFAC-MXC |
| <u>LOT NUMBER:</u> | PFACMXC0617 |
| <u>SOLVENT(S):</u> | Methanol / Water (<1%) |
| <u>DATE PREPARED:</u> (mm/dd/yyyy) | 06/14/2017 |
| <u>LAST TESTED:</u> (mm/dd/yyyy) | 03/19/2019 |
| <u>EXPIRY DATE:</u> (mm/dd/yyyy) | 03/19/2024 |
| <u>RECOMMENDED STORAGE:</u> | Store ampoule in a cool, dark place |

DESCRIPTION:

PFAC-MXC is a solution/mixture of thirteen native perfluoroalkylcarboxylic acids (C₄-C₁₄, C₆, and C₈) and eight native perfluoroalkylsulfonates (C₄-C₁₄ and C₆, J). The full name, abbreviation and concentration for each of the components are given in Table A.

The individual perfluoroalkylcarboxylic acids and perfluoroalkylsulfonates all have chemical purities of >98%.

DOCUMENTATION/ DATA ATTACHED:

Table A: Components and Concentrations of the Solution/Mixture
 Figure 1: LC/MS Data (SIR)
 Figure 2: LC/MS/MS Data (Selected MRM Transitions)

ADDITIONAL INFORMATION:

See page 2 for further details.

Contains 4 mole eq. of NaOH to prevent conversion of the carboxylic acids to their respective methyl esters.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

ATTACHMENT 4

INTENDED USE:

The products prepared by Wellington Laboratories Inc. are for laboratory use only. This certified reference material (CRM) was designed to be used as a standard for the identification and/or quantification of the specific chemical compounds it contains.

HANDLING:

This product should only be used by qualified personnel familiar with its potential hazards and trained in the handling of hazardous chemicals. Due care should be exercised to prevent unnecessary human contact or ingestion. All procedures should be carried out in a well-functioning fume hood and suitable gloves, eye protection, and clothing should be worn at all times. Waste should be disposed of according to national and regional regulations. Safety Data Sheets (SDSs) are available upon request.

SYNTHESIS / CHARACTERIZATION:

Our products are synthesized using single-product unambiguous routes whenever possible. They are then characterized, and their structures and purities confirmed, using a combination of the most relevant techniques, such as NMR, GC/MS, LC/MS/MS, SFC/UV/MS/MS, x-ray crystallography, and melting point. Isotopic purities of mass-labelled compounds are also confirmed using HRGC/HRMS and/or LC/MS/MS.

HOMOGENEITY:

Prior to solution preparation, crystalline material is tested for homogeneity using a variety of techniques (as stated above) and its solubility in a given diluent is taken into consideration. Duplicate solutions of a new product are prepared from the same crystalline lot and, after the addition of an appropriate internal standard, they are compared by GC/MS, LC/MS/MS, and/or SFC/UV/MS/MS. The relative response factors of the analyte of interest in each solution are required to be <5% RSD. New solution lots of existing products, as well as mixtures and calibration solutions, are compared to older lots in a similar manner. This further confirms the homogeneity of the crystalline material as well as the stability and homogeneity of the solutions in the storage containers. In order to maintain the integrity of the assigned value(s), and associated uncertainty, the dilution or injection of a subsample of this product should be performed using calibrated measuring equipment.

UNCERTAINTY:

The maximum combined relative standard uncertainty of our reference standard solutions is calculated using the following equation:

The combined relative standard uncertainty, $u_c(y)$, of a value y and the uncertainty of the independent parameters x_1, x_2, \dots, x_n on which it depends is:

$$u_c(y(x_1, x_2, \dots, x_n)) = \sqrt{\sum_{i=1}^n \left(\frac{\partial y}{\partial x_i} u(x_i) \right)^2}$$

where x is expressed as a relative standard uncertainty of the individual parameter.

The individual uncertainties taken into account include those associated with weights (calibration of the balance) and volumes (calibration of the volumetric glassware). An expanded maximum combined percent relative uncertainty of $\pm 5\%$ (calculated with a coverage factor of 2 and a level of confidence of 95%) is stated on the Certificate of Analysis for all of our products.

TRACEABILITY:

All reference standard solutions are traceable to specific crystalline lots. The microbalances used for solution preparation are regularly calibrated by an external ISO/IEC 17025 accredited laboratory. In addition, their calibration is verified prior to each weighing using calibrated external weights traceable to an ISO/IEC 17025 accredited laboratory. All volumetric glassware used is calibrated, of Class A tolerance, and traceable to an ISO/IEC 17025 accredited laboratory. For certain products, traceability to international interlaboratory studies has also been established.

EXPIRY DATE / PERIOD OF VALIDITY:

Ongoing stability studies of this product have demonstrated stability in its composition and concentration, until the specified expiry date, in the unopened ampoule. Monitoring for any degradation or change in concentration of the listed analyte(s) is performed on a routine basis.

LIMITED WARRANTY:

At the time of shipment, all products are warranted to be free of defects in material and workmanship and to conform to the stated technical and purity specifications.

QUALITY MANAGEMENT:

This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAI Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A 1226), and ISO 17034 by ANSI-ASQ National Accreditation Board (ANAB; AR-1523).



**For additional information or assistance concerning this or any other products from Wellington Laboratories Inc.,

ATTACHMENT 4

Table A: PFAC-MXC; Components and Concentrations (ng/ml, ± 5% in Methanol/ Water (<1%))

| Compound | Abbreviation | Concentration (ng/ml)* | | Peak Assignment in Figure 1 |
|---------------------------------------|--------------|------------------------|--------------|-----------------------------|
| | | As the salt | As the anion | |
| Perfluoro-n-butanoic acid | PFBA | 2000 | | A |
| Perfluoro-n-pentanoic acid | PFPeA | 2000 | | B |
| Perfluoro-n-hexanoic acid | PFHxA | 2000 | | D |
| Perfluoro-n-heptanoic acid | PFHpA | 2000 | | F |
| Perfluoro-n-octanoic acid | PFOA | 2000 | | H |
| Perfluoro-n-nonanoic acid | PFNA | 2000 | | J |
| Perfluoro-n-decanoic acid | PFDA | 2000 | | L |
| Perfluoro-n-undecanoic acid | PFUdA | 2000 | | N |
| Perfluoro-n-dodecanoic acid | PFDoA | 2000 | | P |
| Perfluoro-n-tridecanoic acid | PFTrDA | 2000 | | Q |
| Perfluoro-n-tetradecanoic acid | PFTeDA | 2000 | | S |
| Perfluoro-n-hexadecanoic acid | PFHxDA | 2000 | | T |
| Perfluoro-n-octadecanoic acid | PFODA | 2000 | | U |
| Compound | Abbreviation | Concentration (ng/ml)* | | Peak Assignment in Figure 1 |
| | | As the salt | As the anion | |
| Potassium perfluoro-1-butanesulfonate | L-PFBS | 2000 | 1770 | C |
| Sodium perfluoro-1-pentanesulfonate | L-PFPeS | 2000 | 1880 | E |
| Sodium perfluoro-1-hexanesulfonate | L-PFHxS | 2000 | 1890 | G |
| Sodium perfluoro-1-heptanesulfonate | L-PFHpS | 2000 | 1900 | I |
| Sodium perfluoro-1-octanesulfonate | L-PFOS | 2000 | 1910 | K |
| Sodium perfluoro-1-nonanesulfonate | L-PFNS | 2000 | 1920 | M |
| Sodium perfluoro-1-decanesulfonate | L-PFDS | 2000 | 1930 | O |
| Sodium perfluoro-1-dodecanesulfonate | L-PFDoS | 2000 | 1940 | R |

* Concentrations have been rounded to three significant figures.

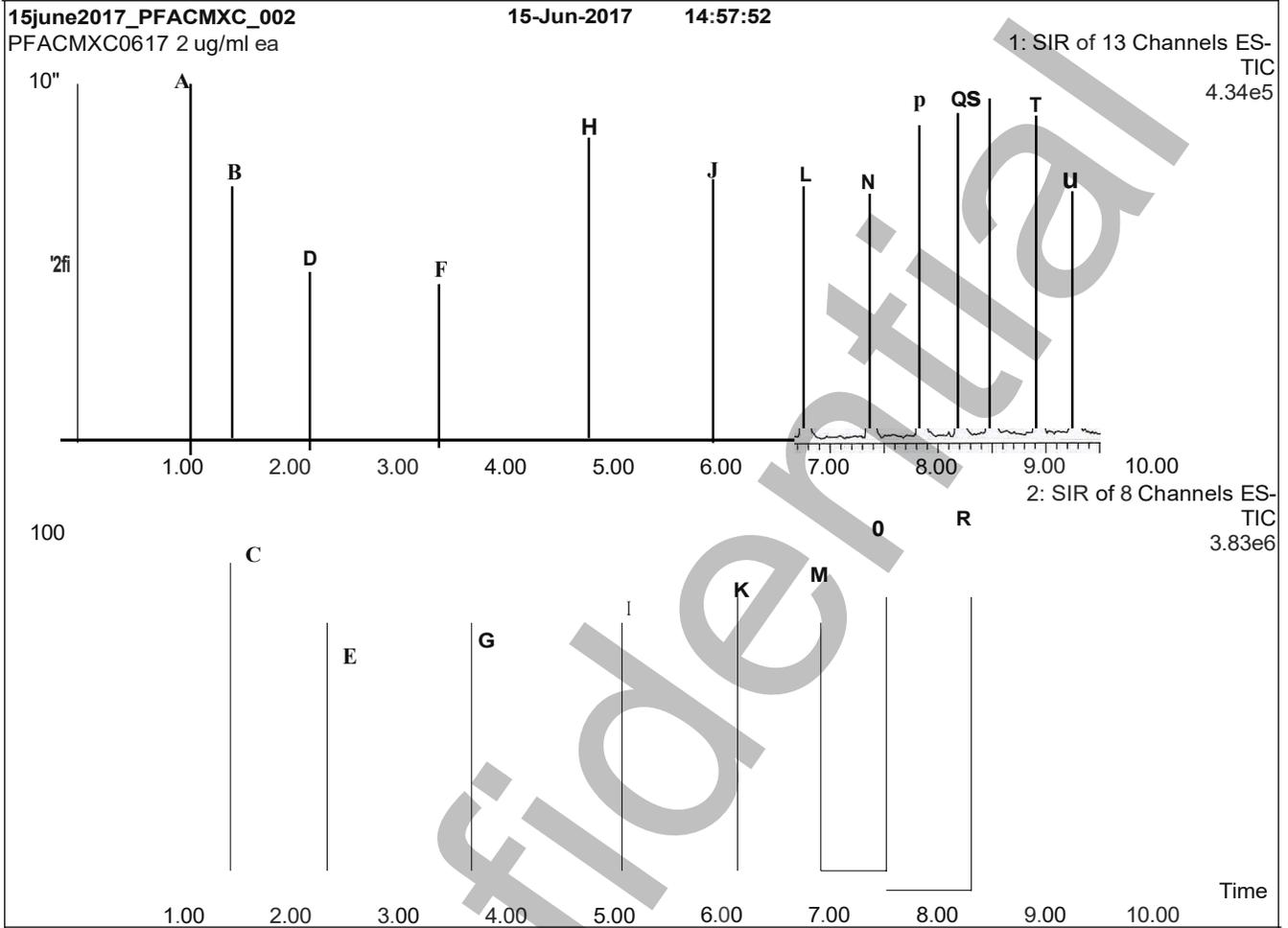
Certified By: _____

B.G. Chittim, General Manager

Date: 06/06/2019
(mm/dd/yyyy)

ATTACHMENT 4

Fi.9yr!L1 PFAC-MXC; LC/MS Data (Total Ion Current Chromatogram; SIR)



Conditions for Figure 1:

LC: Waters Acquity Ultra Performance LC
MS: Micromass Quattro *micro* API MS

Chromatographic Conditions

Column: Acquity UPLC BEH Shield RP.,
1.7 μ m, 2.1 x 100 mm

Mobile phase: Gradient
Start: 50% H₂O / 50% (80:20 MeOH:ACN)
(both with 10 mM NH₄OAc buffer)
Ramp to 90% organic over 8 min and hold for 2 min
before returning to initial conditions in 1 min.

Time: 12 min

Flow: 300 μ l/min

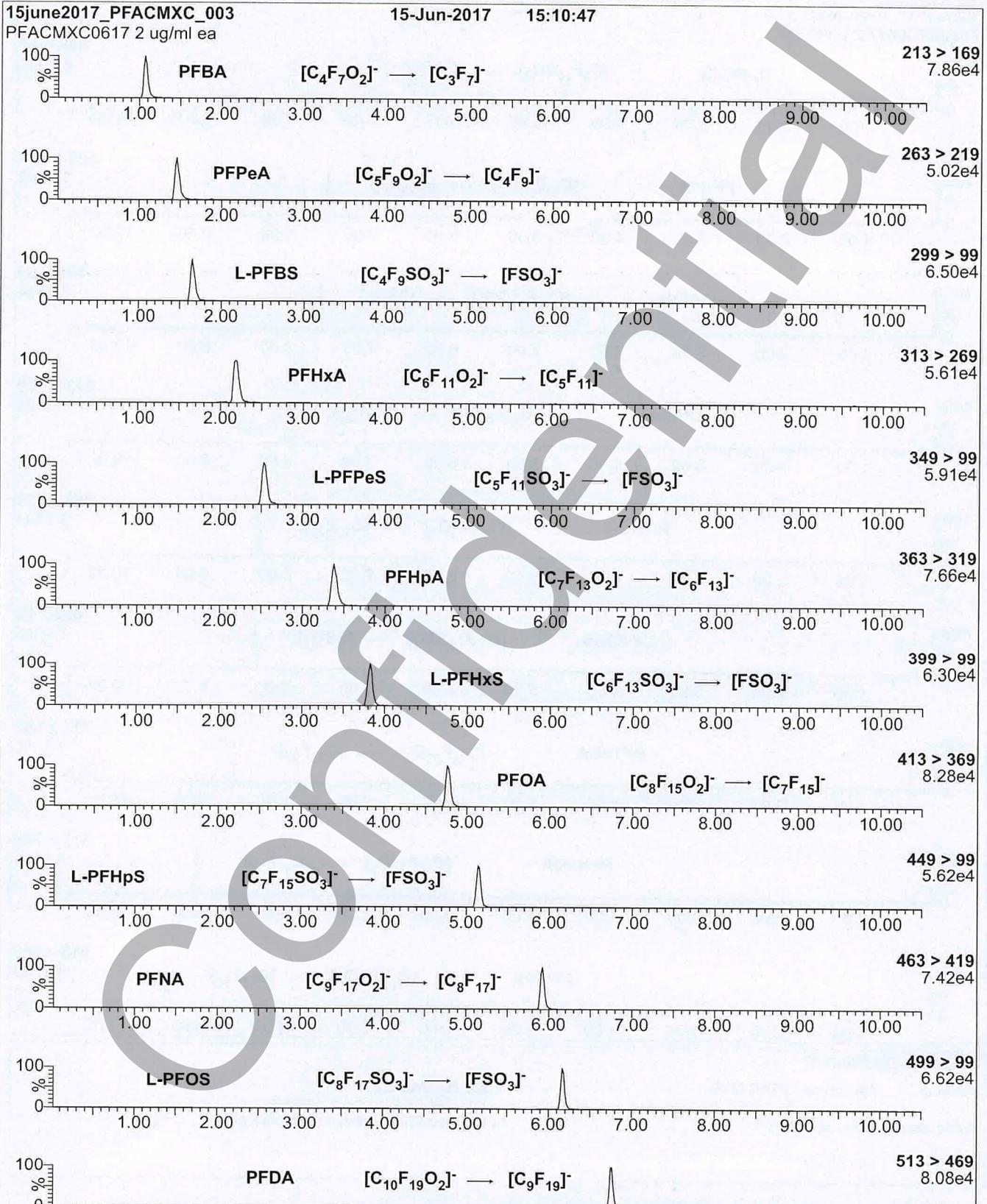
MS Parameters

Experiment: SIR of 21 Channels

Source: Electrospray (negative)
Capillary Voltage (kV) = 3.00
Cone Voltage (V) = variable (10-80)
Cone Gas Flow (l/hr) = 50
Desolvation Gas Flow (l/hr) = 750

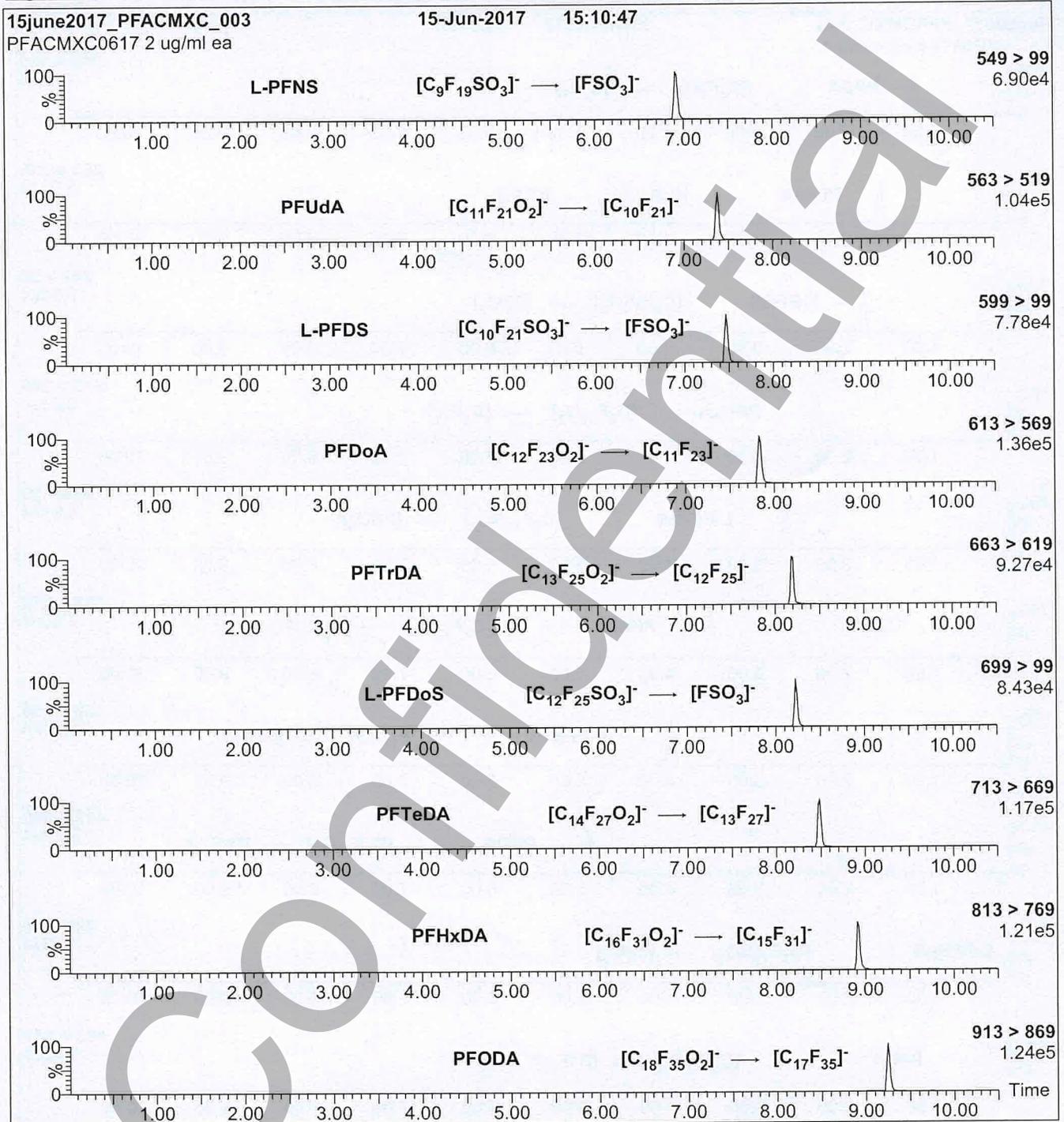
ATTACHMENT 4

Figure 2: PFAC-MXC; LC/MS/MS Data (Selected MRM Transitions)



ATTACHMENT 4

Figure 2: PFAC-MXC; LC/MS/MS Data (Selected MRM Transitions)



Conditions for Figure 2:

Injection: On-column (PFAC-MXC)
 Mobile phase: Same as Figure 1
 Flow: 300 μ l/min

MS Parameters

Collision Gas (mbar) = 3.46e-3
 Collision Energy (eV) = 8-50 (variable)

Eurofins Lancaster Laboratories Environmental, LLC Variance Requests

| Item | Parameter | Scope of Work | ELLE SOP | Approval |
|------|---|--|--|----------|
| 1 | QSM 5.3 Table B-15 PFAS Using LC/MS/MS | Extraction internal standard (EIS) recovery criteria of 50- 150% | For all PFAS compounds if EIS recovery is <50%, we will check for laboratory error and correct if identified. If no laboratory error is identified, additional corrective action will be performed if EIS recovery is <10%. If EIS recovery is >10% and samples have detections above the reporting limit, no additional corrective action is performed. If EIS recovery is >10% and samples have no detections for associated native analytes, the native analyte response will be evaluated to confirm the validity of the reporting limit. Reporting limits will be proportionately increased as necessary and appropriate to ensure that reported values accurately reflect the sensitivity of the analysis. | |
| 2 | QSM 5.3 Table B-15 PFAS Using LC/MS/MS | Extraction internal standard (EIS) recovery criteria of 50- 150% | For all PFAS compounds, if EIS recovery is >150%, we will check for laboratory error and correct if identified. If no laboratory error is identified, additional corrective action will be performed only when field samples have detections above the reporting limits for the associated native target analytes and EIS recovery is >200%. | |
| 3 | QSM 5.3 Table B-15 PFAS Using LC/MS/MS | Sample Preparation | If persistent matrix effects are observed for multiple samples from a given project that necessitate a high rate of additional corrective actions, these matrix effects will be mitigated prior to sample preparation and analysis, typically by processing a smaller sample mass or volume. | |

| | | |
|---|--|---|
| | Always check on-line for validity. | Level:  |
| Document number: T-PFAS-WI36459 | Polyfluorinated Alkyl Substances (PFAS) in Solids by Method 537 Version 1.1 Modified QSM 5.4 Table B-15 Using LC/MS/MS | Work Instruction |
| Old Reference: | | |
| Version: 2 | | Organisation level: 5-Sub-BU |
| Approved by: XL3S Effective Date 25-MAY-2022 | Document users: 5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep | Responsible: 5_EUUSLA_PFAS_Manager |

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|------------------|----------------------|---|
| Revision: | <u>01</u> | Effective date: <u>28-JAN-2021</u> |
| Section | Justification | Changes |
| | NEW | |

Reference

1. Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LCMSMS), EPA 537 Version 1.1, September 2009. Department of Defense Quality System Manual Version 5.4, Table B-15.
2. US EPA Method 537, Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LCMSMS), Version 1.1, September 2009.
3. Standard Test Method for Determination of Perfluorinated Compounds in Soil by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS), ASTM Method D7968, 2014.
4. Method for Trace Level Analysis of C8, C9, C10, C11, and C13 Perfluorocarbon Carboxylic Acids in Water. Karen Risha, John Flaherty, Roice Wille, Warren Buck, Francesco Morandi, and Tsuguhide Isemura. Anal. Chem. 2005, 77, 1503-1508.
5. *Chemical Hygiene Plan*, current version.

Cross Reference

| Document | Document Title |
|--------------------------------|--|
| T-PEST-WI9847 | Common Equations Used During Chromatographic Analyses |
| T-PFAS-WI13881 | Standards Management in the PFAS Laboratory |
| G-DC-FRM23907 | Redacted SOPs |
| QA-SOP11178 | Demonstrations of Capability |
| QA-SOP11892 | Determining Method Detection Limits and Limits of Quantitation |

Scope

This method is applicable for the determination of selected per- and polyfluorinated alkyl substances (PFAS) in soil and solid samples. The compounds analyzed in this method are listed in the table below. The most current MDLs and LOQs are listed in the LIMS.

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| | Old Reference: | Responsible: 5_EUUSLA_PFAS_Manager | |
| Version: 2 | Document users: 5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep | Approved by: XL3S Effective Date 25-MAY-2022 | |

| Analyte | Acronym | CAS# |
|--|----------|-------------|
| Perfluorobutanesulfonic acid | PFBS | 375-73-5 |
| Perfluorodecanoic acid | PFDA | 335-76-2 |
| Perfluorododecanoic acid | PFDoDA | 307-55-1 |
| Perfluoroheptanoic acid | PFHpA | 375-85-9 |
| Perfluorohexanesulfonic acid | PFHxS | 355-46-4 |
| Perfluorohexanoic acid | PFHxA | 307-24-4 |
| Perfluorononanoic acid | PFNA | 375-95-1 |
| Perfluorooctanesulfonic acid | PFOS | 1763-23-1 |
| Perfluorooctanoic acid | PFOA | 335-67-1 |
| Perfluorotetradecanoic acid | PFTeDA | 376-06-7 |
| Perfluorotridecanoic acid | PFTrDA | 72629-94-8 |
| Perfluoroundecanoic acid | PFUnDA | 2058-94-8 |
| Perfluoro-n-butanoic acid | PFBA | 375-22-4 |
| Perfluoro-n-pentanoic acid | PFPeA | 2706-90-3 |
| 8:2 - Fluorotelomersulfonate | 8:2FTS | 39108-34-4 |
| N-methylperfluoro-1-octanesulfonamidoacetic acid | NMeFOSAA | 2355-31-9 |
| N-ethylperfluoro-1-octanesulfonamidoacetic acid | NEtFOSAA | 2991-50-6 |
| 4:2-Fluorotelomersulfonic acid | 4:2-FTS | 757124-72-4 |
| Perfluoropentanesulfonic acid | PFPeS | 2706-91-4 |
| 6:2-Fluorotelomersulfonic acid | 6:2-FTS | 27619-97-2 |
| Perfluoroheptanesulfonic acid | PFHpS | 375-92-8 |
| Perfluorononanesulfonic acid | PFNS | 68259-12-1 |
| Perfluorodecanesulfonic acid | PFDS | 335-77-3 |
| 10:2-Fluorotelomersulfonic acid | 10:2-FTS | 120226-60-0 |

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| Analyte | Acronym | CAS# |
|---|---------------------|---------------|
| Perfluorododecanesulfonic acid | PFD _o DS | 79780-39-5 |
| Perfluorohexadecanoic acid | PFH _x DA | 67905-19-5 |
| Perfluorooctadecanoic acid | PFODA | 16517-11-6 |
| Perfluorooctanesulfonamide | PFOSA | 754-91-6 |
| 2-(N-methylperfluoro-1-octanesulfonamido)- ethanol | NMePFOSAE | 24448-09-7 |
| N-methylperfluoro-1-octanesulfonamide | NMePFOSA | 31506-32-8 |
| 2-(N-ethylperfluoro-1-octanesulfonamido)- ethanol | NEtPFOSAE | 1691-99-2 |
| N-ethylperfluoro-1-octanesulfonamide | NEtPFOSA | 4151-50-2 |
| 2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid | HFPODA | 13252-13-6 |
| 4,8-dioxa-3H-Perfluorononanoic acid | DONA ** | 919005-14-4 * |
| 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid | 9Cl-PF3ONS | 756426-58-1 * |
| 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid | 11Cl-PF3OUdS | 763051-92-9 * |
| 3-Perfluoropropylpropanoic acid | 3:3 FTCA | 356-02-5 |
| 3-Perfluoropentylpropanoic acid | 5:3 FTCA | 914637-49-3 |
| 3-Perfluoroheptylpropanoic acid | 7:3 FTCA | 812-70-4 |
| Perfluoro-3-methoxypropanoic acid | PFMPA/PFECA F | 377-73-1 |
| Perfluoro-4-methoxybutanoic acid | PFMBA/PFECA A | 863090-89-5 |
| Nonafluoro-3,6-dioxaheptanoic acid | NFDHA/PFECA B | 151772-58-6 |
| Perfluoro(2-ethoxyethane)sulfonic acid | PFEESA/PES | 113507-82-7 |

* = CAS number for the free acid form of the analyte

** = The acronym for the free acid form of the analyte

Basic Principles

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A soil sample is fortified with isotopically-labeled extraction standards, and extracted for an hour with acetonitrile:methanol mixture (1:1) using ultrasonic extraction. The sample extracts are vortexed and centrifuged. A 2.0 mL portion of supernatant is transferred and concentrated with nitrogen in a heated water bath and then reconstituted to 1.0 mL with methanol. The sample extract is analyzed by LC/MS/MS operated in negative electrospray ionization (ESI) mode for detection and quantification of the analytes. Quantitative analysis is performed using isotope dilution.

Reference Modifications

EPA Method 537 is written specifically for the analysis of drinking water samples. Modifications are made to accommodate the preparation of soil samples which are not addressed in EPA 537 Version 1.1. In addition, the following modifications have been made.

1. A labeled isotopic analog is spiked into samples for all compounds where an isotopic analog is commercially available. These isotopic compounds are referred to as extraction standards. For those compounds, an isotope dilution calibration model is used. Where labeled isotopes are not available, an internal standard calibration model using the extraction standards is used.
2. Field reagent blanks are not processed as listed in EPA 537 Version 1.1 section 8.3.
3. Peak asymmetry factors are not calculated.
4. MRL confirmation is not performed.
5. Spike concentrations are not rotated between low, medium, and high levels.

MDL studies and IDOCs have been performed to validate method performance.

Interferences

Compounds which have similar structures to the compounds of interest and similar molecular weights would potentially interfere. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, etc. A laboratory blank is performed with each batch of samples to demonstrate that the extraction system is free of contaminants.

Precaution to Minimize Method Interference

1. Proprietary Content
2. Proprietary Content

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- PFAS standards, extracts and samples should not come in contact with any glass containers as these analytes can potentially adsorb to glass surfaces. PFAS analytes and labeled extraction standards commercially purchased in glass ampules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be stored in polypropylene containers.

Safety Precautions and Waste Handling

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. PFOA has been described as "likely to be carcinogenic to humans". Each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized.

Exposure to these chemicals must be reduced to the lowest possible level by whatever means available, such as fume hoods, lab coats, safety glasses, and gloves. Gloves, lab coats, and safety glasses should be worn when preparing standards and handling samples. Avoid inhaling solvents and chemicals and getting them on the skin. Wear gloves when handling neat materials. When working with acids and bases, take care not to come in contact and to wipe any spills. Always add acid to water when preparing reagents containing concentrated acids.

All laboratory waste is accumulated, managed, and disposed of in accordance with all Federal, State, and local laws and regulations. All solvent waste and extracts are collected in approved solvent waste containers in the laboratory and subsequently emptied by personnel trained in hazardous waste disposal into the lab-wide disposal facility. HPLC vials are disposed of in the lab container for waste vials, and subsequently lab packed. Any solid waste material (disposable pipettes and broken glassware, etc.) may be disposed of in the normal solid waste collection containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC).

Each chemist performing the extraction must work with an experienced employee for a period of time until they can independently perform the extraction. Also, several batches of sample extractions must be performed under the direct observation of another experienced chemist to assure the trainee is capable of independent preparation. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

Each LC/MS/MS analyst must work with an experienced employee for a period of time until they can independently calibrate the LC/MS/MS, review and process data, and perform maintenance procedures. Proficiency is measured through a documented Initial Demonstration of Capability (IDOC).

The IDOC and DOC consist of four laboratory control samples (or alternatively, one blind sample for the DOC) that is carried through all steps of the extraction and meets the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

See [QA-SOP11178](#) for additional information on IDOC and DOC.

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Sample Collection, Preservation, and Handling

A. Sample Collection

The samples are collected in 250-mL polyethylene bottles with polyethylene screw caps. Keep the sample sealed from time of collection until extraction.

NOTE: PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

B. Sample Storage and Shipment

1. Samples must be chilled during shipment and must not exceed 10°C during the first 48 hours after collection. Sample temperature must be confirmed to be at or below 10°C when the samples are received at the laboratory.
2. Samples stored in the lab must be held at a temperature of 0° to 6°C, not frozen, until extraction.
3. Samples must be extracted within 28 days of collection. Extracts must be analyzed within 28 days after extraction. Extracts are stored at room temperature.

Apparatus and Equipment

A. Apparatus

1. Centrifuge tubes – 15-mL conical polypropylene with polypropylene screw caps; Fisher, Cat. No. 05-539-5 or equivalent
2. Centrifuge tubes - 50-mL – conical, polypropylene with polypropylene screw caps; Fisher, Cat. No. 06-443-21, or equivalent.
3. 10-mL polypropylene volumetric flask, class A – Fisher Scientific, Cat. No. S02288, or equivalent.
4. HPDE bottles for extraction fluid storage: L; Environmental Sampling Supply, Cat. No. 1000-1902-PC
5. Auto-dispenser/Pump: Dispensette S Analog bottletop dispenser, 5-50ml; Fisher Cat. No. 13-689-017.
6. Analytical Balance – Capable of weighing to 0.0001 g
7. Top-Loading Balance – Capable of weighing to 0.01 g
8. Centrifuge – “Q-Sep 3000”; Restek Corp. Cat. No. 26230 or equivalent, capable of minimum rotational speed of 3000 rpm.

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9. Disposable polyethylene pipette – Fisher Scientific, Cat. No. S30467-1 or equivalent.
10. Auto Pipettes – Eppendorf; capable of accurately dispensing 10- to 1000- μ L. FisherScientific cat # 14-287-150, or equivalent.
11. Polypropylene pipette tips: 0-200 μ L. Fisher Cat. No. 02-681-135
12. Polypropylene pipette tips: 101-1000 μ L. Fisher, Cat. No. 02-707-508
13. Pipettes – Disposable transfer. Fisher, Cat. No. 13-711-7M
14. Disposable polypropylene spatulas – VWR; Cat. No. 80081-190.
15. Vortex mixer, variable speed, Fisher Scientific or equivalent
16. Proprietary Content
17. Ultrasonic Bath – Branson; Model 3800 or 5800, or equivalent.
18. Reagent Water Purification System: Capable of producing ultrapure “Type 1/Milli-Q”-grade water from in-house deionized water system. Millipore SAS; Cat. No. FTPF08831.
19. Proprietary Content
20. Proprietary Content
21. Wooden Tongue Depressors - Fisher; Cat. # 11-700-555, or equivalent.
22. Stainless steel spatula/scoop set. Bel-Art SP Scienceware; Product # 11-865-130

B. Equipment

1. AB Sciex Triple Quad 4500/5500/5500 Plus Turbo V Ion Source
 - ExionLC Controller
 - ExionLC AC Pump
 - ExionLC AC Autosampler
 - Exion AC Column Oven
 - Data system –Analyst 1.7
2. HPLC columns
 - a. Proprietary Content
 - b. Proprietary Content

Reagents and Standards

A. Reagents:

| | | |
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1. Proprietary Content
2. Proprietary Content
3. Proprietary Content
4. Proprietary Content
5. Proprietary Content
6. Proprietary Content
7. Graphitized Non-Porous Carbon - Supelco/Millipore Sigma Superclean ENVI-Carb SPE Bulk Packing; Cat. No. 57210-U, or equivalent.

B. Standards: See SOP [T-PFAS-WI13881](#)

Calibration

A. Initial Calibration

1. A minimum of five calibration standards are required. In general, Cal1, Cal2, Cal3, Cal4, Cal5, Cal6, and Cal7 are included in the initial calibration. The calibration standards contain the branched isomers for PFHxS, PFOS, NMeFOSAA and NEtFOSAA. S/N ratio must be $\geq 10:1$ for all ions used for quantification.
2. Analyze a Cal3 level standard that contains linear and branch chained isomers of PFOA. The analysis of this standard is used to demonstrate where the branch chained isomers elute and not included in the calibration curve. This will assist the chemist in identifying and properly integrating this compound in samples.
3. Isotopically-labeled compounds are not available for PFPeS, PFHpS, PFNS, PFDS, PFDoS, 10:2-FTS, PFTrA, PFHxDA, PFODA, DONA, 9Cl-PF3ONS, 11Cl-PF3OUdS, 3:3FTCA, 5:3FTCA, 7:3FTCA, PFEESA, PFMPA, PFMBA, and NFDHA. See below for referenced extraction standards. See [Attachment 2](#) for additional information about compound relationships.

| Compound | Extraction standard |
|----------|---------------------|
| 10:2-FTS | 13C2-8:2-FTS |
| PFTrDA | 13C2-PFDoDA |
| PFHxDA | 13C2-PFTeDA |

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| | |
|--------------|------------|
| PFODA | |
| PFPeS | 13C3-PFBS |
| PFHpS | 13C3-PFHxS |
| DONA | 13C4-PFHpA |
| PFNS | 13C8-PFOS |
| PFDS | |
| PFDoS | |
| 9CI-PF3ONS | |
| 11CI-PF3OUdS | |
| 3:3FTCA | 13C5-PFPeA |
| PFMPA | |
| PFMBA | |
| 5:3FTCA | 13C5-PFHxA |
| 7:3FTCA | |
| PFEESA | |
| NFDHA | |

4. Fit the curve

- a. If the % RSD for the response factors is less than or equal 20%, the average response factor (Ave RRF) can be used to quantitate the data.
- b. If the %RSD is greater than 20%, then a linear regression with a concentration weighing factor of $1/x$ forced through zero is tried for the compounds not meeting the criteria in 4.a. R^2 for each analyte using the linear regression must be greater than or equal to 0.99.
- c. If the linear regression curve fails, then a quadratic regression with a concentration weighing factor $1/x^2$ is tried for the compounds not meeting 4.a or 4.b. R^2 for each analyte using the quadratic regression must be greater than or equal to 0.99. A minimum of six standards must be analyzed to use a quadratic fit.
- d. For all curve fits, each calibration point is calculated back against the curve. The back calculated concentration should be within $\pm 30\%$ of its true value.
- e. If the criteria are not met, the source of the problem must be determined and corrected. Situations may exist where the initial calibration can be used. In those cases, the data will be reported with a qualifying comment.

NOTE: The concentrations referenced for the sulfonate salts, (for example PFBS, PFHxS and PFOS) have already been corrected to the acid form by the standards supplier as noted in the example Certificate of Analysis (CofA). See [Attachment 4](#).

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5. Initial Calibration Verification (ICV)

A check standard prepared from a second source (ICV) is injected to confirm the validity of the calibration curve/standard. If a second source is not available, a separate preparation from the same stock may be used. The calculated amount for each analyte must be within $\pm 30\%$ of the true value. If this criteria is not met, re-inject or remake the standard. If the criteria is still not met, recalibration is necessary. Instrument maintenance may be needed prior to recalibrating.

B. Continuing calibration

1. Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a continuing calibration verification (CCV) standard every ten field samples and at the end of the analysis sequence.
 - a. The CCV run after the initial calibration must be at the CAL3 level.
 - b. The CCV standards must alternate between the CAL2, CAL3, and CAL4 levels.
2. Acceptance criteria
 - a. The calculated amount for each native compound in the CCV standard must be within $\pm 30\%$ of the true value. Samples that are not bracketed by acceptable CCV analyses must be reanalyzed.
 - b. The absolute areas of the extraction standards for the opening CCV on the day following the ICAL should be within 50-150% of the areas measured during the initial calibration CAL3 standard. On subsequent days where an ICAL is not performed, the absolute areas of the extraction standards from opening CCV are compared to the absolute areas of the extraction standards from the previous day's opening CCV and should be within 50-150%.
 - c. If acceptance criteria are not met, immediately analyze two additional consecutive CCVs. If both pass acceptance criteria, samples may be reported without reanalysis. If either fails, or two consecutive CCVs cannot be run, repeat the CCV and reanalyze all samples since the last successful CCV.

Procedure

A. Sample preparation

NOTE: Prior to weighing out samples, thoroughly mix each sample using a wooden tongue depressor to ensure a homogeneous sample matrix.

1. On a calibrated, top-loading balance, accurately weigh $1.0g \pm 0.10g$ of solid sample into a tared, labeled 15-mL centrifuge tube using a disposable polypropylene spatula. Record sample weight on sample batch log and in the prep entry system.
2. For each batch - maximum 20 samples - include the following quality control samples:
 - a. Method Blank: Weigh $1.0g \pm 0.10g$ of sand.

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| | Always check on-line for validity. | Level:  |
| Document number: T-PFAS-WI36459 | Polyfluorinated Alkyl Substances (PFAS) in Solids by Method 537 Version 1.1 Modified QSM 5.4 Table B-15 Using LC/MS/MS | Work Instruction |
| Old Reference: | | |
| Version: 2 | | Organisation level: 5-Sub-BU |
| Approved by: XL3S Effective Date 25-MAY-2022 | Document users: 5_EUUSLA_PFAS_Manager, 6_EUUSLA_PFAS_Analyst, 6_EUUSLA_PFAS_Data_Reviewers, 6_EUUSLA_PFAS_Management_Team, 6_EUUSLA_PFAS_Sample_Prep | Responsible: 5_EUUSLA_PFAS_Manager |

- b. LCS/LCSD: Fortify 1.0g ± 0.10g of sand with 40 µL of Native Spiking Solution (MSMODSX).
 - c. Matrix Spike (MS): Fortify 1.0g ± 0.10g of sample as specified in sample preparation log with 40 µL of Native Spiking Solution (MSMODSX).
3. Add 50µL working labeled extraction surrogate solution to each sample/QC tube.
 4. Proprietary Content
 5. Proprietary Content
 6. Proprietary Content
 7. Proprietary Content
 8. Proprietary Content
 9. Proprietary Content
 10. Reconstitute extract to 1 mL with methanol. Vortex to thoroughly mix.

The instrument lab chemist performs the next steps.

11. Extract Clean Up: DoD requires all sample/QC extracts be treated with a carbon sorbent to remove potential interferences. Once the extracts have been brought to final volume 1 mL, (see step 10 above), 25 mg loose/bulk "ENVI-Carb" carbon sorbent is added to the extract in its centrifuge tube.

The sorbent is added with a stainless steel scoop such that, when a level scoop is measured, a 25 mg amount of sorbent is drawn. The 25 mg of sorbent is added directly to the extracts in the 15 mL centrifuge tube. The scoop is rinsed with MeOH and dried between samples to avoid cross-contamination.

After addition of the ENVI-Carb the extracts are centrifuged for a complete cycle and are ready for vialing into LC vials for analysis.

12. Transfer 400 µL of the final extract to the labeled auto-sampler vial. Cap and vortex the auto-sampler vial. Samples are now ready for analysis.
13. Cap the centrifuge tube. Store the remaining centrifuged extracts at room temperature for dilution or reinjection if needed.

B. LC/MS/MS Analysis

1. Mass Calibration and Tuning
 - a. At instrument set up and installation and after the performance of major maintenance, calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. The entire mass range must be calibrated.

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- b. When masses fall outside of the ± 0.5 amu of the true value, the instrument must be retuned using PPG according to the manufacturer's specifications. Mass assignments of the tuning standard must be within 0.5 amu of the true value. Refer to the instrument manufacturer's instructions for tuning and conditions. These values are stored in the tune file for future reference.
2. The mass spectral acquisition rate must include a minimum of 10 spectra scans across each chromatographic peak.

See the AB Sciex(4500/5500/5500 plus) Acquisition, Quantitation, Gradient, and detector condition files for the most up to date chromatographic conditions. Modifications to these conditions can be made at the discretion of the analyst to improve resolution or the chromatographic process.
3. Example Acquisition method: See [Proprietary Content](#).
4. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of PFBA, PFPeA, PFOSA, NMePFOSAE, NMEPFOSA, NEtPFOSAE, NEtPFOSA, PFDoS, DONA, 9Cl-PF3ONS, 11Cl-PF3OUdS, PFMPA, 3:3 FTCA, PFMBA, PFEESA, and 7:3 FTCA. See [Attachment 3](#).
5. Instrument Sensitivity Check (ISC) and Instrument Blanks
 - a. Prior to sample analysis and at least every 12 hours, an instrument sensitivity check (ISC) must be performed. The ISC standard concentration must be at the LOQ. The CAL1 standard's concentration is at the LOQ. The CAL1 standard will be analyzed. All analyte concentrations must be within $\pm 30\%$ of their true values. If the criteria is not met, correct problem and rerun ISC. If problem persists, repeat the ICAL. No samples can be analyzed until the ISC meets acceptance criteria.
 - b. Instrument blanks need to be analyzed immediately following the highest standard analyzed and daily or at the start of a sequence. The concentration of all analytes must be less than or equal to 1/2 the LOQ. If acceptance criteria are not met the calibration must be performed using a lower concentration standard for the high standard until the criteria are met.
6. Load sample vials containing standards, quality control samples, and sample extracts into autosampler tray. Allow the instrument adequate time to equilibrate to ensure the mass spec and LC have reached operating conditions (approximately 5 minutes) before the first injection. Analyze several solvent blanks clean the instrument prior to sample acquisition.
7. After the initial calibration, inject an instrument blank, followed by the ICV, Linear Branched (L/B) standard, closing Cal 3 level CCV, extraction batch QC, and samples. Bracket each set of ten samples with a CCV standard, alternating between the Cal2, Cal3, and Cal4 levels.
8. After injections are completed, check all CCV recoveries and absolute areas to make sure they are within method control limits. See Calibration section B.2 for acceptance criteria. Process each chromatogram and closely evaluate all integrations, baseline anomalies, and retention time differences. If manual integrations are performed, they must be documented and a reason given for the change in integrations. The manual integrations are documented during data processing and all original integrations are reported at the end of the sample PDF file with the reason for manual integration clearly listed.

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9. Quantitate results for the extraction blank. No target analytes detected greater than 1/2 the LOQ or greater than 1/10 the regulatory limit, whichever is greater. If the criteria is exceeded, reextract all samples with positive detections associated with the method blank. If the target analyte in the sample is detected at a concentration greater than 10 times the amount detected in the method blank, the data is reported.
10. Calculate the recoveries of spiked analytes for the LCS, matrix spike and matrix spike duplicate (MS/MSD) by comparing concentrations observed to the true values.
- Method defined limits are used for the evaluation of the LCS and MS/MSD recoveries. Where there are no limits stated, an advisory window of 70-130% will be used until sufficient data points are available to generate statistical windows. The QC acceptance limit for the relative percent difference (%RPD) between LCS/LCSD and MS/MSD is less than or equal to 30%.
 - If LCS and/or LCSD recoveries are acceptable, proceed to sample quantitation.
 - If the LCS recoveries are above QC acceptance criteria and there are no detections for the compound(s) in the associated sample(s), the data can be reported with a qualifying comment. In all other cases, the samples associated with the LCS must be reextracted.
 - If MS/MSD recoveries are outside QC acceptance criteria, the associated data will be flagged or noted in the comments section of the report.
11. Isotopically-labeled extraction standards are added to all samples, extraction blank, LCS/LCSD, and MS/MSD prior to extraction. The absolute areas of the extraction standards must be within $\pm 50\%$ of the areas measured in the ICAL mid-point standard (CAL3 standard). On days when an ICAL is not performed, the absolute areas must be within $\pm 50\%$ of the absolute areas measured in the daily opening initial CCV. If the extraction standards fall outside the acceptance window, analyze a second aliquot of the extract. If none remains, reanalyze the first aliquot.
- Refer to [Attachment 5](#) for the Eurofins DOD variances for isotopically-labeled recoveries. If these variances are accepted by the client, the laboratory will follow these guidelines in determining when to re-extract samples. If the client does not accept these variances, samples with recoveries that are outside the $\pm 50\%$ of the true value acceptance criteria must be re-extracted.
12. Compare the retention times of all of the analytes and extraction standards to the retention time from the initial calibration. The retention times should not vary from the expected retention time by more than
- 0.4 minutes for isotopically-labeled compounds
 - 0.1 minutes from their analog for native compounds with an exact isotopically-labeled compound
 - 0.4 minutes from their assigned analog for native compounds without an exact isotopically-labeled compound.

If the retention time is outside of the criteria, the compound is considered a false positive unless it is a compound with branched isomers. Compounds with branched isomers can vary in intensity of the individual isomers that are used for reporting and must be reviewed and compared to the preceding CCV to determine if it should be reported.

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13. Two ion transitions and the ion transition ratio per analyte shall be monitored and documented with the exception of PFBA, PFPeA, PFOSA, NMePFOSAE, NMEPFOSA, NETPFOSAE, NETPFOSA, PFDoS, DONA, 9CI-PF3ONS, 11CI-PF3OUdS, PFMPA, 3:3 FTCA, PFMBAs, PFEESA, and 7:3 FTCA. The expected ion ratio for each compound is calculated by using the average of ion ratios of each compound from initial calibration standards. When an ion ratio for a compound differs from the expected ion ratio by more than 50%, a qualifier is placed on the raw data and on the sample report. No corrective action is required.
14. The linear/branch chain standard is used when assessing the correctness of the computer generated peak integrations for PFOA.
15. If the calculated concentration exceeds the calibration range of the system, determine the appropriate dilution required and dilute the extract with MeOH. If the sample dilution required exceeds 100 fold, the client must be contacted to determine if the data can be reported with result(s) that exceed the calibration range or if the sample should be re-prepped at a reduced volume.

Dilution Example 1/10: Mix 900 µL of MeOH with 100µL of sample extract. Vortex to mix. Using an auto-pipette, transfer 400µL of the mixed solution into a labeled auto-sampler vial containing a plastic insert. Cap and vortex thoroughly to mix.

Calculations

A. Peak Area Ratio

$$\text{Peak Area Ratio} = \frac{\text{Analyte Response}}{\text{Labeled Analyte Response}}$$

B. On-Column Analyte Concentration using linear through zero curves

On-column Concentration = (peak area ratio x Isotope Dilution Analyte concentration) ÷ slope

C. Sample Concentration

Sample concentration (ng/g) = (On-column concentration x Final Sample volume x DF x prep factor) ÷ Sample weight

Where: prep factor = 2

D. Ion Ratio

ion ratio = (peak area or height of quantifier)/(peak area or height of qualifier)

E. See [T-PEST-WI9847](#) for additional calculations used to evaluate the calibrations and quality control samples.

Statistical Information/Method Performance

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The LCS should contain all compounds of interest. LCS, MS, extraction standard recoveries and RPD are compared to the limits stored on the LIMS. These limits are defined by the method. For compounds not defined by the method, these limits are statistically derived when sufficient data points are available. If sufficient data points are not available to generate statistical windows, an advisory window of 70% to 130% will be used. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to [QA-SOP11892](#) for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the supervisor.

Quality Assurance/Quality Control

For each batch of 20 samples extracted, a method blank and an LCS/LCSD (Sand spiked with all compounds to be determined carried through the entire procedure) must be extracted and analyzed. If an MS/MSD is submitted then an LCSD would not be extracted. A batch is defined as the samples to be extracted on any given day, but not to exceed 20 field samples. If more than 20 samples are prepared in a day, an additional batch must be prepared. If any client, state, or agency has more stringent QC or batching requirements, these must be followed.

Attachment:

- [Attachment 1 – Proprietary Content](#)
- [Attachment 2 - Compound Relationships \(.docx\)](#)
- [Attachment 3 - Mass Transitions \(.doc\)](#)
- [Attachment 4 - Example Certificate of Analysis \(.pdf\)](#)
- [Attachment 5 - Eurofins DoD Variances \(.doc\)](#)

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- [G-DC-FRM23907 Redacted SOPs](#)
 - [QA-SOP11178 Demonstrations of Capability](#)
 - [QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)
 - [T-PEST-WI9847 Common Equations Used During Chromatographic Analyses](#)
 - [T-PFAS-WI13881 Standards Management in the PFAS Laboratory](#)
 - [Attachment: Attachment 1 – Proprietary Content](#)
 - [Attachment: Attachment 2 - Compound Relationships \(docx\)](#)
 - [Attachment: Attachment 3 - Mass Transitions \(doc\)](#)
 - [Attachment: Attachment 4 - Example Certificate of Analysis \(pdf\)](#)
 - [Attachment: Attachment 5 - Eurofins DoD Variances \(doc\)](#)

End of document

Version history

| Version | Approval | Revision information |
|---------|-------------|----------------------|
| 1 | 29.JAN.2021 | |
| 2 | 25.MAY.2022 | |

Attachment 2

| |
|---|
| PFAS Native Compounds/Extraction Standards |
|---|

Native PFAS Compounds

| Native | Extraction Standard |
|--------------|---------------------|
| PFBA | 13C4-PFBA |
| PFPeA | 13C5-PFPeA |
| 3:3FTCA | |
| PFMPA | |
| PFMBA | |
| PFBS | 13C3-PFBS |
| PFPeS | |
| 4:2-FTS | 13C2-4:2-FTS |
| PFHxA | 13C5-PFHxA |
| NFDHA | |
| 5:3FTCA | |
| 7:3FTCA | |
| PFEESA | |
| PFHxS | 13C3-PFHxS |
| PFHpS | |
| PFHpA | 13C4-PFHpA |
| DONA | |
| 6:2-FTS | 13C2-6:2-FTS |
| PFOA | 13C8-PFOA |
| PFOS | 13C8-PFOS |
| PFNS | |
| PFDS | |
| 9Cl-PF3ONS | |
| 11Cl-pf3OUdS | |
| PFDoS | |
| PFNA | 13C9-PFNA |
| PFOSA | 13C8-PFOSA |
| PFDA | 13C6-PFDA |

| Native | Extraction Standard |
|---------------------|----------------------------|
| 8:2-FTS | 13C2-8:2-FTS |
| 10:2-FTS | |
| NMePFOSAE | d7-NMePFOSAE |
| NMePFOSA | d3-NMePFOSA |
| NMeFOSAA | d3-NMeFOSAA |
| NEtPFOSAE | d9-NEtPFOSAE |
| NEtPFOSA | d5-NEtPFOSA |
| PFUnDA | 13C7-PFUnDA |
| NEtFOSAA | d5-NEtFOSAA |
| PFD _o DA | 13C2-PFD _o DA |
| PFTrDA | |
| PFTeDA | 13C2-PFTeDA |
| PFH _x DA | |
| PFODA | |
| HFPODA | 13C3-HFPODA |

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Attachment 3

Mass Transitions AB Sciex 4500/5500/5500+

| Compound | Parent Ion | Daughter Ion |
|--------------|------------|--------------|
| 13C4-PFBA | 217 | 172 |
| PFBA | 213 | 169 |
| 13C5-PFPeA | 268 | 223 |
| PFPeA | 263 | 219 |
| 13C3-PFBS | 302 | 80 |
| PFBS | 299 | 80 |
| PFBS (2) | 299 | 99 |
| 13C2-4:2-FTS | 329 | 81 |
| 4:2-FTS | 327 | 307 |
| 4:2-FTS (2) | 327 | 81 |
| 13C5-PFHxA | 318 | 273 |
| PFHxA | 313 | 269 |
| PFHxA (2) | 313 | 119 |
| PFPeS | 349 | 80 |
| PFPeS (2) | 349 | 99 |
| 13C3-PFHxS | 402 | 80 |
| PFHxS | 399 | 80 |
| PFHxS (2) | 399 | 99 |
| 13C4-PFHpA | 367 | 322 |
| PFHpA | 363 | 319 |
| PFHpA (2) | 363 | 169 |
| 13C2-6:2-FTS | 429 | 81 |
| 6:2-FTS | 427 | 407 |
| 6:2-FTS (2) | 427 | 81 |
| PFHpS | 449 | 80 |
| PFHpS (2) | 449 | 99 |
| 13C8-PFOA | 421 | 376 |
| PFOA | 413 | 369 |
| PFOA (2) | 413 | 169 |
| 13C8-PFOS | 507 | 80 |
| PFOS | 499 | 80 |
| PFOS (2) | 499 | 99 |
| 13C9-PFNA | 472 | 427 |
| PFNA | 463 | 419 |
| PFNA (2) | 463 | 169 |
| 13C8-PFOSA | 506 | 78 |
| PFOSA | 498 | 78 |

Attachment 3

| Compound | Parent Ion | Daughter Ion |
|--------------|------------|--------------|
| PFNS | 549 | 80 |
| PFNS (2) | 549 | 99 |
| 13C6-PFDA | 519 | 474 |
| PFDA | 513 | 469 |
| PFDA (2) | 513 | 169 |
| 13C2-8:2-FTS | 529 | 81 |
| 8:2-FTS | 527 | 507 |
| 8:2-FTS (2) | 527 | 81 |
| d7-NMePFOSAE | 623 | 59 |
| NMePFOSAE | 616 | 59 |
| d3-NMePFOSA | 515 | 169 |
| NMEPFOSA | 512 | 169 |
| d3-NMeFOSAA | 573 | 419 |
| NMeFOSAA | 570 | 419 |
| NMeFOSAA (2) | 570 | 483 |
| d9-NEtPFOSAE | 639 | 59 |
| NEtPFOSAE | 630 | 59 |
| d5-NETPFOSA | 531 | 169 |
| NEtPFOSA | 526 | 169 |
| PFDS | 599 | 80 |
| PFDS (2) | 599 | 99 |
| 13C7-PFUnDA | 570 | 525 |
| PFUnDA | 563 | 519 |
| PFUnDA (2) | 563 | 169 |
| d5-NEtFOSAA | 589 | 419 |
| NEtFOSAA | 584 | 419 |
| NEtFOSAA (2) | 584 | 526 |
| 13C2-PFDoDA | 615 | 570 |
| PFDoDA | 613 | 569 |
| PFDoDA (2) | 613 | 169 |
| 10:2-FTS | 627 | 607 |
| 10:2-FTS (2) | 627 | 81 |
| PFDoS | 699 | 80 |
| PFTrDA | 663 | 619 |
| PFTrDA (2) | 663 | 169 |
| 13C2-PFTeDA | 715 | 670 |
| PFTeDA | 713 | 669 |
| PFTeDA (2) | 713 | 169 |
| PFHxDA | 813 | 769 |
| PFHxDA (2) | 813 | 169 |

Attachment 3

| Compound | Parent Ion | Daughter Ion |
|--------------------|------------|--------------|
| PFODA | 913 | 869 |
| PFODA (2) | 913 | 169 |
| 13C3-HFPODA | 332 | 287 |
| HFPODA | 329 | 285 |
| HFPODA (2) | 285 | 169 |
| DONA | 377 | 251 |
| 9Cl-PF3ONS | 531 | 351 |
| 11Cl-PF3OUdS | 631 | 451 |
| PFECA B (NFDHA) | 201 | 85 |
| PFECA B(NFDHA) (2) | 295 | 201 |
| PFECA F (PFMPA) | 229 | 85 |
| 3:3 FTCA | 241 | 177 |
| PFECA A (PFMBA) | 279 | 85 |
| PFEESA (PES) | 315 | 135 |
| 5:3 FTCA | 341 | 237 |
| 5:3 FTCA (2) | 339 | 217 |
| 7:3 FTCA | 441 | 317 |

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PFAC-MXC

Native Perfluorinated Compound Solution/Mixture

| | |
|---|-------------------------------------|
| <u>PRODUCT CODE:</u> | PFAC-MXC |
| <u>LOT NUMBER:</u> | PFACMXC0617 |
| <u>SOLVENT(S):</u> | Methanol / Water (<1%) |
| <u>DATE PREPARED:</u> (mm/dd/yyyy) | 06/14/2017 |
| <u>LAST TESTED:</u> (mm/dd/yyyy) | 03/19/2019 |
| <u>EXPIRY DATE:</u> (mm/dd/yyyy) | 03/19/2024 |
| <u>RECOMMENDED STORAGE:</u> | Store ampoule in a cool, dark place |

DESCRIPTION:

PFAC-MXC is a solution/mixture of thirteen native perfluoroalkylcarboxylic acids (C₄-C₁₄, C₆, and C₈) and eight native perfluoroalkylsulfonates (C₄-C₁₄ and C₆, J). The full name, abbreviation and concentration for each of the components are given in Table A.

The individual perfluoroalkylcarboxylic acids and perfluoroalkylsulfonates all have chemical purities of >98%.

DOCUMENTATION/ DATA ATTACHED:

Table A: Components and Concentrations of the Solution/Mixture
Figure 1: LC/MS Data (SIR)
Figure 2: LC/MS/MS Data (Selected MRM Transitions)

ADDITIONAL INFORMATION:

See page 2 for further details.

Contains 4 mole eq. of NaOH to prevent conversion of the carboxylic acids to their respective methyl esters.

FOR LABORATORY USE ONLY: NOT FOR HUMAN OR DRUG USE

ATTACHMENT 4

INTENDED USE:

The products prepared by Wellington Laboratories Inc. are for laboratory use only. This certified reference material (CRM) was designed to be used as a standard for the identification and/or quantification of the specific chemical compounds it contains.

HANDLING:

This product should only be used by qualified personnel familiar with its potential hazards and trained in the handling of hazardous chemicals. Due care should be exercised to prevent unnecessary human contact or ingestion. All procedures should be carried out in a well-functioning fume hood and suitable gloves, eye protection, and clothing should be worn at all times. Waste should be disposed of according to national and regional regulations. Safety Data Sheets (SDSs) are available upon request.

SYNTHESIS / CHARACTERIZATION:

Our products are synthesized using single-product unambiguous routes whenever possible. They are then characterized, and their structures and purities confirmed, using a combination of the most relevant techniques, such as NMR, GC/MS, LC/MS/MS, SFC/UV/MS/MS, x-ray crystallography, and melting point. Isotopic purities of mass-labelled compounds are also confirmed using HRGC/HRMS and/or LC/MS/MS.

HOMOGENEITY:

Prior to solution preparation, crystalline material is tested for homogeneity using a variety of techniques (as stated above) and its solubility in a given diluent is taken into consideration. Duplicate solutions of a new product are prepared from the same crystalline lot and, after the addition of an appropriate internal standard, they are compared by GC/MS, LC/MS/MS, and/or SFC/UV/MS/MS. The relative response factors of the analyte of interest in each solution are required to be <5% RSD. New solution lots of existing products, as well as mixtures and calibration solutions, are compared to older lots in a similar manner. This further confirms the homogeneity of the crystalline material as well as the stability and homogeneity of the solutions in the storage containers. In order to maintain the integrity of the assigned value(s), and associated uncertainty, the dilution or injection of a subsample of this product should be performed using calibrated measuring equipment.

UNCERTAINTY:

The maximum combined relative standard uncertainty of our reference standard solutions is calculated using the following equation:

The combined relative standard uncertainty, $u_c(y)$, of a value y and the uncertainty of the independent parameters x_1, x_2, \dots, x_n on which it depends is:

$$u_c(y(x_1, x_2, \dots, x_n)) = \sqrt{\sum_{i=1}^n \left(\frac{\partial y}{\partial x_i} u(x_i) \right)^2}$$

where x is expressed as a relative standard uncertainty of the individual parameter.

The individual uncertainties taken into account include those associated with weights (calibration of the balance) and volumes (calibration of the volumetric glassware). An expanded maximum combined percent relative uncertainty of $\pm 5\%$ (calculated with a coverage factor of 2 and a level of confidence of 95%) is stated on the Certificate of Analysis for all of our products.

TRACEABILITY:

All reference standard solutions are traceable to specific crystalline lots. The microbalances used for solution preparation are regularly calibrated by an external ISO/IEC 17025 accredited laboratory. In addition, their calibration is verified prior to each weighing using calibrated external weights traceable to an ISO/IEC 17025 accredited laboratory. All volumetric glassware used is calibrated, of Class A tolerance, and traceable to an ISO/IEC 17025 accredited laboratory. For certain products, traceability to international interlaboratory studies has also been established.

EXPIRY DATE / PERIOD OF VALIDITY:

Ongoing stability studies of this product have demonstrated stability in its composition and concentration, until the specified expiry date, in the unopened ampoule. Monitoring for any degradation or change in concentration of the listed analyte(s) is performed on a routine basis.

LIMITED WARRANTY:

At the time of shipment, all products are warranted to be free of defects in material and workmanship and to conform to the stated technical and purity specifications.

QUALITY MANAGEMENT:

This product was produced using a Quality Management System registered to the latest versions of ISO 9001 by SAI Global, ISO/IEC 17025 by the Canadian Association for Laboratory Accreditation Inc. (CALA; A 1226), and ISO 17034 by ANSI-ASQ National Accreditation Board (ANAB; AR-1523).



**For additional information or assistance concerning this or any other products from Wellington Laboratories Inc.,

ATTACHMENT 4

Table A: PFAC-MXC; Components and Concentrations (ng/ml, ± 5% in Methanol/ Water (<1%))

| Compound | Abbreviation | Concentration (ng/ml)* | | Peak Assignment in Figure 1 |
|---------------------------------------|--------------|------------------------|--------------|-----------------------------|
| | | As the salt | As the anion | |
| Perfluoro-n-butanoic acid | PFBA | 2000 | | A |
| Perfluoro-n-pentanoic acid | PFPeA | 2000 | | B |
| Perfluoro-n-hexanoic acid | PFHxA | 2000 | | D |
| Perfluoro-n-heptanoic acid | PFHpA | 2000 | | F |
| Perfluoro-n-octanoic acid | PFOA | 2000 | | H |
| Perfluoro-n-nonanoic acid | PFNA | 2000 | | J |
| Perfluoro-n-decanoic acid | PFDA | 2000 | | L |
| Perfluoro-n-undecanoic acid | PFUdA | 2000 | | N |
| Perfluoro-n-dodecanoic acid | PFDoA | 2000 | | P |
| Perfluoro-n-tridecanoic acid | PFTrDA | 2000 | | Q |
| Perfluoro-n-tetradecanoic acid | PFTeDA | 2000 | | S |
| Perfluoro-n-hexadecanoic acid | PFHxDA | 2000 | | T |
| Perfluoro-n-octadecanoic acid | PFODA | 2000 | | U |
| Compound | Abbreviation | Concentration (ng/ml)* | | Peak Assignment in Figure 1 |
| | | As the salt | As the anion | |
| Potassium perfluoro-1-butanesulfonate | L-PFBS | 2000 | 1770 | C |
| Sodium perfluoro-1-pentanesulfonate | L-PFPeS | 2000 | 1880 | E |
| Sodium perfluoro-1-hexanesulfonate | L-PFHxS | 2000 | 1890 | G |
| Sodium perfluoro-1-heptanesulfonate | L-PFHpS | 2000 | 1900 | I |
| Sodium perfluoro-1-octanesulfonate | L-PFOS | 2000 | 1910 | K |
| Sodium perfluoro-1-nonanesulfonate | L-PFNS | 2000 | 1920 | M |
| Sodium perfluoro-1-decanesulfonate | L-PFDS | 2000 | 1930 | O |
| Sodium perfluoro-1-dodecanesulfonate | L-PFDoS | 2000 | 1940 | R |

* Concentrations have been rounded to three significant figures.

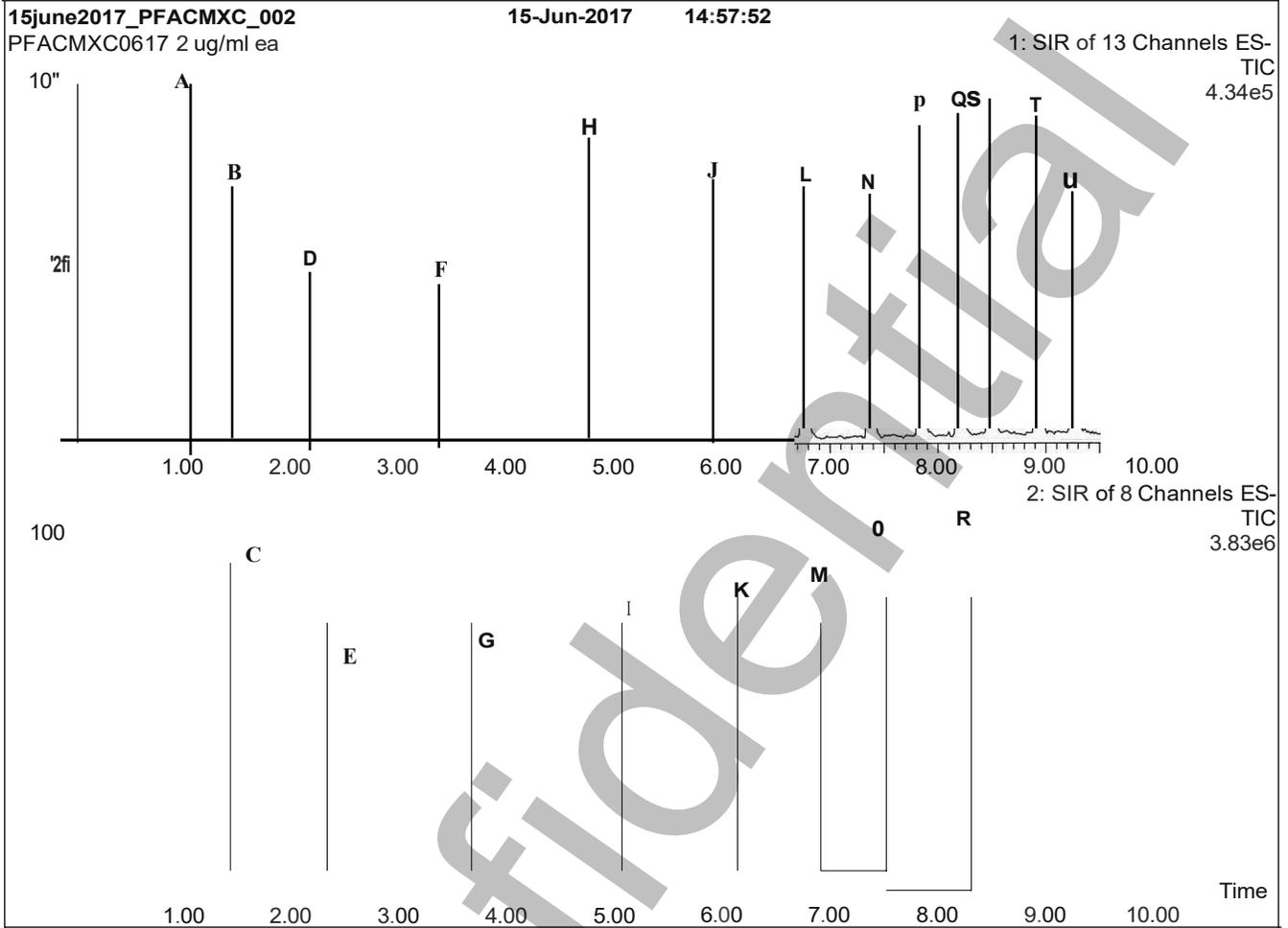
Certified By: _____

B.G. Chittim, General Manager

Date: 06/06/2019
(mm/dd/yyyy)

ATTACHMENT 4

Fi.9yr!L1 PFAC-MXC; LC/MS Data (Total Ion Current Chromatogram; SIR)



Conditions for Figure 1:

LC: Waters Acquity Ultra Performance LC
MS: Micromass Quattro *micro* API MS

Chromatographic Conditions

Column: Acquity UPLC BEH Shield RP.,
 1.7 μ m, 2.1 x 100 mm

Mobile phase: Gradient
 Start: 50% H₂O / 50% (80:20 MeOH:ACN)
 (both with 10 mM NH₄OAc buffer)
 Ramp to 90% organic over 8 min and hold for 2 min
 before returning to initial conditions in 1 min.

Time: 12 min

Flow: 300 μ l/min

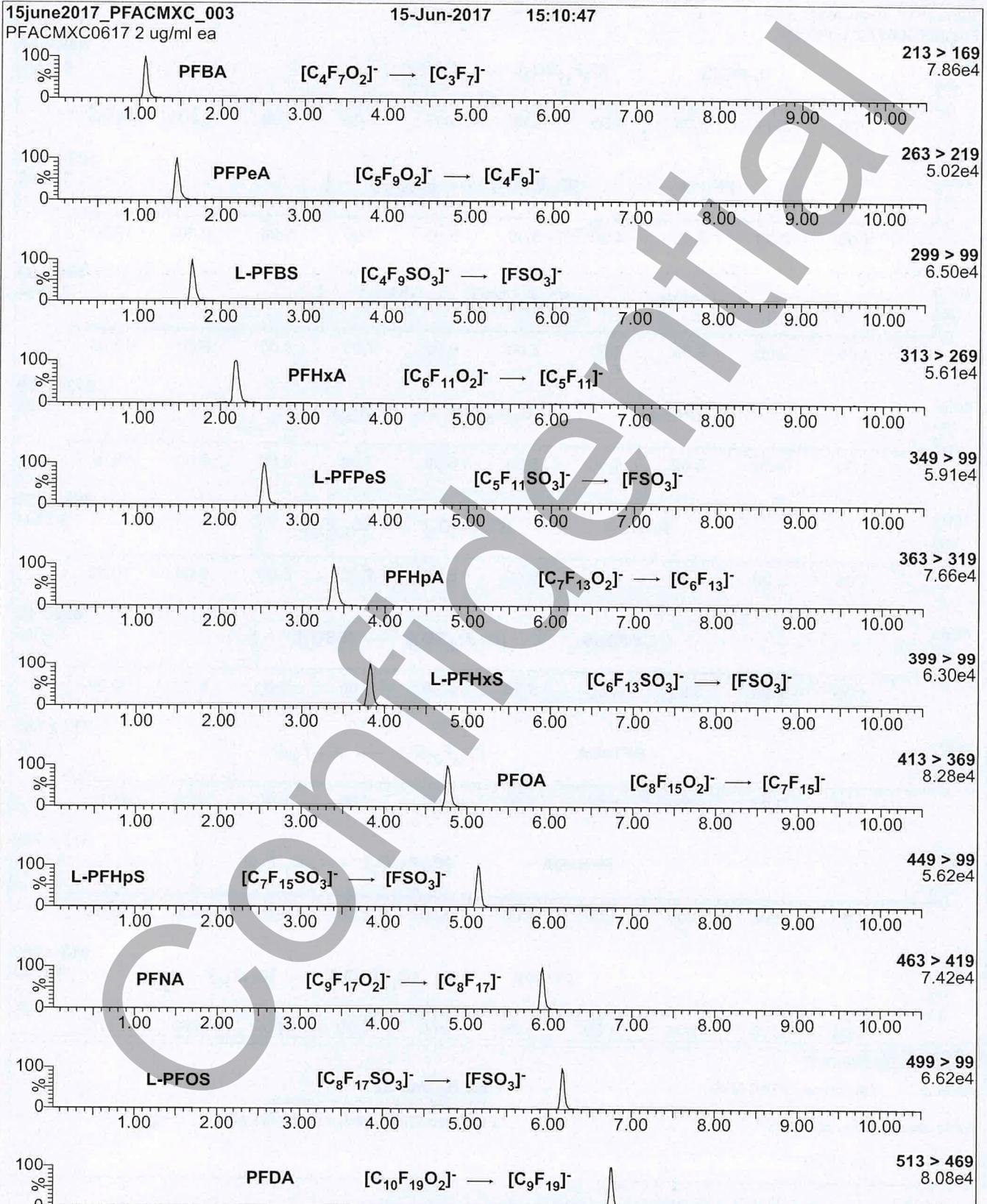
MS Parameters

Experiment: SIR of 21 Channels

Source: Electrospray (negative)
 Capillary Voltage (kV) = 3.00
 Cone Voltage (V) = variable (10-80)
 Cone Gas Flow (l/hr) = 50
 Desolvation Gas Flow (l/hr) = 750

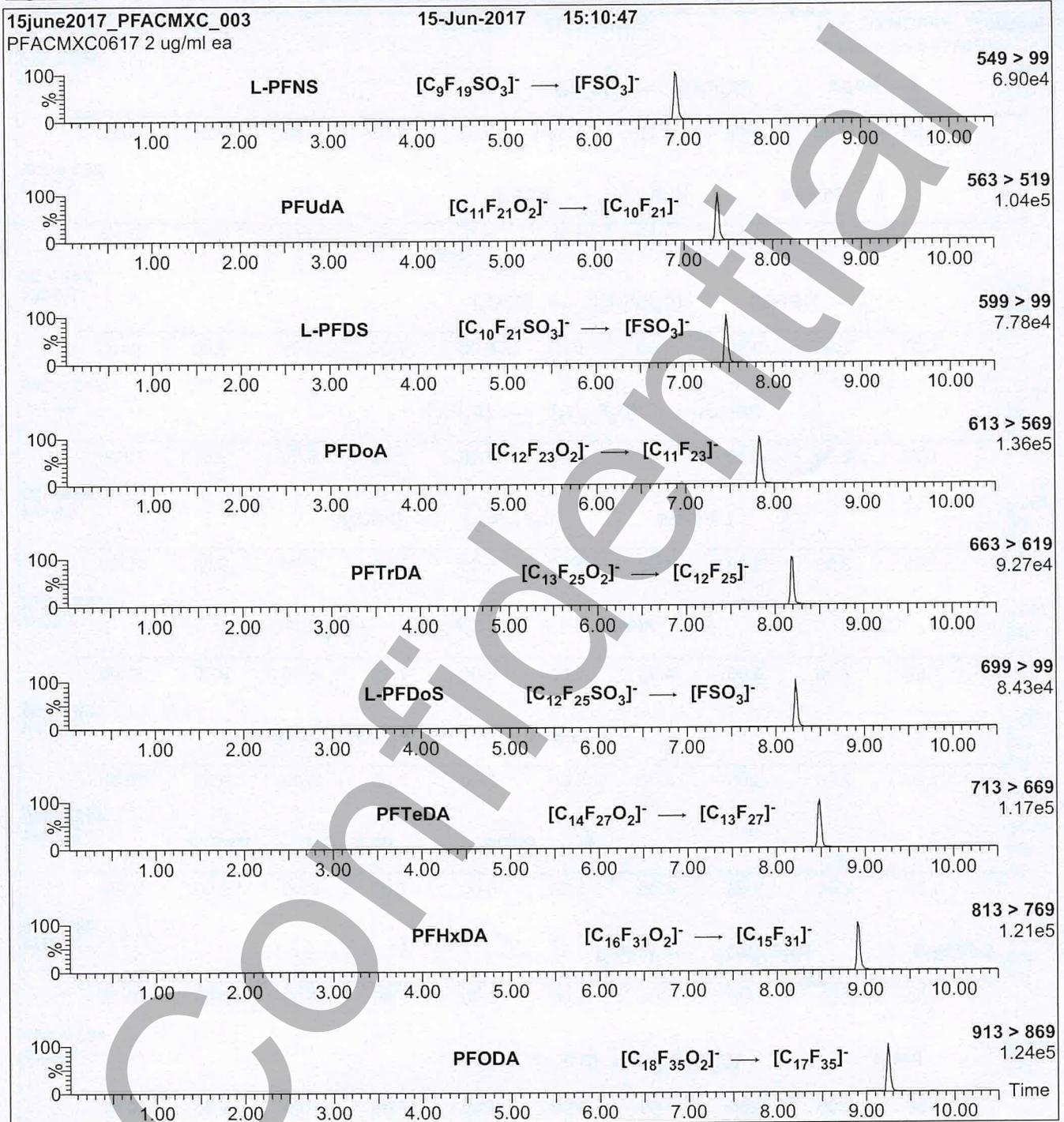
ATTACHMENT 4

Figure 2: PFAC-MXC; LC/MS/MS Data (Selected MRM Transitions)



ATTACHMENT 4

Figure 2: PFAC-MXC; LC/MS/MS Data (Selected MRM Transitions)



Conditions for Figure 2:

Injection: On-column (PFAC-MXC)
 Mobile phase: Same as Figure 1
 Flow: 300 μ l/min

MS Parameters

Collision Gas (mbar) = 3.46e-3
 Collision Energy (eV) = 8-50 (variable)

Eurofins Lancaster Laboratories Environmental, LLC Variance Requests

| Item | Parameter | Scope of Work | ELLE SOP | Approval |
|------|---|--|--|----------|
| 1 | QSM 5.3 Table B-15 PFAS Using LC/MS/MS | Extraction internal standard (EIS) recovery criteria of 50- 150% | For all PFAS compounds if EIS recovery is <50%, we will check for laboratory error and correct if identified. If no laboratory error is identified, additional corrective action will be performed if EIS recovery is <10%. If EIS recovery is >10% and samples have detections above the reporting limit, no additional corrective action is performed. If EIS recovery is >10% and samples have no detections for associated native analytes, the native analyte response will be evaluated to confirm the validity of the reporting limit. Reporting limits will be proportionately increased as necessary and appropriate to ensure that reported values accurately reflect the sensitivity of the analysis. | |
| 2 | QSM 5.3 Table B-15 PFAS Using LC/MS/MS | Extraction internal standard (EIS) recovery criteria of 50- 150% | For all PFAS compounds, if EIS recovery is >150%, we will check for laboratory error and correct if identified. If no laboratory error is identified, additional corrective action will be performed only when field samples have detections above the reporting limits for the associated native target analytes and EIS recovery is >200%. | |
| 3 | QSM 5.3 Table B-15 PFAS Using LC/MS/MS | Sample Preparation | If persistent matrix effects are observed for multiple samples from a given project that necessitate a high rate of additional corrective actions, these matrix effects will be mitigated prior to sample preparation and analysis, typically by processing a smaller sample mass or volume. | |

| | | |
|--|---|--|
|  | Always check on-line for validity. TOC and TC in Solids and Sludges by Combustion by SM 5310B, EPA 415.1, SW-846 9060/9060A, Lloyd Kahn | Level:  Work Instruction |
| Document number: T-WC-WI11627 | | |
| Old Reference: 1-P-QM-WI-9013418 | | |
| Version: 17.1 | | Organisation level: 5-Sub-BU |
| Approved by: X6TJ Effective Date 06-JUL-2021 | Document users: 6_EUUSLA_Instrumental Water Quality _TOC Analyst, 6_EUUSLA_Instrumental Water Quality _TOC Verifier | Responsible: 5_EUUSLA_Instrumental Water Quality_Manager |

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Revision Log

| | | | |
|---------------------|------------------------|--|--------------|
| Revision: | 18 | Effective Date: | This version |
| Section | Justification | Changes | |
| Revision Log | Formatting requirement | Removed revision logs up to the previous version | |
| Reference | MUR | Added method reference to comply with MUR | |
| Throughout Document | LIMs update | Corrected IDs to match current LIMs | |

| | | | |
|------------------|------------------------|---|---------------|
| Revision: | 17 | Effective Date: | July 09, 2019 |
| Section | Justification | Changes | |
| Revision Log | Formatting requirement | Removed revision logs up to the previous version. | |
| Scope | Current Process | Removed LIMS analyses 6623 and 11763 | |

| Revision: | 17 | Effective Date: | July 09, 2019 |
|-------------------------|--------------------|--|---------------|
| Reference Modifications | Method Requirement | Removed 10% dup instead of a quadruplicate per batch | |
| Procedure 8. | Current Process | Removed the process of analyzing three blanks prior to calibration. | |
| Procedure 10. | Current Process | Removed the concentration of the CCV, this changes dependent on the CRM provided by the manufacturer | |
| QA/QC | Method Requirement | One quadruplicate per batch for Lloyd Kahn | |

Reference

1. Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005, Method 5310B-2011,
2. Standard Methods for the Examination of Water and Wastewater, Method 5310B-2014.
3. *Method 415.1, Methods for Chemical Analysis of Water and Wastes USEPA 600.*
4. *Test Methods for Evaluating Solid Wastes, SW-846 Method 9060, September 1986.*
5. Test Methods for Evaluating Solid Wastes, SW-846 Method 9060A, November 2004
6. *The Primacs SNC-100 Analyzer Manual, Skalar..*
7. *Determination of Total Organic Carbon in Sediment, U. S. EPA, Region II, July 27, 1988. ("Lloyd Kahn Method.").*
8. *Chemical Hygiene Plan, current version.*

Cross Reference

| Document | Document Title |
|-----------------------------|--|
| QA-SOP11892 | Determining Method Detection Limits and Limits of Quantitation |
| QA-SOP11896 | Establishing Control Limits |
| QA-SOP11188 | Reagents and Standards |

Scope

This method is applicable for the determination of total organic carbon (TOC) in soils and other samples not easily analyzed by the TOC waters method. The limit of quantitation (LOQ) for this method can be found in the analysis information file. Quantitative TOC results up to 1,000,000 mg/kg may be obtained by this method.

Basic Principles

TOC is determined by acidifying a sample and heating it to remove the TIC. An aliquot of sample (1 mg to 1 g) is weighed into a sample cup. The sample is then heated to 900°C for combustion of the remaining TOC. The resulting carbon dioxide from the TOC is detected by a nondispersive infrared (NDIR) detector that has been calibrated to directly display the mass of carbon dioxide detected. The mass is proportional to the mass of TOC in the sample. Samples analyzed by this method include solids such as soils or sediments, slurries, sludges, brines, and corrosives.

The TC measurement is identical to the TOC measurement, with the exception that there is not an acidification and heating step to remove the TIC.

TIC is the calculation subtracting the TOC soil result from the result obtained when analyzing the TC.

Reference Modifications

There is no referenced method for the determination of Total Carbon in solids the analysis references a modified TOC method. The calibration standards, QC standards, and LCS employed for this analysis are composed of only organic sources of carbon (Sucrose for the calibration and QC standards, and a purchased standard for the LCS solid). Therefore, the Total Organic Carbon content is equal to the Total Carbon content in these standards.

Interferences

Carbon is ubiquitous in nature. Therefore, extra care must be taken to avoid contamination of reagents, glassware, and any other materials that come in contact with the sample. Samples which are light in weight may need to be analyzed at smaller aliquots to fit within a sample cup. These samples are reported with an elevated LOQ.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

1. Normal laboratory practices for safety must be followed.
2. Extreme caution must be used when handling sample cups after they have been analyzed.
3. Discard acid waste in acid waste containers.
4. See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and a documented Demonstration of Capability.

Analysts are considered proficient when they have successfully completed a Demonstration of Capability for the analysis. A Demonstration of Capability consists of four laboratory control standards that are carried through all steps of the analysis and that meet the acceptance criteria for the LCS and LCSD. Documentation for these studies are in each individual's training records.

Demonstration of Capability is performed annually and is maintained in the analyst's training records.

Sample Collection, Preservation, and Handling

Samples must be collected and stored in glass containers unpreserved. Samples must be stored under refrigeration at 0° to 6°C, not frozen.

Because very small amounts (1 mg to 1 g) of sample are used for the analysis, the sample must be as homogeneous as possible.

The holding time for analysis by SM 5310 B, EPA 415.1 and SW-846 9060/9060A is 28 days. The holding time for analysis by the Lloyd Kahn method is 14 days.

Apparatus and Equipment

1. Skalar-Primacs SC100
2. PC and SNAcess software
3. 2-stage gas regulator (two required)
4. Sample cups
5. Fiber quartz wool

6. Analytical balance, capable of accurately weighing to 1. mg
7. Glassware – General laboratory glassware as needed for preparing reagents and standards
8. Microliter syringe, various volumes

Reagents and Standards

NOTE: All chemical used must be ACS reagent grade unless otherwise noted. Different volumes or weights may be used provided the ratios remain equivalent. See [QA-SOP11188](#), for the appropriate labeling and documentation of reagent and standard preparation.

1. Ultra pure nitrogen gas (60 psi)
2. Ultra pure oxygen gas (60 psi)
3. Hydrochloric acid, concentrated (HCl) – Purchased. Store at room temperature.
4. 1+1 Phosphoric acid (WC_TOC_1:1PA)

| | |
|---|-------|
| Phosphoric acid (H ₃ PO ₄) | 50 mL |
| Reagent water | 50 mL |

Take 50 mL of H₃PO₄ and add slowly while swirling to a 100-mL volumetric flask containing 50 mL reagent water. Store at room temperature. Prepare fresh every 6 months.

5. TOC stock calibration standard (sucrose containing 30% carbon) (WC_TOC_30%SUC), purchased. See label for expiration date. Store at room temperature.
6. TOC LCS standard, purchased (WC_TOC_SLSCC). See label for expiration date, and the certified concentration. Store at room temperature.

Sample Cup Preparation

Sample cups must be conditioned prior to analysis. This is achieved by heating the cups to 750°C for a period of 2 to 5 minutes.

Calibration

1. Working standards for calibration
Pipette a volume of the TOC stock calibration standard into the sample cups as follows:

| Working Std. (mg C) | Vol. Stock TOC (µL) |
|---------------------|---------------------|
| 0.30 | 1.0 |
| 0.90 | 3.0 |
| 3.0 | 10.0 |
| 6.0 | 20.0 |

Prepare fresh daily. Store at room temperature.

2. Continuing calibration verification standard (CCV)

This is a purchased standard, and the acceptable range is specified by the manufacturer.

Procedure

1. Turn on nitrogen to 60 psig.
 2. Turn on oxygen to 60 psig.
 3. Add water and a drop of HCl to the scrubber.
 4. Measure the carrier gas (oxygen) flow rate and adjust to 200 mL/min.
 5. Measure the dryer gas (nitrogen) flow rate and adjust to 160 to 300 mL/min.
 6. Power up instrument.
 7. Program in a sequence file.
 8. Run the calibration sequence as follows: 0.30, 0.90, 3.0, and 6.0 mg C (Performed monthly).
 9. The instrument automatically calculates the R and list the calibration data.
 10. Samples are analyzed along with the appropriate laboratory control standard and preparation blank. A check standard (CCV) and a continuing calibration blank (CCB) must be run at the beginning of each run and after every ten samples.
 11. Add sufficient glass wool to each cup to cover the bottom surface.
 12. Weigh samples into sample cups (up to 1000 mg). Add 1+1 H₃PO₄ drop wise until effervescence stops. Heat at 75°C for 15 minutes. Record the oven ID, time and temperature of this step in the log book.
- NOTE:** This procedure will convert inorganic carbonates and bicarbonates to carbon dioxide and eliminate it from the sample. The addition of H₃PO₄ and the heating to 75°C is not performed for analysis 10065.
13. Analyze the residue according to the instrument manufacturer's instructions for the remaining TOC result.

Calculations

1. To determine mg/kg TOC or TC

$$\text{mg/kg} = (\text{Raw result in mg C}) \times (1000/\text{weight in mg}) \times (1000)$$

2. To determine LOQ/MDL factors

$$\text{Factor} = \frac{\text{Maximum sample aliquot weight (1000.mg)}}{\text{weight of sample (mg)}}$$

3. To determine Total Inorganic Carbon (TIC)

$$\text{TIC} = \text{Total Carbon (TC)} - \text{Total Organic Carbon (TOC)}$$

Statistical Information/Method Performance

1. The method detection limit (MDL) is determined annually by following the procedure outlined in [QA-SOP11892](#).

2. The quality control acceptance windows are generated annually by following the procedure outlined in [QA-SOP11896](#).

Quality Assurance/Quality Control

1. A calibration must be performed every 30 days. The acceptable range for the calibration is $R = 0.995$ or greater. If this criteria is not met, the instrument must be recalibrated.
2. A batch must contain no more than 20 field samples.
3. A batch blank (MB) must be analyzed every batch or each day samples are prepared (not to exceed 20 samples). An acceptable result is $<$ the limit of quantitation. If the PBS does not meet this criterion, it must be rerun twice. If either of the two additional trials do not meet the acceptance criterion, all samples in the batch must be repeated.
4. A laboratory control standard (LCSS) must be analyzed every batch or each day samples are analyzed (not to exceed 20 field samples). For TOC this is a purchased standard and the acceptable range is specified by the manufacturer. The LCSS should undergo the same steps as the samples. If the LCSS does not meet the acceptable criterion, it must be repeated twice. If either of the two additional trials do not meet the acceptance criterion, all samples in the batch must be reanalyzed.
5. Based upon client requirements, a laboratory control standard duplicate (LCSD) may need prepared and analyzed under the same conditions as the LCS. The acceptance criterion for the LCSD is the same as that of the LCS. The relative percent difference between the LCS and the LCSD is calculated statistically.
6. 9060 - a duplicate is analyzed every 10 samples. Lloyd Kahn - one sample is analyzed in quadruplicate every batch. The acceptable relative percent difference is statistically determined. The duplicate relative percent differences must be tracked to continually monitor method performance.
7. 9060, 5310B - A spike must be analyzed for every 10 samples. The sample is spiked with 3 ul of the purchased TOC stock calibration standard. The acceptance range is determined statistically. The spike recoveries must be tracked to continually monitor method performance.
8. Based upon client requirements, a matrix spike duplicate (MSD) may need prepared and analyzed under the same conditions as the MS. The acceptance criterion for the MSD is the same as that of the MS. The relative percent difference between the MS and the MSD is calculated statistically.
9. A check standard (CCV) and blank (CCB) must be run after every ten injections (including blanks and standards). The acceptable range for the CCV determined by the manufacturer. An acceptable CCB result is $<$ the limit of quantitation. If either, or both, of these injections do not meet the acceptance criterion, the unacceptable original must be repeated twice. If either of the two additional trials do not meet the acceptance criterion, all samples since the last compliant CCV/CCB must be reanalyzed.
10. A CCV and CCB shall be analyzed at the beginning and the end of each run. At any time when the instrument has been idle for a period of 4 hours or more, a CCV and CCB must be analyzed. If either of these parameters cannot meet specifications, the instrument must be recalibrated.

[QA-SOP11188 Reagents and Standards](#)

[QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation](#)

[QA-SOP11896 Establishing Control Limits](#)

End of document

Version history

| Version | Approval | Revision information | |
|---------|-------------|----------------------|--|
| 16 | 17.OCT.2018 | | |
| 17 | 03.JUL.2019 | | |
| 17.1 | 22.JUN.2021 | | |

| | | |
|--|--|--|
|  | Always check on-line for validity. pH by EPA 9045C, 9045D and Corrosivity by SW-846 Chap 7 of Solids, Soils, and Solvents using Electrometic Methods | Level:  Work Instruction |
| Document number: T-WC-WI11518 | | Organisation level: 5-Sub-BU |
| Old Reference: 1-P-QM-WI-9011685 | | Responsible: 5_EUUSLA_Water Quality_Manager |
| Version: 14 | | |
| Approved by: X6TJ Effective Date 30-APR-2021 | Document users: 6_EUUSLA_Water Quality_24/48 Hour Analyst, 6_EUUSLA_Water Quality_24/48 Hour Verification | |

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Revision Log

| Revision | 14 | Effective Date: | This version |
|---------------------|--|---|--------------|
| Section | Justification | Changes | |
| Revision Log | Formatting requirement | Removed revision logs up to the previous version | |
| Throughout Document | Current process | Update to new LIMS | |
| Cross Reference | Higher level documents are not required to be referenced | Removed QA-SOP11880 | |
| Procedure 10. | Current process | Added step 10 - to read the 2 and 12 standards when analyzing samples for corrosivity | |
| Procedure | Current Process | Removed previous step 11. Units are reported as S.U. | |

| Revision | 14 | Effective Date: | This version |
|---------------------------------------|-----------------|-----------------|--|
| Quality Assurance/ Quality Control | Clarification | | Added acceptance range for CCVs. |
| | Current process | | Removed requirement to analyze the 2 and 12 standards <u>before</u> any samples Removed step to read pH 3 times for corrosivity |

| Revision | 13 | Effective Date: | 08-JUL-2019 |
|--------------|------------------------|---|-------------|
| Section | Justification | Changes | |
| Revision Log | Formatting requirement | Removed revision logs up to the previous version | |
| Procedure 11 | Clarification | Added how solid pH is reported (soil pH measured in water at recorded temp at time of analysis) | |

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 9045C, January 1995.
2. Test Methods for Evaluating Solid Wastes, SW-846 Method 9045D, November 2004.
3. Test Methods for Evaluating Solid Wastes, SW-846 Chapter 7.
4. *Chemical Hygiene Plan*, current version.

Cross Reference

| Document | Document Title |
|------------------------------|--|
| T-WC-WI11519 | pH Probes and Meters |
| T-WC-WI10360 | Quality Control Data for Wet Chemistry |

Scope

This SOP provides the guidelines for analysts performing pH on solid, soil, and solvent samples. This procedure is applicable to solid/soil/solvent samples.

The sensitivity limit for this technique is 0.01 pH units.

Basic Principles

A 1:1 slurry is prepared and the activity of hydrogen ions in the supernatant is measured using a combination pH electrode.

Determination of corrosivity is based on the pH value of the sample.

Reference Modifications

Method 9045C/D has been modified for the analysis of solid, soil, and solvent samples in the following ways:

1. An Automatic Temperature Compensator is used for all samples instead of manually performing calculations to correct measured pH values if the sample and buffer solution temperatures differ by more than 2°C.
2. 25 g of soil to 25 g reagent water is used instead of the 20 g: 20 mL ratio.
3. The samples are tumbled for approximately 30 minutes instead of being stirred with a stir bar for 5 minutes.

These modifications are performed in order to allow for adequate agitation and to provide sufficient supernatant to immerse the pH electrode during analysis.

Interferences

Interferences occur when oily or particulate matter adheres to the electrodes and reduces the response. Gentle wiping or rinsing with reagent water usually corrects this problem. Temperature effects are compensated for by calibrating the pH meter at the temperature of the sample or using a pH meter equipped with temperature compensators. There are no means of controlling temperature effects caused by shifts in ionic equilibria of the sample.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention. Standard safe laboratory procedure must be followed as outlined in the Chemical Hygiene Plan.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four pH readings of the 7.00 pH buffer (which is used as the laboratory control standard) that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Sample must be collected in an unpreserved container and stored at 0° to 6°C; not frozen, until the time of analysis. There is no published holding time for pH analysis on soil; analysis is performed as soon as possible after sample is received in the laboratory.

Apparatus and Equipment

1. Analytical balance capable of weighing 0.0001 g
2. pH meter equipped with an ATC probe (Automatic Temperature Compensator)
3. Combination electrode or equivalent
4. Stir bar and stir plate

Reagents and Standards

1. 7.00 pH Buffer (ISO 17025 approved vendor) – purchased; see container for shelf life information.
2. Appropriate pH electrode filling solution for electrode, purchased. Store at room temperature. See label for expiration date.

Calibration

Balances must be calibrated each day before use.

Calibrate pH meter as described in [T-WC-WI11519](#).

Procedure

1. Make sure the pH meter has been calibrated within the last 24 hours.
2. Weigh 25 ± 0.5 g of sample into a clean specimen cup and add 25 ± 0.5 mL of reagent water (makes a 1:1 slurry).

If the sample absorbs the water, add an additional 25 ± 0.5 mL of reagent water (makes a 1:2 slurry). If a 1:2 slurry does not provide sufficient supernatant to immerse the pH electrode, use less sample and add reagent water in proportion to the weight selected. Enter a comment in LIMS indicating the dilution.

3. Tightly place the screw-cap lid on the sample and mix the slurry in the tumbler for approximately 30 minutes.
4. Remove the sample from the tumbler and allow the sample to settle for about 1 hour.
5. Rinse and shake off any water on the electrodes.
6. Dip the electrodes into the supernatant (aqueous layer) of the sample and allow to equilibrate. If necessary, decant or pipette this layer into another container.
7. Using the calibrated, pH meter note the pH value of the sample after the meter equilibrates, and enter the value in the LIMS.

NOTE: If pH reading is < 4.00 or > 10.00 , then the pH result will be reported with a qualifying flag.

8. Note the temperature of the sample and record the value in LIMS.
9. Rinse and clean the electrodes before proceeding to the next sample.
10. If corrosivity is to be reported, read the pH 2 and 12 standards and record the pH value.
11. Corrosivity is determined from the sample's pH reading. A "Yes" or "No" response is entered for the corrosivity result, depending on the pH value. A sample is considered corrosive if the pH is < 2 or > 12 , warranting a "Yes" result. A sample with a pH value from 2 to 12 has a corrosivity result of "No".

Calculations

Not applicable.

Statistical Information/Method Performance

Not applicable to this procedure

Quality Assurance/Quality Control

One batch consists of no more than 20 samples.

A Laboratory Control Standard (LCS; 7.00 pH Solution) must be analyzed at the beginning of each batch.

A CCV (7.00 pH Solution) must be analyzed after every ten samples and at the end of the batch. The acceptance range for the CCV is 90 - 110%.

Two matrix duplicates must be analyzed per batch of 20 samples. If 10 or less samples are on a batch then only one matrix duplicate is needed.

When analyzing for Corrosivity, a pH check using buffers 2 and 12 must be analyzed.

If the meter must be re-calibrated during the analysis, a LCS must be analyzed after the calibration is performed.

See LIMS for current quality control acceptance windows.

Refer to [T-WC-WI10360](#) if any of the QC samples do not meet required specifications.

[QA-SOP11880 Laboratory Equipment Verifications - Balance, Syringe, Pipette, Weights, and Other Equipment](#)
[T-WC-WI10360 Quality Control Data for Wet Chemistry](#)
[T-WC-WI11519 pH Probes and Meters](#)

End of document

Version history

| Version | Approval | Revision information | |
|---------|-------------|--|--|
| 12 | 27.JUL.2018 | | |
| 13 | 27.JUN.2019 | | |
| 14 | 22.APR.2021 | Annual review completed on 4/28/2022, no changes needed. | |

| | | |
|--|--|--|
|  Document number: T-WC-WI11514 Old Reference: 1-P-QM-WI-9014165 Version: 11 | Always check on-line for validity. <p style="text-align: center;">Particle Size Distribution of Soils and Solids/Grain Size Classification by ASTM D422-63 (reapproved 2007)</p> | Level:  Work Instruction |
| Approved by: X6TJ Effective Date 31-JAN-2021 | Document users: 6_EUUSLA_Water Quality_Misc Analysis, 6_EUUSLA_Water Quality_Misc Verification | Organisation level: 5-Sub-BU Responsible: 5_EUUSLA_Water Quality_Manager |

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Revision Log

| Revision | 11 | Effective Date: | This version |
|---------------------------------------|--|---|--------------|
| Section | Justification | Changes | |
| Revision Log | Formatting requirement | Removed revision logs up to the previous version | |
| Throughout Document | Reflect current active method | Updated to current LIMS IDs | |
| Cross Reference | Reflect current process | Removed form T_WC_FRM11472 (NLIU) | |
| Cross Reference | Higher level documents are not required to be referenced | Removed QA-SOP11880 and QA-SOP11188 | |
| Personnel Training and Qualifications | Required wording | Updated to include required wording. | |
| | | | |

| Revision 10 | | Effective Date: 24-Jan 2019 |
|-------------------------------------|-------------------------------------|--|
| Section | Justification | Changes |
| Revision Log | Formatting requirement | Removed revision logs up to the previous version |
| Reference | Reflect current active method | Added ASTM D422 |
| Cross Reference/throughout document | Reflect current designations in ETQ | Updated to ETQ designation |
| Reagents and Standards | No longer used | Removed item #2 n-hexane |
| Procedure B:NOTE; | New requirement | Added that hydrometer will be calibrated yearly by certified vendor. |
| Procedure F | No longer used | Removed oil extraction section F from SOP |
| Entire document | Reflect current forms being used | Added form T-WC-FRM11472 (form to use when only analysis 7103) |

Reference

1. ASTM D422-63 (reapproved 2007), Standard Test Method for Particle-Size Analysis of Soil
2. ASTM D422, Standard Test Method for Particle-Size Analysis of Soil
3. ASTM D421 - 85, *Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants.*
4. ASTM E868 - 82, "Conducting Performance Tests on Mechanical Conveying Equipment Used in Resource Recovery Systems," Sec 9.9, "Measuring Bulk Density of Material."
5. Method 160.3, *EPA Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020.
6. *Chemical Hygiene Plan*, current version.

Cross Reference

| Document | Document Title |
|-------------------------------|---|
| S-SS-WI10697 | Moisture (Gravimetric) |
| T-WC-FRM11473 | D422 Particle Size Distribution / Grain Size Classification |
| Q-EQA-WI6815 | ETM System Probe Calibration |
| T-WC-WI9901 | Equipment Muffle Furnaces and Ovens |

Scope

This procedure is applicable to the determination of the distribution of particle sizes in soils and solids. Particle sizes in the range 75 to 0.075 mm are determined by sieving. Distribution of particle sizes smaller than 0.075 mm is determined by a

sedimentation process using a hydrometer.

Basic Principles

The process determines the particle sizes and distribution in a soil/solid sample. The sample is dried, ground, and sieved using sieves of different sizes. Particle sizes in the range 75 to 0.075 mm are determined by sieving. Distribution of particle sizes smaller than 0.075 mm is determined by a sedimentation process using a hydrometer.

Interferences

When high levels of organic material are present in the soil (material with a specific gravity less than one), the hydrometer readings are subject to error. After air-drying the sample, remove as much of the organic material (leaves, roots, sticks, etc.) as possible.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Analysts are considered proficient when they have successfully demonstrated competency under supervision of a supervisor or other trained analyst.

Sample Collection, Preservation, and Handling

There is no holding time for this analysis. Samples are stored at 0 to 6°C; not frozen.

1. Place the sample as received in a glass baking dish. Allow to air dry at room temperature for approximately 24 hours or until thoroughly dried. Use enough sample so that the material needed to pass through the No. 8 sieve is approximately 100 g or more.
2. Break and grind the dried sample using the rubber-covered pestle. Continue to do so until the sample is reduced to a fine material. **Do not break rocks that are part of the sample matrix as this is likely to affect the sample sedimentation process.**

All information needs to be recorded on Form [T-WC-FRM11473](#) as soon as the sample preparation is started.

Apparatus and Equipment

1. Analytical balance, pan balance, or equivalent
2. Mechanically operated sieving device
3. Mechanically operated stirring device
4. Hydrometer - graduated to read in specific gravity of the suspension
5. Sedimentation cylinder – 1000-mL
6. Thermometer - accurate to 0.5°C
7. Sieves - Numbers 4, 6, 8, 16, 30, 50, 100, 200, 0.75", 1.5", and 3"
8. Sieve pan
9. Rubber covered pestle, or equivalent
10. Beakers – 150-mL and 250-mL
11. Specimen collection cups (120-mL), or equivalent
12. Glass baking dishes, or equivalent

13. Glass stir rod, or equivalent
14. Oven maintained at $110^{\circ} \pm 5^{\circ}\text{C}$; adjust as needed to stay in this range
15. Whatman Grade 3 filter paper - 6 micron pore size
16. Buchner funnel

Reagents and Standards

Alternate weights may be used as long as the final concentrations remain the same.

1. Sodium hexametaphosphate solution
 - a. Using a 1000-mL volumetric flask, dissolve 40 g of sodium hexametaphosphate in reagent water.
 - b. Dilute to final volume once dissolved
 - c. Store at room temperature in a glass or plastic container
 - d. Solution expires one month from date of preparation.

Calibration

Balance calibration must be checked each day before use.

Procedure

A. The sieved analysis of the portion retained on the No. 8 sieve.

Check to be sure that the balance has been calibrated each day before use.

1. Tare the sieving pan, No. 8, No. 6, No. 4, 0.75", 1.5", and the 3" sieved consecutively (to 0.01 g) using a pan balance.
2. Using the prepared sample, place the sample in the tared sieves and begin the sieving operation using the mechanical sieving device. Agitate for approximately one minute. Continue sieving until no more than 1% of the residue on each sieve

passes that sieve during 1 minute of sieving.

NOTE: If sample particle size is too great to pass through the sieve, continue to break and grind as needed. Do not break stones or rocks that are part of the sample matrix.

3. Determine the mass retained (M_r) on the 3" sieve. Weigh on the balance and record the combined sieve and sample weight. Continue sieving with the remaining sieves recording all determined sample masses on the sieves.

To calculate the mass retained:

$$M_r = \text{Tare} + \text{sample weight value} - \text{the tare weight value}$$

4. Retain the sample collected in the sieving pan after weighing.

a. Weigh approximately 50 g of sample into a 250-mL beaker.

b. Place approximately 30 g or more of the remaining sample into a specimen cup, for use when performing the moisture and bulk density analyses.

B. Hydrometer analysis

NOTE: The hydrometer used in the process will be calibrated on an annually basis by a certified vendor.

The hydrometer must be visually inspected for any cracks or breaks before each use.

1. Using the sample in the 250-mL beaker add 125 mL of sodium hexametaphosphate solution. Stir the soil-water slurry until it is thoroughly moistened. Cover and label beaker with sample number. Allow this slurry to soak for at least 16 hours, but not to exceed 24 hours.

2. At the end of the soaking period, transfer the soil-water slurry into a dispersion cup using room temperature reagent water. Be sure to rinse all residue from the beaker into the dispersion cup.

NOTE: Extra care needs to be taken when rinsing the sample into another container or when using any apparatus. **All particles need to be rinsed into the new container or a loss in sample weight is possible to occur.**

3. Stir for one minute using the stirring apparatus. **Rinse stirring apparatus well into dispersion cup to prevent loss of sample.**

4. Transfer the soil-water slurry into a glass sedimentation cylinder, rinsing well. Add room temperature reagent water until the total volume is 1000 mL.

5. Cover the open end of the cylinder with parafilm, then placing the palm of the hand over the end, invert the cylinder and agitate for a period of one minute. Place the cylinder in a location where it will not be disturbed or moved during the sedimentation readings (for 24 hours).

6. Remove the parafilm and immediately begin the hydrometer readings, recording the temperature reading after each suspension. Record the readings at the following intervals (measured from the beginning of sedimentation): 2, 5, 15, 30, 60, 250, and 1440 minutes. Insert the hydrometer about 20 to 25 seconds before the reading is due to the approximate depth. Clean the hydrometer between readings by placing it into reagent water and twisting with a spinning motion.

NOTE: The basic temperature to be maintained for the hydrometer test is 68°F (20°C). Small variations of temperature do not introduce differences that are of practical significance.

C. Final sieving analysis of the portion of sample passing the No. 8 sieve.

1. After taking the final hydrometer reading, pour the suspension through a No. 200 sieve and rinse with reagent water until the wash water is clear.

2. Transfer the remaining sample from the sieve into a 150-mL beaker, rinsing well with room temperature reagent water and dry in an oven at $110^{\circ} \pm 5^{\circ}\text{C}$ for at the least 12 hours or longer.

3. After the sample has dried and no liquid is remaining. Remove from the oven and allow to cool. When the sample has reached room temperature use a glass stir rod to scrape the beaker and crush the harden residue into fine particles.

4. Tare the collection pan, the No. 200, No. 100, No. 50, No. 30, and No. 6 sieves consecutively (to 0.01 g) using a pan balance.

5. Place the oven-dried sample into the sieves and cover. The sieves need to be covered at this point of agitation due to the fine and lightweight particle sizes. Agitate for approximately 5 minutes to allow the fine particles to pass within the sieves.

6. Determine the mass retained (M_r) on each sieve by weighing and recording the combined weight of the sieve and the sample.

D. Moisture analysis

Using approximately 5 g of sample from the specimen collection cup (Procedure: Section A.4.b.) Perform a moisture analysis according to [S-SS-WI10697](#). Record all information on form [T-WC-FRM11473](#).

E. Bulk density analysis

1. Weigh out approximately 15 g of sample from the specimen collection cup (Procedure A.4.b.).

2. Tare a 100 mL volumetric flask and record data on form *T-WC-FRM11473*.
3. Add sample to tared flask.
4. Add approximately 50 mL reagent water and agitate flask until soil is thoroughly mixed.
5. Remove air bubbles by inserting a vacuum tube inside the neck of flask being careful not touch the contents of the flask.
6. Once air bubbles are removed, bring mixture to a final volume of 100 mL using reagent water.
7. Reweigh the flask and its contents and record the reading on the data sheet (form *T-WC-FRM11473*)

F. Grain Size Classification

To report the % Gravel, % Sand, % Silt, and % Clay fractions electronically, a separate grain size classification scan with the % passing at each type as a separate piece will be determined and recorded on form *T-WC-FRM11473*. See below for further information on % classification. For sediments that do not have sufficient sample volume to perform the hydrometer portion of the test, the sum of the % Silt and Clay can be reported as one aggregate value.

1. % Gravel – determined by the amount of particles passing 3-in. and retained on No. 4 sieve (size 4.75-mm).
2. % Sand - determined by the amount of particles passing No. 4 sieve (size 4.75-mm) and retained on No. 200 sieve (size 0.075-mm).
3. % Silt – determined by the amount of particles passing 0.074 to 0.005-mm.
4. % Clay – determined by the amount of particles smaller than 0.005-mm.

Calculations

A spreadsheet using Excel, or an equivalent, has been prepared to facilitate the calculation of data, the plotting of the graph, and the interpolation of the hydrometer results from the graph. It is possible for data to be reported directly from the spreadsheets; however, the plotted hydrometer data may be more accurate than the computer interpolations. Refer to this spreadsheet for the appropriate tables to hand calculate data.

Use the mass retained (Mr) values recorder for each sieve as Mr8, Mr6, Mr4, etc. to determine the percent passing each sieve.

To calculate the mass retained:

$M_r = \text{Tare} + \text{sample weight value} - \text{the tare weight value}$

A. Sieve analysis values for the portion coarser than the No. 8 sieve

1. $M_t = M_c + M_f$

Where:

$M_t =$ total mass

$M_c =$ mass of the coarse material

$M_f =$ mass of the fine material

2. $P_8 = M_f/M_t \times 100$

Where:

$P_8 =$ percentage passing No. 8 sieve

3.

$$P_6 = \frac{M_f + M_{r8}}{M_t} \times 100$$

Where:

$P_6 =$ percentage passing No. 6 sieve

$M_{r8} =$ mass retained on No. 8 sieve

4.

$$P_4 = \frac{M_f + M_{r8} + M_{r6}}{M_t} \times 100$$

Where:

$M_{r6} =$ mass retained on the No. 6 sieve

$P_4 =$ percentage passing No. 4 sieve

5. Continue calculations as above for the 0.75", 1.5", and 3" sieves.

B. Percentages of soil in suspension

1. $H_f = O_s/A_s$

Hygroscopic moisture correction factor

Where:

H_f = hygroscopic moisture correction factor

A_s = air dry mass of the soil

O_s = oven dry mass of the soil = (oven dry weight minus the tare weight)

2. Percentages of soil in suspension

a. Calculate the oven dry mass of soil used in the hydrometer analysis by multiplying the air-dry mass of the soil used in the hydrometer analysis by the moisture correction factor.

b.

$$W = \frac{\text{oven dry mass in hydrometer}}{P8} \times 100$$

Where:

W = oven dry mass of soil in a total test sample represented by the mass of the soil dispersed, in grams

$P8$ = percentage passing the No. 8 sieve (from calculation A.2.)

c. $P = [(100,000/W) \times G/(G-G_1)] \times (R-G_1)$

Where:

G = specific gravity of the soil

G_1 = specific gravity of the liquid in which the soil was suspended (use 1)

W = oven dry mass of soil in a total test sample represented by the mass of the soil dispersed, in grams

P = percentage of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension

R = Hydrometer reading with composite correction applied

d.

$$D = K \sqrt{L/T}$$

Where:

D = diameter of the particle (in mm)

K = constant using the temperature of the suspension and the specific gravity of the soil particles (see the Excel spreadsheet or equivalent for the Analysis #7103, for the specific "K" values to use)

L = distance from the surface of the suspension to the level at which the density of the suspension is being measured (the effective depth from Table I)

T = interval of time from the beginning of sedimentation to the taking of the reading in minutes

C. Sieve analysis values of the portion finer than the No. 8 sieve

1. Calculate the percentage of particles retained on the No. 8 sieve as follows:

$$Pr8 = 100 - P8$$

Where:

Pr8 = percentage of particles retained on the No. 8 sieve

P8 = percentage of particles passing the No.8 sieve (from calculation A.2.)

2. Calculate the mass retained on the No. 8 sieve represented by the sample volume used in the hydrometer analysis as follows:

$$Mf8 = \frac{Pr8 \times W}{100}$$

Where:

Mf8 = mass retained on the No. 8 sieve represented by the sample volume used in the hydrometer analysis

W = oven dry mass of the soil in a total test sample represented by the mass of the soil dispersed (as calculated in B.2.b.)

3. Calculate the mass passing the No. 200 sieve as follows:

$$M_{p200} = W - (M_{r200} + M_{r100} + M_{r50} + M_{r30} + M_{r16} + M_{f8})$$

Where:

Mp200 = mass passing the No. 200 sieve

Mr200, 100, 50, etc. = mass retained on the sieves used for the portion finer than the No. 8 sieve

Mf8 = mass retained on the No. 8 sieve represented by the mass of the soil used in the hydrometer analysis

W = oven dry mass of the soil in a total test sample represented by the mass of the soil dispersed (as calculated in B.2.b.)

4.

$$P_{200} = \frac{M_{p200}}{W} \times 100$$

Where:

P200 = percentage passing the No. 200 sieve

Mp200 = mass passing the No. 200 sieve (use calculation from C.3.)

W = oven dry mass of the soil in a total test sample represented by the mass of the soil dispersed (as calculated in B.2.b.)

5.

$$P_{100} = \frac{M_{p200} + M_{r200}}{W} \times 100$$

Where:

P100 = percentage passing the No. 100 sieve

Mr200 = mass retained on the No. 200 sieve

Mp200 = mass passing the No. 200 sieve (use calculation from C.3.)

W = oven dry mass of the soil in a total test sample represented by the mass of the soil dispersed (as calculated in B.2.b.)

6.

$$P50 = \frac{Mp200 + Mr200 + Mr100}{W} \times 100$$

Where:

P50 = percentage passing the No. 50 sieve

Mr100 = mass retained on the No. 100 sieve

Mp200 = mass passing the No. 200 sieve

Mr200 = mass retained on the No. 200 sieve

W = oven dry mass of the soil in a total test sample represented by the mass of the soil dispersed (as calculated in B.2.b.)

7. Continue calculations as above for the No. 30 and No. 16 sieves.

D. Prepare a graph of the test results on a logarithmic scale, plotting the diameter of the particles as the abscissa and the percentages smaller than the corresponding diameters as the ordinate. Report (from the graph or directly from the tabulated data) the percentage of particles passing the 3", 1.5", 0.75", No. 4, No. 6, No. 8, No. 16, No. 30, No. 50, No. 100, and No. 200 sieves. Report the hydrometer results (from the graph) as the percentage passing 0.050 mm, 0.020 mm, 0.005 mm, 0.002 mm, and 0.001 mm.

Statistical Information/Method Performance

Not applicable to this procedure.

Quality Assurance/Quality Control

When possible, perform duplicate analyses for particle size on a routine basis.

Batch size is limited to 20 samples or less.

Table I

**Values of Effective Depth Based on Hydrometer
and Sedimentation Cylinder of Specific Sizes^A**

| Hydrometer 151H | |
|--|---------------------------------------|
| Actual Hydrometer Reading | Effective Depth, L, cm |
| 1.000 | 16.3 |
| 1.001 | 16.0 |
| 1.002 | 15.8 |
| 1.003 | 15.5 |
| 1.004 | 15.2 |
| 1.005 | 15.0 |
| 1.006 | 14.7 |
| 1.007 | 14.4 |
| 1.008 | 14.2 |
| 1.009 | 13.9 |
| 1.010 | 13.7 |
| 1.011 | 13.4 |
| 1.012 | 13.1 |
| 1.013 | 12.9 |
| 1.014 | 12.6 |
| 1.015 | 12.3 |
| 1.016 | 12.1 |
| 1.017 | 11.8 |
| 1.018 | 11.5 |
| 1.019 | 11.3 |
| 1.020 | 11.0 |
| 1.021 | 10.7 |
| 1.022 | 10.5 |
| 1.023 | 10.2 |
| 1.024 | 10.0 |
| 1.025 | 9.7 |

| Hydrometer 151H | |
|---------------------------------|------------------------------|
| Actual Hydrometer Reading | Effective Depth, L, cm |
| 1.026 | 9.4 |
| 1.027 | 9.2 |
| 1.028 | 8.9 |
| 1.029 | 8.6 |
| 1.030 | 8.4 |
| 1.031 | 8.1 |
| 1.032 | 7.8 |
| 1.033 | 7.6 |
| 1.034 | 7.3 |
| 1.035 | 7.0 |
| 1.036 | 6.8 |
| 1.037 | 6.5 |
| 1.038 | 6.2 |

Values of effective depth are calculated from the equation:

$$L = L1 + \frac{1}{2} \times \left[L2 - \left(\frac{V_B}{A} \right) \right]$$

Where:

L = effective depth, cm

L1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm

L₂ = overall length of the hydrometer bulb, cm

V_B = volume of hydrometer bulb, cm³, and

A = cross-sectional area of sedimentation cylinder, cm²

Values used in calculating the values in Table I are as follows:

For hydrometer 151H:

L₂ = 14.0 cm

V_b = 67.0 cm³

A = 27.8 cm²

L1 = 10.5 cm for a reading of 1.000

= 2.3 cm for a reading of 1.031

Q-EQA-WI6815 ETM System Probe Calibration

QA-SOP11188 Reagents and Standards

QA-SOP11880 Laboratory Equipment Verifications - Balance, Syringe, Pipette, Weights, and Other Equipment

S-SS-WI10697 % Moisture Calculation and % Solids Calculation (Gravimetric)

T-WC-FRM11472 #7103 Particle Size Distribution

T-WC-FRM11473 Particle Size Distribution/ Grain Size Classification

T-WC-WI9901 Equipment Muffle Furnaces and Ovens

End of document

Version history

| Version | Approval | Revision information | |
|---------|-------------|----------------------|--|
| 9 | 18.AUG.2014 | | |
| 10 | 17.JAN.2019 | | |
| 11 | 29.JAN.2021 | | |

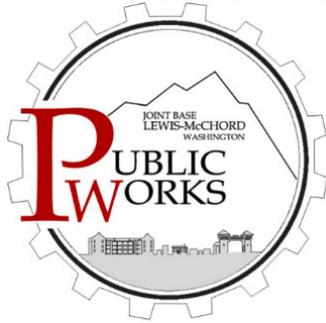
Appendix H
Accident Prevention Plan
(under separate cover)

Appendix I
Project Schedule

| ID | Task Name | Duration | Actual Start | Actual Finish | Start | Finish | % Complete | 2024 | | | | | | | | | | | | | | | | |
|----|--|------------------|---------------------|---------------|---------------------|--------------------|------------|-------------------------------------|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|----|
| | | | | | | | | Q3 | Q4 | Q1 | Q2 | Q3 | Q4 | Q1 | Q2 | Q3 | Q4 | Q1 | Q2 | Q3 | Q4 | Q1 | Q2 | Q3 |
| 1 | Environmental Remediation Service (ERS) at Yakima Training Center, WA | 1842 days | Wed 9/14/22 | NA | Wed 9/14/22 | Wed 9/29/27 | 7% | [Gantt bar from Q3 2022 to Q3 2024] | | | | | | | | | | | | | | | | |
| 2 | Contract | 1828 days | Wed 9/28/22 | NA | Wed 9/28/22 | Wed 9/29/27 | 16% | [Gantt bar from Q3 2022 to Q3 2024] | | | | | | | | | | | | | | | | |
| 3 | Task Order Award & Notice to Proceed | 1 day | Wed 9/28/22 | Wed 9/28/22 | Wed 9/28/22 | Wed 9/28/22 | 100% | [Vertical tick at start of Q3 2022] | | | | | | | | | | | | | | | | |
| 4 | Post Award Kickoff Meeting with Army | 1 day | Fri 10/14/22 | Fri 10/14/22 | Fri 10/14/22 | Fri 10/14/22 | 100% | [Vertical tick at start of Q4 2022] | | | | | | | | | | | | | | | | |
| 5 | YTC Site Visit and Orientation Meeting | 2 days | Wed 11/16/22 | Thu 11/17/22 | Wed 11/16/22 | Thu 11/17/22 | 100% | [Vertical tick at start of Q1 2023] | | | | | | | | | | | | | | | | |
| 6 | Monthly Progress Meetings | 1827 days | Thu 9/29/22 | NA | Thu 9/29/22 | Wed 9/29/27 | 16% | [Red bar from Q3 2022 to Q3 2024] | | | | | | | | | | | | | | | | |
| 7 | Quarterly Contract Line Item Review Meetings | 1827 days | Thu 9/29/22 | NA | Thu 9/29/22 | Wed 9/29/27 | 16% | [Red bar from Q3 2022 to Q3 2024] | | | | | | | | | | | | | | | | |
| 8 | Project Management and Support | 1827 days | Thu 9/29/22 | NA | Thu 9/29/22 | Wed 9/29/27 | 16% | [Red bar from Q3 2022 to Q3 2024] | | | | | | | | | | | | | | | | |
| 9 | General Requirements | 1828 days | Wed 9/28/22 | NA | Wed 9/28/22 | Wed 9/29/27 | 51% | [Gantt bar from Q3 2022 to Q3 2024] | | | | | | | | | | | | | | | | |
| 10 | Project Training to Include AT Level 1 Training, iWatch Training, Level I training, OPSEC Training | 22 days | Wed 9/28/22 | Fri 10/28/22 | Wed 9/28/22 | Fri 10/28/22 | 100% | [Vertical tick at start of Q3 2022] | | | | | | | | | | | | | | | | |
| 11 | Monthly Progress Review Meetings and Minutes Base Yr | 210 days | Fri 10/14/22 | NA | Fri 10/14/22 | Tue 8/15/23 | 80% | [Gantt bar from Q3 2022 to Q3 2024] | | | | | | | | | | | | | | | | |
| 12 | Monthly Progress Review Meetings and Minutes Yr 1 - 1 | 1 day | Fri 10/14/22 | Fri 10/14/22 | Fri 10/14/22 | Fri 10/14/22 | 100% | [Vertical tick at start of Q3 2022] | | | | | | | | | | | | | | | | |
| 13 | Monthly Progress Review Meetings and Minutes Yr 1 - 2 | 0 days | Fri 10/14/22 | Fri 10/14/22 | Fri 10/14/22 | Fri 10/14/22 | 100% | [Vertical tick at start of Q3 2022] | | | | | | | | | | | | | | | | |
| 14 | Monthly Progress Review Meetings and Minutes Yr 1 - 3 | 1 day | Tue 11/15/22 | Tue 11/15/22 | Tue 11/15/22 | Tue 11/15/22 | 100% | [Vertical tick at start of Q4 2022] | | | | | | | | | | | | | | | | |
| 15 | Monthly Progress Review Meetings and Minutes Yr 1 - 4 | 1 day | Tue 12/20/22 | Tue 12/20/22 | Tue 12/20/22 | Tue 12/20/22 | 100% | [Vertical tick at start of Q1 2023] | | | | | | | | | | | | | | | | |
| 16 | Monthly Progress Review Meetings and Minutes Yr 1 - 5 | 0 days | Tue 1/24/23 | Tue 1/24/23 | Tue 1/24/23 | Tue 1/24/23 | 100% | [Vertical tick at start of Q2 2023] | | | | | | | | | | | | | | | | |
| 17 | Monthly Progress Review Meetings and Minutes Yr 1 - 6 | 1 day | Fri 2/17/23 | Fri 2/17/23 | Fri 2/17/23 | Fri 2/17/23 | 100% | [Vertical tick at start of Q3 2023] | | | | | | | | | | | | | | | | |
| 18 | Monthly Progress Review Meetings and Minutes Yr 1 - 7 | 1 day | Mon 3/20/23 | Mon 3/20/23 | Mon 3/20/23 | Mon 3/20/23 | 100% | [Vertical tick at start of Q4 2023] | | | | | | | | | | | | | | | | |
| 19 | Monthly Progress Review Meetings and Minutes Yr 1 - 8 | 1 day | Wed 4/19/23 | Wed 4/19/23 | Wed 4/19/23 | Wed 4/19/23 | 100% | [Vertical tick at start of Q1 2024] | | | | | | | | | | | | | | | | |
| 20 | Monthly Progress Review Meetings and Minutes Yr 1 - 9 | 1 day | Tue 5/16/23 | Tue 5/16/23 | Tue 5/16/23 | Tue 5/16/23 | 100% | [Vertical tick at start of Q2 2024] | | | | | | | | | | | | | | | | |
| 21 | Monthly Progress Review Meetings and Minutes Yr 1 - 1 | 1 day | Tue 6/20/23 | Tue 6/20/23 | Tue 6/20/23 | Tue 6/20/23 | 100% | [Vertical tick at start of Q3 2024] | | | | | | | | | | | | | | | | |
| 22 | Monthly Progress Review Meetings and Minutes Yr 1 - 1 | 1 day | NA | NA | Tue 7/18/23 | Tue 7/18/23 | 0% | [Vertical tick at start of Q4 2023] | | | | | | | | | | | | | | | | |
| 23 | Monthly Progress Review Meetings and Minutes Yr 1 - 1 | 1 day | NA | NA | Tue 8/15/23 | Tue 8/15/23 | 0% | [Vertical tick at start of Q1 2024] | | | | | | | | | | | | | | | | |
| 24 | Monthly Progress Review Meetings and Minutes Yr 2 | 232 days | NA | NA | Thu 9/14/23 | Thu 8/8/24 | 0% | [Gantt bar from Q3 2023 to Q3 2025] | | | | | | | | | | | | | | | | |
| 25 | Monthly Progress Review Meetings and Minutes Yr 2 - 1 | 1 day | NA | NA | Thu 9/14/23 | Thu 9/14/23 | 0% | [Vertical tick at start of Q4 2023] | | | | | | | | | | | | | | | | |
| 26 | Monthly Progress Review Meetings and Minutes Yr 2 - 2 | 1 day | NA | NA | Mon 10/16/23 | Mon 10/16/23 | 0% | [Vertical tick at start of Q1 2024] | | | | | | | | | | | | | | | | |
| 27 | Monthly Progress Review Meetings and Minutes Yr 2 - 3 | 1 day | NA | NA | Wed 11/15/23 | Wed 11/15/23 | 0% | [Vertical tick at start of Q2 2024] | | | | | | | | | | | | | | | | |
| 28 | Monthly Progress Review Meetings and Minutes Yr 2 - 4 | 1 day | NA | NA | Fri 12/15/23 | Fri 12/15/23 | 0% | [Vertical tick at start of Q3 2024] | | | | | | | | | | | | | | | | |
| 29 | Monthly Progress Review Meetings and Minutes Yr 2 - 5 | 1 day | NA | NA | Tue 1/16/24 | Tue 1/16/24 | 0% | [Vertical tick at start of Q4 2024] | | | | | | | | | | | | | | | | |
| 30 | Monthly Progress Review Meetings and Minutes Yr 2 - 6 | 1 day | NA | NA | Wed 2/14/24 | Wed 2/14/24 | 0% | [Vertical tick at start of Q1 2025] | | | | | | | | | | | | | | | | |
| 31 | Monthly Progress Review Meetings and Minutes Yr 2 - 7 | 1 day | NA | NA | Thu 3/14/24 | Thu 3/14/24 | 0% | [Vertical tick at start of Q2 2025] | | | | | | | | | | | | | | | | |
| 32 | Monthly Progress Review Meetings and Minutes Yr 2 - 8 | 1 day | NA | NA | Fri 4/12/24 | Fri 4/12/24 | 0% | [Vertical tick at start of Q3 2025] | | | | | | | | | | | | | | | | |
| 33 | Monthly Progress Review Meetings and Minutes Yr 2 - 9 | 1 day | NA | NA | Mon 5/13/24 | Mon 5/13/24 | 0% | [Vertical tick at start of Q4 2025] | | | | | | | | | | | | | | | | |
| 34 | Monthly Progress Review Meetings and Minutes Yr 2 - 1 | 1 day | NA | NA | Tue 6/11/24 | Tue 6/11/24 | 0% | [Vertical tick at start of Q1 2026] | | | | | | | | | | | | | | | | |
| 35 | Monthly Progress Review Meetings and Minutes Yr 2 - 1 | 1 day | NA | NA | Wed 7/10/24 | Wed 7/10/24 | 0% | [Vertical tick at start of Q2 2026] | | | | | | | | | | | | | | | | |
| 36 | Monthly Progress Review Meetings and Minutes Yr 2 - 1 | 1 day | NA | NA | Thu 8/8/24 | Thu 8/8/24 | 0% | [Vertical tick at start of Q3 2026] | | | | | | | | | | | | | | | | |
| 37 | Monthly Progress Review Meetings and Minutes Yr 3 | 232 days | NA | NA | Fri 9/6/24 | Mon 7/28/25 | 0% | [Gantt bar from Q3 2024 to Q3 2026] | | | | | | | | | | | | | | | | |
| 38 | Monthly Progress Review Meetings and Minutes Yr 3 - 1 | 1 day | NA | NA | Fri 9/6/24 | Fri 9/6/24 | 0% | [Vertical tick at start of Q4 2024] | | | | | | | | | | | | | | | | |
| 39 | Monthly Progress Review Meetings and Minutes Yr 3 - 2 | 1 day | NA | NA | Mon 10/7/24 | Mon 10/7/24 | 0% | [Vertical tick at start of Q1 2025] | | | | | | | | | | | | | | | | |
| 40 | Monthly Progress Review Meetings and Minutes Yr 3 - 3 | 1 day | NA | NA | Tue 11/5/24 | Tue 11/5/24 | 0% | [Vertical tick at start of Q2 2025] | | | | | | | | | | | | | | | | |
| 41 | Monthly Progress Review Meetings and Minutes Yr 3 - 4 | 1 day | NA | NA | Wed 12/4/24 | Wed 12/4/24 | 0% | [Vertical tick at start of Q3 2025] | | | | | | | | | | | | | | | | |
| 42 | Monthly Progress Review Meetings and Minutes Yr 3 - 5 | 1 day | NA | NA | Thu 1/2/25 | Thu 1/2/25 | 0% | [Vertical tick at start of Q4 2025] | | | | | | | | | | | | | | | | |
| 43 | Monthly Progress Review Meetings and Minutes Yr 3 - 6 | 1 day | NA | NA | Fri 1/31/25 | Fri 1/31/25 | 0% | [Vertical tick at start of Q1 2026] | | | | | | | | | | | | | | | | |
| 44 | Monthly Progress Review Meetings and Minutes Yr 3 - 7 | 1 day | NA | NA | Mon 3/3/25 | Mon 3/3/25 | 0% | [Vertical tick at start of Q2 2026] | | | | | | | | | | | | | | | | |
| 45 | Monthly Progress Review Meetings and Minutes Yr 3 - 8 | 1 day | NA | NA | Tue 4/1/25 | Tue 4/1/25 | 0% | [Vertical tick at start of Q3 2026] | | | | | | | | | | | | | | | | |
| 46 | Monthly Progress Review Meetings and Minutes Yr 3 - 9 | 1 day | NA | NA | Wed 4/30/25 | Wed 4/30/25 | 0% | [Vertical tick at start of Q4 2026] | | | | | | | | | | | | | | | | |
| 47 | Monthly Progress Review Meetings and Minutes Yr 3 - 1 | 1 day | NA | NA | Thu 5/29/25 | Thu 5/29/25 | 0% | [Vertical tick at start of Q1 2027] | | | | | | | | | | | | | | | | |
| 48 | Monthly Progress Review Meetings and Minutes Yr 3 - 1 | 1 day | NA | NA | Fri 6/27/25 | Fri 6/27/25 | 0% | [Vertical tick at start of Q2 2027] | | | | | | | | | | | | | | | | |
| 49 | Monthly Progress Review Meetings and Minutes Yr 3 - 1 | 1 day | NA | NA | Mon 7/28/25 | Mon 7/28/25 | 0% | [Vertical tick at start of Q3 2027] | | | | | | | | | | | | | | | | |
| 50 | Monthly Progress Review Meetings and Minutes Yr 4 | 232 days | NA | NA | Tue 8/26/25 | Wed 7/15/26 | 0% | [Gantt bar from Q3 2025 to Q3 2027] | | | | | | | | | | | | | | | | |
| 51 | Monthly Progress Review Meetings and Minutes Yr 4 - 1 | 1 day | NA | NA | Tue 8/26/25 | Tue 8/26/25 | 0% | [Vertical tick at start of Q4 2025] | | | | | | | | | | | | | | | | |
| 52 | Monthly Progress Review Meetings and Minutes Yr 4 - 2 | 1 day | NA | NA | Wed 9/24/25 | Wed 9/24/25 | 0% | [Vertical tick at start of Q1 2026] | | | | | | | | | | | | | | | | |
| 53 | Monthly Progress Review Meetings and Minutes Yr 4 - 3 | 1 day | NA | NA | Thu 10/23/25 | Thu 10/23/25 | 0% | [Vertical tick at start of Q2 2026] | | | | | | | | | | | | | | | | |
| 54 | Monthly Progress Review Meetings and Minutes Yr 4 - 4 | 1 day | NA | NA | Fri 11/21/25 | Fri 11/21/25 | 0% | [Vertical tick at start of Q3 2026] | | | | | | | | | | | | | | | | |
| 55 | Monthly Progress Review Meetings and Minutes Yr 4 - 5 | 1 day | NA | NA | Mon 12/22/25 | Mon 12/22/25 | 0% | [Vertical tick at start of Q4 2026] | | | | | | | | | | | | | | | | |
| 56 | Monthly Progress Review Meetings and Minutes Yr 4 - 6 | 1 day | NA | NA | Tue 1/20/26 | Tue 1/20/26 | 0% | [Vertical tick at start of Q1 2027] | | | | | | | | | | | | | | | | |
| 57 | Monthly Progress Review Meetings and Minutes Yr 4 - 7 | 1 day | NA | NA | Wed 2/18/26 | Wed 2/18/26 | 0% | [Vertical tick at start of Q2 2027] | | | | | | | | | | | | | | | | |
| 58 | Monthly Progress Review Meetings and Minutes Yr 4 - 8 | 1 day | NA | NA | Thu 3/19/26 | Thu 3/19/26 | 0% | [Vertical tick at start of Q3 2027] | | | | | | | | | | | | | | | | |
| 59 | Monthly Progress Review Meetings and Minutes Yr 4 - 9 | 1 day | NA | NA | Fri 4/17/26 | Fri 4/17/26 | 0% | [Vertical tick at start of Q4 2027] | | | | | | | | | | | | | | | | |
| 60 | Monthly Progress Review Meetings and Minutes Yr 4 - 1 | 1 day | NA | NA | Mon 5/18/26 | Mon 5/18/26 | 0% | [Vertical tick at start of Q1 2028] | | | | | | | | | | | | | | | | |
| 61 | Monthly Progress Review Meetings and Minutes Yr 4 - 1 | 1 day | NA | NA | Tue 6/16/26 | Tue 6/16/26 | 0% | [Vertical tick at start of Q2 2028] | | | | | | | | | | | | | | | | |
| 62 | Monthly Progress Review Meetings and Minutes Yr 4 - 1 | 1 day | NA | NA | Wed 7/15/26 | Wed 7/15/26 | 0% | [Vertical tick at start of Q3 2028] | | | | | | | | | | | | | | | | |
| 63 | Monthly Progress Review Meetings and Minutes Yr 5 | 1235 days | Wed 12/21/22 | NA | Wed 12/21/22 | Wed 9/29/27 | 65% | [Gantt bar from Q3 2022 to Q3 2024] | | | | | | | | | | | | | | | | |
| 64 | Monthly Progress Review Meetings and Minutes Yr 5 - 1 | 1 day | NA | NA | Thu 8/20/26 | Thu 8/20/26 | 0% | [Vertical tick at start of Q4 2026] | | | | | | | | | | | | | | | | |
| 65 | Monthly Progress Review Meetings and Minutes Yr 5 - 2 | 1 day | NA | NA | Fri 9/25/26 | Fri 9/25/26 | 0% | [Vertical tick at start of Q1 2027] | | | | | | | | | | | | | | | | |
| 66 | Monthly Progress Review Meetings and Minutes Yr 5 - 3 | 1 day | NA | NA | Tue 11/3/26 | Tue 11/3/26 | 0% | [Vertical tick at start of Q2 2027] | | | | | | | | | | | | | | | | |
| 67 | Monthly Progress Review Meetings and Minutes Yr 5 - 4 | 1 day | NA | NA | Wed 12/9/26 | Wed 12/9/26 | 0% | [Vertical tick at start of Q3 2027] | | | | | | | | | | | | | | | | |
| 68 | Monthly Progress Review Meetings and Minutes Yr 5 - 5 | 1 day | NA | NA | Thu 1/14/27 | Thu 1/14/27 | 0% | [Vertical tick at start of Q4 2027] | | | | | | | | | | | | | | | | |
| 69 | Monthly Progress Review Meetings and Minutes Yr 5 - 6 | 1 day | NA | NA | Mon 2/22/27 | Mon 2/22/27 | 0% | [Vertical tick at start of Q1 2028] | | | | | | | | | | | | | | | | |
| 70 | Monthly Progress Review Meetings and Minutes Yr 5 - 7 | 1 day | NA | NA | Tue 3/30/27 | Tue 3/30/27 | 0% | [Vertical tick at start of Q2 2028] | | | | | | | | | | | | | | | | |

| ID | Task Name | Duration | Actual Start | Actual Finish | Start | Finish | % Complete | 2024 | | | | | | | | | | | | | | | | | | | | |
|-----|---|-----------------|--------------------|-------------------|--------------------|--------------------|-------------|------|----|----|----|----|----|----|----|----|----|----|----|----|--|--|--|--|--|--|--|--|
| | | | | | | | | Q3 | Q4 | Q1 | Q2 | Q3 | Q4 | Q1 | Q2 | Q3 | Q4 | Q1 | Q2 | Q3 | | | | | | | | |
| 136 | Army Review and concurrence with RTCs | 15 days | Tue 3/7/23 | Tue 3/21/23 | Tue 3/7/23 | Tue 3/21/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 137 | Submit Draft PIP to Regulators | 1 day | Tue 3/7/23 | Tue 3/7/23 | Tue 3/7/23 | Tue 3/7/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 138 | Regulators Review of Draft PIP | 15 days | Tue 3/7/23 | Tue 3/21/23 | Tue 3/7/23 | Tue 3/21/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 139 | Respond to Comments Draft PIP | 3 days | Wed 3/22/23 | Fri 3/24/23 | Wed 3/22/23 | Fri 3/24/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 140 | Army/Regulators concurrence with Final PIP | 1 day | Fri 3/24/23 | Fri 3/24/23 | Fri 3/24/23 | Fri 3/24/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 141 | Concurrence with Final PIP | 0 days | Fri 3/24/23 | Fri 3/24/23 | Fri 3/24/23 | Fri 3/24/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 142 | Remedial Investigation Field Work (Delineation) (BASE CLIN 0004) | 983 days | Mon 1/23/23 | NA | Mon 1/23/23 | Wed 10/1/25 | 13% | | | | | | | | | | | | | | | | | | | | | |
| 143 | Fieldwork - Baseline Sampling Event | 43 days | Mon 1/23/23 | Mon 3/6/23 | Mon 1/23/23 | Mon 3/6/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 144 | Base Coordination, Utility Locates/Clearance, Permitting | 15 days | Mon 1/23/23 | Mon 2/6/23 | Mon 1/23/23 | Mon 2/6/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 145 | Baseline Sampling | 3 days | Tue 2/7/23 | Thu 2/9/23 | Tue 2/7/23 | Thu 2/9/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 146 | Post-Field Work Sample Analysis & Data Processing | 16 days | Fri 2/10/23 | Mon 3/6/23 | Fri 2/10/23 | Mon 3/6/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 147 | Preliminary CSM Tech Memo | 134 days | Tue 3/7/23 | NA | Tue 3/7/23 | Tue 7/18/23 | 99% | | | | | | | | | | | | | | | | | | | | | |
| 148 | Prepare and Submit Army Draft Preliminary CSM Tech Memo | 92 days | Tue 3/7/23 | Tue 6/6/23 | Tue 3/7/23 | Tue 6/6/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 149 | Army Review of Army Draft Preliminary CSM Tech Memo | 21 days | Wed 6/7/23 | Tue 6/27/23 | Wed 6/7/23 | Tue 6/27/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 150 | Concurrence with Final Preliminary CSM Tech Memo | 0 days | Tue 6/27/23 | Tue 6/27/23 | Tue 6/27/23 | Tue 6/27/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 151 | Technical Project Planning Meeting # 2 | 29 days | Wed 6/7/23 | NA | Wed 6/7/23 | Tue 7/18/23 | 90% | | | | | | | | | | | | | | | | | | | | | |
| 152 | Schedule TPP Meeting | 1 day | Wed 6/7/23 | Wed 6/7/23 | Wed 6/7/23 | Wed 6/7/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 153 | Prepare and Submit Draft Agenda/Meeting Material to the Army | 3 days | Fri 6/16/23 | Tue 6/20/23 | Fri 6/16/23 | Tue 6/20/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 154 | Army Review/Concurrence | 2 days | Wed 6/21/23 | Thu 6/22/23 | Wed 6/21/23 | Thu 6/22/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 155 | Conduct TPP Meeting | 1 day | Tue 6/27/23 | Tue 6/27/23 | Tue 6/27/23 | Tue 6/27/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 156 | Prepare and Submit Draft TPP Meeting Minutes | 2 days | Wed 6/28/23 | Thu 6/29/23 | Wed 6/28/23 | Thu 6/29/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 157 | Army Review of Draft TPP Meeting Minutes | 5 days | Fri 6/30/23 | Fri 7/7/23 | Fri 6/30/23 | Fri 7/7/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 158 | Prepare and Submit Final TPP Meeting Minutes | 2 days | Mon 7/10/23 | Tue 7/11/23 | Mon 7/10/23 | Tue 7/11/23 | 100% | | | | | | | | | | | | | | | | | | | | | |
| 159 | Army Review and Acceptance of Final TPP Meeting Minutes | 5 days | Wed 7/12/23 | NA | Wed 7/12/23 | Tue 7/18/23 | 60% | | | | | | | | | | | | | | | | | | | | | |
| 160 | Field Work - Phase I Sampling Event | 519 days | NA | NA | Mon 10/2/23 | Wed 10/1/25 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 161 | Base Coordination and Permitting | 20 days | NA | NA | Mon 10/2/23 | Mon 10/30/23 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 162 | Utility Clearance | 10 days | NA | NA | Tue 10/31/23 | Tue 11/14/23 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 163 | Install/Develop/Survey Monitoring Wells | 15 days | NA | NA | Wed 9/4/24 | Wed 9/18/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 164 | Phase I Sampling | 45 days | NA | NA | Wed 11/15/23 | Thu 1/18/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 165 | Post-Field Work Sample Analysis & Data Processing | 45 days | NA | NA | Fri 1/19/24 | Thu 3/21/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 166 | Laboratory Analysis - Phase I Sampling | 30 days | NA | NA | Fri 1/19/24 | Thu 2/29/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 167 | Data Validation - Phase I Sampling | 15 days | NA | NA | Fri 3/1/24 | Thu 3/21/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 168 | Technical Project Planning Meeting* | 29 days | NA | NA | Thu 3/14/24 | Tue 4/23/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 169 | Schedule TPP Meeting | 1 day | NA | NA | Thu 3/14/24 | Thu 3/14/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 170 | Prepare and Submit Draft Agenda/Meeting Material to the Army | 3 days | NA | NA | Mon 3/25/24 | Wed 3/27/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 171 | Army Review/Concurrence | 2 days | NA | NA | Thu 3/28/24 | Fri 3/29/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 172 | Conduct TPP Meeting | 1 day | NA | NA | Wed 4/3/24 | Wed 4/3/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 173 | Prepare and Submit Draft TPP Meeting Minutes | 2 days | NA | NA | Thu 4/4/24 | Fri 4/5/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 174 | Army Review of Draft TPP Meeting Minutes | 5 days | NA | NA | Mon 4/8/24 | Fri 4/12/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 175 | Prepare and Submit Final TPP Meeting Minutes | 2 days | NA | NA | Mon 4/15/24 | Tue 4/16/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 176 | Army Review and Acceptance of Final TPP Meeting Minutes | 5 days | NA | NA | Wed 4/17/24 | Tue 4/23/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 177 | Field Work - Phase II Sampling Event | 117 days | NA | NA | Fri 3/22/24 | Mon 9/2/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 178 | Base Coordination and Permitting | 20 days | NA | NA | Fri 3/22/24 | Thu 4/18/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 179 | Utility Clearance | 10 days | NA | NA | Fri 4/19/24 | Thu 5/2/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 180 | Obtain ROE off-post wells | 60 days | NA | NA | Fri 3/22/24 | Mon 5/20/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 181 | Install/Develop/Survey Monitoring Wells | 15 days | NA | NA | Tue 5/21/24 | Tue 6/4/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 182 | Well Installation Summary Report | 90 days | NA | NA | Wed 6/5/24 | Mon 9/2/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 183 | Prepare and Submit Well Installation Summary Report | 10 days | NA | NA | Wed 6/5/24 | Fri 6/14/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 184 | Army Review/Comment | 30 edays | NA | NA | Fri 6/14/24 | Sun 7/14/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 185 | Response to Army Comments | 10 days | NA | NA | Mon 7/15/24 | Wed 7/24/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 186 | Army Approval of Comment Responses | 30 edays | NA | NA | Wed 7/24/24 | Fri 8/23/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 187 | Prepare/Submit Final | 10 days | NA | NA | Sat 8/24/24 | Mon 9/2/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 188 | Concurrence with Final Well Installation Summary Report | 0 days | NA | NA | Mon 9/2/24 | Mon 9/2/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 189 | Phase II Sampling | 10 days | NA | NA | Fri 5/3/24 | Thu 5/16/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 190 | Post-Field Work Sample Analysis & Data Processing | 45 days | NA | NA | Fri 5/17/24 | Thu 7/18/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 191 | Laboratory Analysis - Phase II Sampling | 30 days | NA | NA | Fri 5/17/24 | Thu 6/27/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 192 | Data Validation - Phase II Sampling | 15 days | NA | NA | Fri 6/28/24 | Thu 7/18/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 193 | Technical Project Planning Meeting* | 29 days | NA | NA | Thu 7/11/24 | Tue 8/20/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 194 | Schedule TPP Meeting | 1 day | NA | NA | Thu 7/11/24 | Thu 7/11/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 195 | Prepare and Submit Draft Agenda/Meeting Material to the Army | 3 days | NA | NA | Mon 7/22/24 | Wed 7/24/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 196 | Army Review/Concurrence | 2 days | NA | NA | Thu 7/25/24 | Fri 7/26/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 197 | Conduct TPP Meeting | 1 day | NA | NA | Wed 7/31/24 | Wed 7/31/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 198 | Prepare and Submit Draft TPP Meeting Minutes | 2 days | NA | NA | Thu 8/1/24 | Fri 8/2/24 | 0% | | | | | | | | | | | | | | | | | | | | | |
| 199 | Army Review of Draft TPP Meeting Minutes | 5 days | NA | NA | Mon 8/5/24 | Fri 8/9/24 | 0% | | | | | | | | | | | | | | | | | | | | | |

Appendix J
Field Change Form Template



DRAFT

Date (Month YYYY)

PER- AND POLYFLUOROALKYL SUBSTANCES REMEDIAL INVESTIGATION, FIELD CHANGE REPORT

Yakima Training Center

Yakima, Washington

Joint Base Lewis-McChord Public Works – Environmental Division

IMLM-PWE

MS 17 Box 339500

Joint Base Lewis-McChord, Washington 98433



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**Per- and Polyfluoroalkyl Substances Remedial Investigation
Draft Baseline Sampling Work Plan
Yakima Training Center, Washington**

| | |
|-----------------------|---|
| Installation Name: | Yakima Training Center, Washington (YTC) |
| Contract No.: | W9124J-18-D-0004 |
| Delivery Order No.: | W9124J-22-F-0144 |
| Sampling Dates: | <i>Populate with planned dates</i> |
| Applicable Documents: | <i>Populate with applicable documents</i> |

1. Introduction

Introduction to work to be completed.

2. Approach

Describe work to be completed. Include relevant details such as basis for change, data needs, rationale, and approach to complete the work.

3. Data Use and Validation

Describe the methods for data use and validation.

4. References

Figures:

Figure 1 – As Needed

Tables:

Table 1 – As Needed

Attachments:

Attachment A – As Needed

Figures

Tables

Attachments

Appendix K
Field Standard Operating Procedures and Technical Guidance Instructions

QP 3.06 – Field Activities Documentation

Rev: 1

Rev Date: November 30, 2021

Version Control

| Issue | Revision No. | Date Issued | Page No. | Description | Reviewed By |
|-------|--------------|-------------------|----------|--|--|
| | 0 | November 8, 2016 | All | QP Issued | QMS |
| | 1 | November 30, 2021 | All | Updated Template for QMS Relaunch October 12, 2021 | Matt Spurlin Brian Webb David Gerber |

Approval Signatures

| | | |
|-------------------|--|------------|
| Prepared by: | Matt Spurlin / Brian Webb | 11/30/2021 |
| | _____ | _____ |
| | Names (Preparers) | Date |
| Quality Reviewer: |   | 11/30/2021 |
| | _____ | _____ |
| | Signatures (Quality Reviewers) | Date |
| QMS Approver: |  | 11/30/2021 |
| | _____ | _____ |
| | David Gerber (QMS Approver) | Date |

STATEMENT OF POLICY:

It is Arcadis Environment Business Line (ENV) policy that field activities must be documented to facilitate the interpretation of data; show compliance with project plans, work plans, and contract terms; and to serve as evidentiary records. Documentation reflecting activities performed must be legible, organized, and complete. Applicable regulatory and client requirements should be considered when documenting field activities. Project-specific requirements for documentation typically should be described in the Work Plan, Field Sampling Plan (FSP), and/or in the Quality Assurance Project Plan (QAPP).

1 Purpose

The purpose of this Quality Procedure (QP) is to provide a standard procedure for the documentation of fieldwork activities. This documentation pertains to site-related projects, but is not limited to the collection of samples, subsurface information, and oversight of construction activities. Field documentation must include, at a minimum, project title and number, date and times of activities, the identification of the employee performing the work, and the specifics of the work being performed.

2 Responsibilities

Certified Project Manager (CPM) – is responsible for the project-related administration of this QP.

Quality Consultant – is responsible for providing quality assurance and quality control guidance to the CPM in implementing this procedure. Note that for federal projects, there are specific requirements and qualifications for the QA Officer assigned to the project.

Project Team Members – who are assigned to document field activities, are responsible for compliance with this procedure.

Quality Reviewer – The Quality Reviewer is responsible for final review of this Quality Procedure (QP). Quality Reviewers may be a Quality Consultant, QMS Document Owner, Technical Solution Leader, Community of Practice Leader, or another qualified subject matter expert (SME).

3 Terms and Conditions

Field Sampling Plan (FSP) – A document that describes the procedures and protocols necessary to complete field sampling and data collection activities.

Work Plan – A document that describes proposed project activities.

Quality Assurance Project Plan (QAPP) – A document that prescribes the quality assurance/quality control (QA/QC) procedures to be followed. Uniform Federal Policy (UFP) QAPPs are now frequently required for environmental projects by most federal regulatory agencies. A UFP QAPP includes Worksheets used to document the entire project plan developed following the systematic planning process. For more details on the UFP QAPP see <http://www.epa.gov/fedfac/documents/qualityassurance.htm>. Note that if the project QAPP is written following the UFP format, it will also contain a description of the sampling rationale and sampling locations as well as QA/QC requirements. The UFP QAPP format is designed to capture the entire systematic planning

process. If a UFP QAPP is written for a project, a separate FSP is generally not required unless specified by the particular client or contract.

Technical Guidance Instruction (TGI) – Document describes the procedure and/or protocol necessary to conduct a specific activity.

4 Related Documents

Forms used for documenting field activities may be included as attachments to the FSP or the QAPP and may include the following examples:

- Chain-of-custody (COC) form;
- Sample data log;
- Field modification form;
- Sample receipt form;
- Corrective action form;
- Field activity log;
- Calibration log;
- Analysis request and chain-of-custody record;
- Daily quality control reports;
- Purge log;
- Soil boring log.

Examples of TGIs with forms and check-lists can be found in the [QMS Document Library](#).

5 Description of the Procedure

1. General Requirements

1.1 Documentation Format

Documentation of field activities provides an accurate and comprehensive record of the work performed sufficient for a technical peer to reconstruct the day's activities and confirm that necessary client, regulatory, contract, and work plan requirements were met. General requirements include:

- Use of field books (preferably bound) as the primary source for information collection and recording. Field books should be dedicated to the project and appropriately labeled.
- Use of a Field Activity Log is suggested to formally document activities and events as a supplement to bound field books. The Field Activity Log can be a standard or project-specific form or a bound field book. Preprinted standard forms are available for many activities and should be used whenever possible. These forms will provide prompts and request additional information that may be useful and/or needed. Project-specific field forms may be generated, or existing forms may be modified to meet specific project needs. Client-supplied forms may be substituted, as required.
- Appropriate header information is documented on the first page of notes for each day of fieldwork, including project title, project number, date, time, author, and relevant setting information such as weather conditions, topography, surface water conditions, observed site activities/uses, and other persons in field team. In addition, include on every page of notes the page number and date. Project-specific information depends on

the nature of work being performed and should be discussed by the project team prior to commencing fieldwork. As appropriate, dedicated field logs/journals or forms should be used. When Field Activity Log Forms are used, information fields that are not applicable should be noted as such with the symbol "N/A" or other appropriate notation.

- Field documentation entries shall be made using indelible ink.
- Data entries shall be legible. A single line should be drawn through incorrect entries and the corrected entry written next to the original strikeout. Strikeouts are to be initialed and dated by the originator.
- Units of measurement shall be specified. The level of accuracy shall be indicated (e.g., observed estimate vs. quantified census from direct count).
- Field records are to be maintained in project files unless otherwise specified by a client or stipulated by a contract.
- Unless addressed specifically by a client or stipulated by a contract, site photographs should be taken to document the general setting and landscape as well as site-specific issues/resources of interest. Photo locations and the compass direction of view should be recorded in the notes with the photo number.
- Alternatively, use of field tablets with data plans and electronic data collection software supported by Cloud services (e.g., Fulcrum Collector, Survey123) that are pre-loaded with appropriate data collection forms (e.g., method-specific sampling, soil logging, photo logs, H&S tailgate meetings, well/piezometer/lysimeter installations, electronic chain of custody, etc.). All forms adhere to the relevant documentation format requirements (described above and below this section) for data collection with conventional paper methods. Electronic data collection services, as well as training, are available to Arcadis field staff through the FieldNow® program and while optional are highly encouraged.

1.2 Documentation Entries

A chronology of field events should be recorded. General entry requirements include:

- Visitors to the site, including owner and regulatory agency representatives
- Summary of pertinent project communications with the client, regulators, or other site visitors during the fieldwork
- Other contractors or entities working on site
- A description of the day's field activities, generally in chronological sequence or in order of significance, using military time notation (e.g., 9:00 a.m. as 0900, and 5:00 p.m. as 1700)
- If applicable, calibration of measuring and test equipment and identification of the calibration standard(s) (use a Calibration Log, if available, with cross-reference entered into the field book)
- Field equipment identification, including information such as the type, manufacturer, model number, or other specific information
- If applicable sampling activities are being performed, weather information such as temperature, wind speed and direction, precipitation, time of measurement, and units
- Documentation of safety meeting (e.g., tailgates and tailboards) topics and attendees
- Verification of subsurface utility clearance in accordance with ENV policy
- Safety and/or monitoring equipment readings, including time of measurements and units
- If applicable, specific forms used for collection of data are referenced in the field notebook
- Subcontractor progress and/or problems encountered
- Changes in the scope of work
- Other unusual events.

2. Specific Requirements

2.1 Sample Collection

Sample collection data are documented in a bound field book, electronic field forms provided by the FieldNow® program, and/or on a Field Activity Log. Where both are being used, information contained in one is cross-referenced to the other. Entries such as the following examples should be consistent with the requirements in the project-specific Work Plan, FSP, and QAPP:

- Sample identification number, location taken, depth interval, sample media, sample preservative, collection time, and date
- Sample collection method and protocol
- Physical description of the sample (using a standard classification system for soil)
- If a composite sample, include the number, location(s), and depth(s) of grab samples incorporated in the composite
- Quality-related samples (e.g., field duplicates, trip blanks, equipment rinse, blanks matrix spikes, and matrix spike duplicates)
- Container description and sample volume
- Pertinent technical data, such as pH, conductivity, temperature, and head-space readings
- Pertinent technical comments
- Identification of personnel collecting the sample.

2.2 Sample Labeling

Sample labels must be prepared and attached to sample containers. Labels are either provided by the laboratory performing the analyses or are generated internally. Labels should be indelible and securely attached to the container. The information to be provided may include:

- Sample identification number
- Sample date, initials, or name of who collected the sample, and collection time
- Physical description of the sample (e.g., water, solid, gas, or other physical medium)
- Analytical parameters and method(s)
- Preservatives, if present
- Sample location and depth, if applicable
- Client.

Although this information is typically written out, it can also be recorded in an electronic tracking system if a bar code is used.

2.3 Analysis Request and Chain-of-Custody Record

A critical component of data collection is the documentation that the samples were obtained from specific locations and received by the laboratory or archive without alteration. Evidence of collection, shipment, laboratory receipt, and laboratory custody until disposal or archive must be properly documented. Documentation will be accomplished through a COC record that documents each sample and identifies the individuals responsible for sample collection, shipment, and receipt. A sample is considered in custody if at least one of the following criteria is met:

- The sample is in a person's actual possession
- The sample is in unobstructed view, after being in the person's actual possession
- The sample is locked and only accessible by the custodian after having been in the person's actual possession
- The sample is in a secured area, restricted to authorized personnel (e.g., laboratory).

An example COC form to be used by ENV personnel in collecting and shipping samples can be found on the corporate Intranet. A laboratory typically will not accept samples for analysis without a correctly prepared COC form. The COC must be signed by each individual who has the sample in his/her custody. Each sample shipped to a laboratory for analyses must be documented on the COC. Information on this form correlates with other supporting documentation, including the field logbook, sample labels, and sample collection logs.

The COC documents the elapsed time and the custodians of the sample from the time of its collection. The individuals who have physically handled the sample(s) or witnessed initial sample collection and packaging (sample team member) must be identified on the form. A sample team member relinquishes the sample by signing the COC. Individuals who either relinquish or receive samples must include their complete names, company affiliation, and the date and time the sample(s) were relinquished. The times that the samples are relinquished and received by the next custodian should coincide, with the exception of transfer by commercial carriers. These carriers will not be required to sign the COC.

If a sample is to be stored for a period of time (e.g., overnight), measures are taken to secure the sample container in a manner that only provides access to the custodian of record. If samples are relinquished to a commercial carrier (i.e., UPS or Federal Express), the carrier waybill number is recorded, and a copy of the waybill is attached to the COC. These documents are maintained with other field documentation. The original COC is sealed inside a zip-top plastic bag and placed inside the shipping container with the samples.

If corrections are made to the COC, the corrections should be made (single line through the error, initial, and date) by the originator of the change, and, if necessary, an explanation of the change should be provided. The documentation should be of a level of detail that clearly documents the change to a third-party reviewer.

Guidance for choosing a laboratory and completing analyses requests and COC can be found in QP 2.09- Subcontracting Laboratory Services and on the corporate Intranet for the Arcadis Laboratory Program (ALP) and should also be described in the project-specific planning documents (i.e., Work Plan, FSP, or QAPP).

The option to use the Electronic Chain of Custody (eCOC) form in conjunction with the appropriate sample application(s) may be available through the FieldNow® program but is currently limited to a select list of approved analytical laboratories. Use of the eCOC application is intended to reduce common transcription errors both by field staff and laboratory staff on a conventional handwritten paper COC. Once the eCOC form is completed and approved on the field tablet by field staff, a PDF version of the form is automatically emailed to each assigned team member. In addition, a dedicated or mobile printer is recommended for printing a hard copy of the completed eCOC to be included in each sample cooler to meet laboratory requirements.

2.4 Subsurface Logs

Test pits, soil borings, monitoring wells or rock coreholes wells, and piezometer installations are to be recorded in bound field books or electronic soil logging field form provided by the FieldNow® program and may be supplemented with prepared forms. Personnel completing the log are to supply the following information:

- Administrative and technical information included in the header.
- Types of equipment used (e.g., drill rig type, drilling tools used [including diameter and length], or backhoe model).
- Subcontractor/driller used.
- Descriptions of subsurface materials encountered, and the number and type of samples collected, if any.
- Subsurface exploration depth and units of measure.
- For drilling, length of recovery.
- Sample type and sample number for geotechnical or analytical samples collected. These data are to be also entered on the sample collection log (if used) and the sample label.
- Classification standard protocol used, if any (e.g., ASTM International Standard Penetration Test).
- Narrative description of the soil, sediment, or bedrock (using standard classification system) and other pertinent information.
- Additional data, such as background and sample vapor/gas readings, observation of sheens, non-aqueous-phase liquid, depth to water (if encountered), presence of (but generally not description of) odors, changes in drilling conditions, and other pertinent information.
- Description of the materials used to seal the boring unless it is completed as a well or piezometer.

When using the electronic soil logging field form, draft logs can be exported to appropriate formats through Arcadis's internal reporting services for QC. Revisions can be made to the soil logging forms using the field tablets, then re-synced to the server and re-exported to PDF as final draft.

2.5 Monitoring Well/Piezometer Installation

In addition to requirements in Section 2.4, subsequent well or piezometer development activities may involve transcription of field data from the field book onto a computer-based boring log. The field notebook or electronic soil logging/well construction field form provided by the FieldNow® program is to be used to identify the chronology and major events of the installation activity, and the computer-based boring log is to be used to correlate the geologic strata to the major elements of the monitoring well construction. Information to be collected and recorded must meet the regulatory and client requirements and may include the following:

- Location identity
- Screen and riser type, length, diameter, and location
- Diameter
- Total depth
- Sump location and depth and diameter
- Materials of construction (e.g., stainless steel, polyvinyl chloride, or other material)
- Seal type(s) and or depth(s)
- Sand or gravel pack type, including materials (e.g., silica) and gradation
- Depth to water before and after installation.

When using the electronic soil logging/well construction field form, draft logs can be exported to appropriate formats through Arcadis's internal reporting services for QC. Revisions can be made to the soil logging/well construction forms using the field tablets, then re-synced to the server and re-exported to PDF as final draft.

2.6 Air Sampling Logs

At a minimum, air sampling documentation should include:

- Start and finish time of sampling
- Sampling location
- Sampling method/media
- Volume sampled.

2.7 Construction, Demolition, Abandonment, and Related Activities

Monitoring and documentation of construction and comparable activities shall be documented in bound field books and/or on appropriate company forms and should include similar information as specified above, including information such as:

- Project name and number
- Owner or client name
- Contractor or subcontractors performing the work
- Contractor or subcontractor superintendent(s) and personnel (as available) on site
- Chronological sequence and description of work activities performed, including workday start and completion times
- Reference to contract sections, work plans, or specifications describing work being performed
- Reference to relevant permit conditions and regulatory requirements and/or reference to regulatory guidance documents controlling work approach
- Listing of all trades performing work by contractor and subcontractor
- Hours worked per trade
- Work hours per day per shift, if applicable
- Equipment on site (e.g., description, model number, size, and type) and hours of use
- Listing of equipment on site being left idle
- Description and quantity of materials used or incorporated, with reference to contract or specification item number, if feasible; include simple sketch of excavation with approximate dimension, if applicable
- Calculations with dimensions for quantities of material used or incorporated
- Delineation of the work area and access routes (e.g., fencing, flagging, or staking), confirmation that activities occurred within the work area or description of work occurring outside the delineated work area and justification (as needed), and characterization of impacts outside the designated work area
- Documentation of compliance with speed limits, dust control, erosion control best management practices, and other basic elements of construction activities as dictated by project work plans and applicable permits and regulatory criteria.

2.8 Daily Safety Meeting

A Daily Safety Meeting is to be conducted and documented each workday prior to the initiation of field activities, with on-site ENV personnel, contractors, subcontractors, and visitors if possible. Safety topics discussed are entered on the Daily Safety Meeting Form (available on the corporate Intranet). Topics discussed should include site-specific conditions, procedures to be followed that day, and protective equipment. A printed listing of the attendees at the meeting and their signatures should be included. Other required data are:

- Identification of the individual conducting the meeting and his/her signature
- Identification of the project supervisor and project manager.

The option to use the electronic H&S tailgate meeting field form is available through the FieldNow® program. The completed form can be exported to appropriate formats through Arcadis's internal reporting services for documentation purposes.

2.9 Calibration

Documentation of the calibration and calibration results shall be made for field equipment requiring calibration measuring and test equipment calibration data are recorded in the field book or on the Field Activity Log. Calibration data include the following:

- Unique identification of instrument being calibrated, including type, model, and serial number
- Date and time of calibration
- Standards used in the calibration, including standard identity, concentration, lot number, and manufacturer of the standard
- Instrument reading with respect to each calibration standard
- Comments, as necessary, regarding instrument performance.

2.10 Photographs and Videos

When the client allows, photographs and videos may be used to help document pre-, active, and post-field activities. In sensitive areas (e.g., secured, or confidential), the client must be contacted to evaluate security procedures concerning use of photographs or videos. Photographic and video documentation should include project title, project number, date, time, and description of conditions. The time should also be documented if time is important to a sequence of photographs.

Photographs are documented by numbering digital photographs and identifying the number and subject on the Field Activity Log or the electronic photograph log through the FieldNow® program. Individual prints may be marked with a stamp or preprinted self-adhesive labels, or by writing the project number and sequential number of each photograph and referencing the numbers in the field book, the Field Activity Log, or a dedicated photo log. Videos used for field documentation are to be identified by project title, project number, and description.

2.11 Subcontractor Preparedness Checklist

Prior to starting work, a review is to be made and documented of a subcontractor's preparedness to perform specified activities. This review may be documented on the Field Activities Log or on checklists that may be developed according to requirements for subcontracted work activities. Particular emphasis should be on site-specific issues that may require special consideration such as health and safety, access, and unique settings. These should be discussed in advance with the CPM and the client in developing and implementing the Scope of Work.

- END OF PROCEDURE -

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TGI – Sample Chain of Custody

Rev: 3

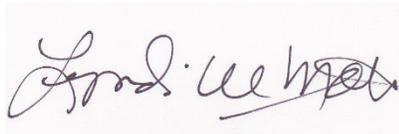
Rev Date: March 28, 2022

Version Control

| Issue | Revision No. | Date Issued | Page No. | Description | Reviewed By |
|-------|--------------|-------------------|----------|---|-----------------|
| | 0 | April 19, 2017 | All | Re-write to COC only | Richard Murphy |
| | 1 | May 23, 2017 | 4,7,9 | Add: Guidance on use of previous version of TGI. Add: Info on COCs for multiple shipping containers Modify: Move letter i. to letter m. and change to “when appropriate” | Peter Frederick |
| | 2 | April 29, 2020 | 4, 11 | Remove obsolete link | Lyndi Mott |
| | 3 | December 28, 2022 | All | Updated Arcadis format Added to 6c. Collection time between COC and container must match. Added to 6o. Add name of overnight courier when relinquishing samples. Updated reference documents and added internet links. | Lyndi Mott |

Approval Signatures

Prepared by:

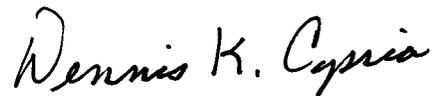


3/28/2022

Lyndi Mott (Preparer)

Date

Reviewed by:

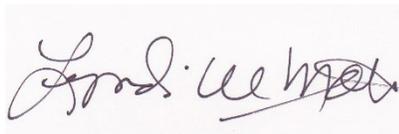


3/28/2022

Dennis Capria (Chain of Custody Reviewer)

Date

Reviewed by:



12/22/2021

Lyndi Mott (Subject Matter Expert)

Date

1 Introduction

This Technical Guidance Instruction (TGI) provides the procedure for Arcadis field personnel for required documentation during the collection of environmental field samples and transfer of custody to a laboratory. It provides direction for completion of the Chain of Custody form that must accompany collected field samples for analysis by a laboratory.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

This TGI describes the general Chain of Custody (COC) procedures and guidance instructions for samples collected from project sites that are relinquished from Arcadis' possession.

COC is defined as the maintenance of an unbroken record of possession of an item from the time of its collection through some analytical or testing procedure. COC is typically documented by a written record of the collection, possession, and handling of samples collected from a project location. Each sample will be tracked by a documented record that efficiently documents the individuals who were responsible for the sample during each successive transfer of that sample to various recipients beyond Arcadis' possession. This information can be used to legally establish the integrity of the samples and therefore the analytical results derived from the samples. This

information can be used in addition to other records and documentation regarding the samples, such as field forms, field logs, and photographs.

A sample is considered under custody if:

- It is in your possession; or
- It is in your view, after being in your possession; or
- It was in your possession and then you then locked it up to prevent tampering; or
- It is in a designated secure area.

Continued use of previous version of TGI:

Although not recommended, Arcadis program-, project-, and client-teams may be able to use the previous version of this TGI provided that it meets all of the quality expectations of Arcadis and client and meets applicable regulatory requirements. It is up to the program, project, and/or client-team leader to determine whether it is appropriate to adopt the current TGI or to continue using the previous version.

However, all new work not associated with the previous version of this TGI must be performed with the current version of the TGI.

When adopting this new TGI, users of the previous versions must be aware that specific handling, packing, and shipping procedures and guidance has been removed and that those should be addressed within program or project plans (e.g., Quality Assurance Project Plans (QAPP), Work Plans, Sampling and Analysis Plans (SAPs), etc.) or in a more detailed TGI specific to that sampling activity, whether related to media, constituent/analyte, client, state, etc.

In addition, adopting this new TGI will require users to refer to the Arcadis Department of Transportation (DOT) Safety Program for procedures and guidance on the determination and handling, packing, and shipping of samples that are or may be considered hazardous materials.

4 Personnel Qualifications

Arcadis personnel performing work under the purview of this TGI will have received appropriate training and have field experience regarding the collection of samples from project locations. Arcadis personnel will have all other applicable and appropriate training relevant to the sampling work and project site.

5 Equipment List

The following list provides materials that may be required for each COC. Project reporting and documentation requirements must be reviewed with the CPM prior to execution of work. Additional materials, tools, equipment, etc. may be required, and project staff are required to verify with the CPM and/or Technical Expert what specific equipment is required to complete the COC.

- Indelible ink pen (preferably either black or blue ink);
- COC form (**Appendix A**) from either Arcadis, laboratory receiving and analyzing the samples, or other applicable and appropriate entity for the work performed;
- When appropriate, such as for litigation or expert testimony work, custody seals or tape.

6 Cautions

One way in which the law tries to ensure the integrity of evidence is by requiring proof of the chain of custody by the party who is seeking to introduce a particular piece of evidence.

A proper chain of custody requires three types of affirmations: (1) affirmation that a sample is what it purports to be (for example, soil collected from a specified location and depth); (2) affirmation of continuous possession by each individual who has had possession of the sample from the time it is collected until the time it is analyzed or held by a laboratory; and (3) affirmation by each person who has had possession that sample remained in substantially the same condition and not contaminated or affected by outside influences from the moment one person took possession until the moment that person released the evidence into the custody of another (for example, affirmation that the sample was stored in a secure location where no one but the person in custody had access to it).

Proving chain of custody is necessary to "lay a foundation" for the samples in question, by showing the absence of alteration, substitution, or change of condition.

Ensure that appropriate sample containers with applicable preservatives, coolers, and packing material are planned for and provided at the site at the time of sample collection.

Understand the offsite transfer requirements of the samples for the facility at which samples are collected.

If overnight courier service is required schedule pick-up or know where the drop-off service center is located and the hours of operation.

An Arcadis employee appropriately trained at the correct level of internal hazardous materials/DOT)shipping must complete an Arcadis shipping determination to address applicable DOT and International Air Transport Association (IATA) shipping requirements. Review the applicable Arcadis procedures and guidance instructions for sample packaging, and labeling. Prior to using air transportation, confirm air shipment is acceptable under DOT and IATA regulations.

The person relinquishing possession of the samples or other member of the project team should contact the final recipient of the samples to confirm receipt and review any special provisions on the COC or questions that they may have.

7 Health and Safety Considerations

Follow the health and safety procedures outlined in the project/site Health and Safety Plan (HASP) as well as other applicable H&S requirements, such as:

- Arcadis Hazardous Material/DOT handling, packaging, and shipping training
- Project site-specific H&S training
- Client-specific H&S training
- Constituent-specific H&S training
- Media-specific H&S training

8 Procedure

Collected samples must be uniquely identified, and properly documented, containerized, labeled with unique identifier, possessed in a secure manner during remainder of sampling event, packaged, and shipped to recipient laboratory.

Sample Identification

The method of sample identification depends on the type of measurement or analyses performed. In some cases, in-situ measurements of existing conditions and/or sample location must be made during sample collection.

These data will be recorded directly on field forms, logbooks, or other project record data sheets used to permanently retain this information for the project file. Examples of location identification information includes: latitude/longitudinal measurements, compass directions, well number, building number, floor number, room name, or proximity to a site feature unique to the site. Examples of in-situ measurements are pH, temperature, conductivity, flow measurement, or physical condition of the media being sampled. Physical samples collected are identified by a unique identifying number or code on a sample tag or label. These physical samples are removed from the sample location and transported to a laboratory for analyses.

In some cases, before samples are placed into individual containers and labeled as individual samples, samples may be separated into portions depending upon the analytical methods and required duplicate or triplicate analyses to be performed.

When completing a COC for samples, personnel must complete the following:

1. Written COCs must be completed with indelible ink (preferably either black or blue colored ink).
2. Written COCs must be completed using legible printed writing, and not cursive writing.
3. All entry fields on the COC form must be completed. If information is not applicable for a specific entry field, personnel will either put "N/A" or use a strike-out line or dash like "-----" to indicate no applicable information is needed for that field.
4. Use of quotation marks or lines/down arrows to represent repetitive/duplicative text in similar fields.
5. Regardless of the type or specific COC form, the following pertinent information must be provided on the COC form:
 - a. Arcadis project number
 - b. Arcadis project name
 - c. Project location, including street address, city, state, building number, providing as much detail as appropriate
 - d. Recipient laboratory contact and sample receiving shipping location information
 - e. Entities'/persons' contact information for who will be receiving analytical results
 - f. Name of sampler, i.e., person collecting sample and relinquishing possession of samples to the next entity in the chain of custody
 - g. Date of sample collection
 - h. If appropriate for the sample media, contaminant/constituent of concern, or analytical method, document time of sample collection using standard military time
 - i. Sample analytical method(s)

- j. Turnaround time required for analyses and/or reporting
- k. Instructions to laboratory regarding handling, timing, analyses, etc. as applicable and appropriate.
- l. Printed name and signature of the individual person who collected the samples and relinquishing possession of the samples
- m. If appropriate or when documentation of the specific sample collection method will influence how the laboratory handles, prepares, or analyzes the samples, document the sample collection methodology used for collecting the samples (e.g., ASTM D5755)

6. The following additional specific information will be entered on the COC form, regardless of what type of COC is being used:

- a. Unique Sample Identifier – The sample identifier (ID) must be unique to the individual sample it is applied to. The information in which the sample ID conveys is determined by the CPM, Technical Expert, and/or other project team members in advance of sample collection so that sample identification is consistently applied for the project. The sample nomenclature may be dictated by a specific client, program, or project database and require unique identification for each sample collected for the project. Consult with the CPM and/or Technical Expert for additional information regarding sample identification.

The sample ID could convey specific information regarding the sample to aid personnel in recognizing what the sample represents, or they may be arbitrary so as to facilitate the anonymity of the sample location, media, constituent of concern, project site, etc.

Examples of unique identifiers include:

- 1. Well locations, grid points, or soil boring identification numbers (e.g., MW-3, X-20, SB-30). When the depth interval is included, the complete sample ID would be “SB-30 (0.5-1.0) where the depth interval is in feet. Please note it is very important that the use of hyphens in sample names and depth units (i.e., feet or inches) remain consistent for all samples entered on the chain of custody form. DO NOT use the apostrophe or quotes in the sample ID.
 - 2. Sample names may also use the abbreviations “FB,” “TB,” “FD” and “DUP” as prefixes or suffixes to indicate that the sample is a field blank, trip blank, or field duplicate, respectively.
- b. List the date of sample collection. All indicated dates must be formatted using either mm/dd/yy (e.g., 03/07/09) or mm/dd/yyyy (e.g., 03/07/2009).
 - c. List the local time that the sample was collected. The time value should be presented using military format. For example, 3:15 P.M. should be entered as 15:15. The time listed on the COC form must match the sample collection time on the sample container(s).
 - d. Samples should be indicated to be either “Grab” or “Composite”. Grab samples are collected from only one unique location at one specific point in time.
 - e. Composite samples are a group of individual samples that are combined for analysis in their totality. Composite samples need to be documented if they are either collected from a number of different locations over a broader area to be representative of the entire area being sampled, or if they are representative of a single location over an extended period of time.

- f. If used, preservatives for the individual sample will be noted.
 - g. The requested analytical method(s) that the samples are being analyzed for must be indicated. As much detail, as necessary, should be presented to allow the analytical laboratory to properly analyze the samples. For example, polychlorinated biphenyl (PCB) analyses may be represented by entering “EPA Method 8082 – PCBs” or “EPA PLM 600-R93-116.” In cases where multiple analytical methods and/or analytical parameters are required for an individual sample, each method should be indicated for the sample (e.g., EPA 8082/8260/8270 or EPA PLM/400-point count).
 - h. If there are project-specific sample analytes to be reported, they should be specifically listed for each individual sample (e.g., 40 CFR 264 Appendix IX).
 - i. The total number of containers for each analytical method requested should be documented. This information may be included under the parameter or as a total for the sample.
 - j. When necessary, note which samples should be used for site specific matrix spikes in the Remarks or Comments field.
 - k. Indicate special project-specific requirements pertinent to the handling, shipping, or analyses. These requirements may be on a per sample basis such as “extract and hold sample until notified,” or may be used to inform the laboratory of special reporting requirements for the entire sample delivery group (SDG).
 - l. Indicate turnaround time (TAT) required for samples on COC. If individual samples have differing TATs, the different TATs for each sample or groups of samples must be clearly indicated.
 - m. Provide contact name and phone number in the event that problems are encountered when samples are received at the laboratory. The person relinquishing possession of the samples or other member of the project team should contact the final recipient of the samples to confirm receipt and review any special provisions on the COC or questions that they may have.
 - n. If available, attach the Laboratory Task Order or Work Authorization forms.
 - o. The “Relinquished By” field must contain the signature of the Arcadis person who relinquished custody of the samples to the next entity in the chain of custody, which may be another person, the shipping courier, or the analytical laboratory. If a courier, enter the shipping courier in the “Received by” such as FedEx. The date/time relinquished should be when the person signs the COC and seals the cooler or shipping container for pick-up by the shipping courier.
 - p. Dates and times must be indicated using the following format:
 - 1) Date: either mm/dd/yy e.g., 01/01/17 OR mm/dd/yyyy e.g., 01/01/2017
 - 2) Time: use military format, e.g., 9:30 a.m. is 0930 and 9:30 p.m. is 2130
 - q. The “Received By” section is signed by sample courier or laboratory representative who received the samples from the sampler. The laboratory will sign upon laboratory receipt from the overnight courier service.
7. When more than one page of the COC form is required to complete the total number of samples, use as many sheets as necessary to accurately and clearly, document the samples and information. Some COCs may have a standard first page/cover page, and subsequent pages may not contain all the detailed fields as

the first page/cover page. Ensure that any subsequent pages convey all of the necessary and pertinent information for each individual sample as required in this procedure document.

8. Pages of the COC must retain a page count of the total number of pages; e.g., Page 1 of 3, Page 2 of 3, Page 3 of 3.
9. Upon completing the COC forms, forward the original signed COC with the sample package. Ensure that the original COC form is secured with the sample package so that it remains with the physical samples for the duration of transport and handling to its final destination and ensure that the COC form will not be become damaged or rendered unreadable due to sample breakage/leakage if stored inside the sample shipping container or outside influences if COC is stored in an outside plastic pouch to the container.
10. If you've collected enough samples that would require more than one container to ship them all to the same laboratory or location, then each separate/individual container that contains any number of samples must have a separate COC representing only those samples contained within that specific container. For example, if you have 3 total shipping containers for all of your samples, you must have a total of 3 separate, individual COCs for each of the 3 containers representing only those samples in their representative container. Thus, every container holding samples must have its own, individual COC.
11. If electronic chain of custody (eCOC) forms are utilized, ensure that the requirements of this procedure and guidance instructions are followed to the extent possible. Verify that proper signature and COC procedures are maintained with the CPM and/or Technical Expert when using eCOC.

9 Waste Management

Not Applicable.

10 Data Recording and Management

The original signed COC shall be submitted with the samples. Copies of COC records will be transmitted to the CPM or designee at the end of each day unless otherwise directed by the CPM. The sampling team leader retains copies of the chain of custody forms for filing in the project file. Record retention shall be in accordance with client- and project-specific requirements and Arcadis policies, the most stringent will apply.

The option to use the Electronic Chain of Custody (eCOC) form in conjunction with the appropriate sample application(s) may be available through the FieldNow® program but is currently limited to a select list of approved analytical laboratories. Use of the eCOC application is intended to reduce common transcription errors both by field staff and laboratory staff on a conventional handwritten paper COC. Once the eCOC form is completed and approved on the field tablet by field staff, a PDF version of the form is automatically emailed to each assigned team member. In addition, a dedicated or mobile printer is recommended for printing a hard copy of the completed eCOC to be included in each sample cooler to meet laboratory requirements.

11 Quality Assurance

COC forms will be legibly completed in accordance with this procedure and guidance instruction document, as well as other applicable and appropriate project documents such as SAP, Quality QAPP, Work Plan, or other project guidance documents.

COC records will be reviewed by the CPM or their appropriate designee for completeness and accuracy to the applicable requirements. Non-conformances will be noted and corrected in a timely manner on the copies retained by Arcadis as well as contacting the ultimate receiving entity for correction to the originally signed COC in their possession.

12 References

Arcadis Transportation Safety Program requirements, procedures, and guidance instructions.

EPA Samplers' Guide – Contract Laboratory Program Guidance for Field Samplers, EPA document EPA-540-R014-013 October 2014 https://www.epa.gov/sites/default/files/2015-03/documents/samplers_guide.pdf.

EPA Region III – Sample Submission Procedures for the Office of Analytical Services and Quality Assurance (OASQA) Laboratory Branch revision 14.0 October 18, 2018, <https://www.epa.gov/sites/default/files/2018-12/documents/sample-submission-procedures-rev14.pdf>.

EPA Region IV Science and Ecosystem Support Division Operating Procedure for Sample and Evidence Management May 25, 2016, <https://www.epa.gov/sites/default/files/2015-06/documents/Sample-and-Evidence-Management.pdf>.

Attachment A

Chain of Custody and Laboratory Analysis Request Form

| | | | | | | | | | | | | | | | | |
|---|-------------------------|---|--|--|---------------|------------------------|--|-------------------------------|--|---------------|--|---|-------------------|---|----------------|--|
|  | | ID# <input style="width: 80px; height: 20px;" type="text"/> | CHAIN OF CUSTODY & LABORATORY ANALYSIS REQUEST FORM | | | | | | | | | | Page ____ of ____ | Lab Work Order # <input style="width: 100px; height: 20px;" type="text"/> | | |
| Send Results to: | Contact & Company Name: | Telephone: | Preservative | | | | | | | | | | | Keys Preservation Key: A. H ₂ SO ₄ B. HCL C. HNO ₃ D. NaOH E. None F. Other: _____ G. Other: _____ H. Other: _____ Containment Information Key 1. 40 ml Vial 2. 1 L Amber 3. 250 ml Plastic 4. 500 ml Plastic 5. Encore 6. 2 oz. Glass 7. 4 oz. Glass 8. 8 oz. Glass 9. Other: _____ 10. Other: _____ Matrix Key: SO - Soil W - Water T - Tissue SE - Sediment SL - Sludge A - Air NL - NAPL/Oil SW - Sample Wipe Other: _____ | | |
| | Address: | Fax: | Filtered (✓) | | | | | | | | | | | | | |
| | City State Zip | E-mail Address: | # of Containers | | | | | | | | | | | | | |
| Project Name/Location (City, State): | | Project #: | Container Information | | | | | | | | | | | | | |
| Sampler's Printed Name: | | Sampler's Signature | | PARAMETER ANALYSIS & METHOD | | | | | | | | | | | | |
| SAMPLE ID | Collection | | Type (✓) | | Matrix | | | | | | | | | | REMARKS | |
| | Date | Time | Comp | Grab | | | | | | | | | | | | |
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| Special Instructions/Comments | | | | | | | | | | | | <input type="checkbox"/> Special QA/QC Instructions (✓) | | | | |
| Laboratory Information and Receipt | | Relinquished By | | Received By | | Relinquished By | | Laboratory Received By | | | | | | | | |
| Last Name: | | Cooler Custody Seal (✓) | | Printed Name: | | Printed Name: | | Printed Name: | | Printed Name: | | | | | | |
| | | <input type="checkbox"/> Intact <input type="checkbox"/> Not Intact | | Signature: | | Signature: | | Signature: | | Signature: | | | | | | |
| <input type="checkbox"/> Cooler packed with ice (✓) | | Sample Receipt | | Firm: | | Firm: | | Firm: | | Firm: | | | | | | |
| Specify Turnaround Requirements: | | Condition/Cooler Temp: _____ | | Date/Time: | | Date/Time: | | Date/Time: | | Date/Time: | | | | | | |
| Shipping Tracking #: | | | | | | | | | | | | | | | | |

SOP – Sample Chain of Custody Rev1_May 23, 2017

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QP 3.07 – Calibration and Control of Measuring and Test Equipment

Rev: 1

Rev Date: October 20, 2021

Version Control

| Issue | Revision No. | Date Issued | Page No. | Description | Reviewed By |
|-------|--------------|------------------|----------|--|--------------|
| | 0 | November 8, 2016 | All | QP Issued | QMS |
| | 1 | October 20, 2021 | All | Updated for QMS Relaunch October 12, 2021 | Thomas Darby |

Approval Signatures

| | | |
|-------------------|--|---------------|
| Prepared by: | Thomas Darby | 10/20/2021 |
| | _____ Name (Preparer) | _____ Date |
| Quality Reviewer: |  | 10/20/2021 |
| | _____ Thomas Darby (Quality Reviewer) | _____ Date |
| QMS Approver: |  | 10/20/2021 |
| | _____ David Gerber (QMS Approver) | _____ Date |

STATEMENT OF POLICY:

The Arcadis Environment Business Line (ENV) uses measuring and test equipment in the course of its activities. Equipment used by ENV and their subcontractors must be in the condition required for the performance of specified activities. A procedure for performing and documenting calibration and for the preventive maintenance of measuring and test equipment will be followed to provide necessary controls.

1 Purpose

The objective of this Quality Procedure (QP) is to provide a standard procedure for the calibration and control of measuring and test equipment, including establishing the correct equipment type, range, accuracy, and precision to meet data collection needs. Equipment must be uniquely identified, calibrated against recognized standards that are clearly identified and documented, and maintained to provide reliable performance and to meet ENV quality requirements.

2 Responsibilities

Certified Project Manager – responsible for implementation of this procedure.

Field Supervisor – is responsible for field equipment and for communicating calibration and maintenance procedures for equipment used by ENV staff. Similar requirements for field equipment calibration and maintenance should also be communicated to subcontractors using field equipment. Subcontractors are responsible for following those requirements and are subject to performance audits.

Quality Consultant – is responsible for providing quality assurance and quality control guidance to the CPM in implementing this procedure.

Quality Reviewer – is responsible for final review of this Quality Procedure (QP). Quality Reviewers may be a Quality Consultant, QMS Document Owner, Technical Solution Leader, Community of Practice Leader, or other qualified subject matter expert (SME).

Project Team Members – project team members are responsible for verifying calibration status prior to using the equipment, and for operating equipment by approved procedures, documenting information, and reporting equipment malfunctions.

3 Terms and Conditions

Accuracy – a qualitative evaluation of the agreement between an individual value (or the central tendency of a set of values) and the correct value or the accepted reference value.

Calibration – the process of evaluating and standardizing an instrument by determining the deviation from a known standard.

Measuring and Test Equipment – devices or systems used to calibrate, measure, gauge, test, or inspect in order to acquire data.

Precision – a qualitative evaluation of measurement data used to describe the dispersion of a set of numbers with respect to its central tendency.

4 Related Documents

Not Applicable.

5 Description of the Procedure

Measuring and test equipment will be controlled by a calibration and preventive maintenance program. Instruments that measure a quantity or whose performance must meet stated criteria will be subject to calibration. Calibration of equipment may be performed internally using reference equipment and standards, or externally by agencies or manufacturers. Two types of calibration are presented in this procedure:

- Operational calibration, which is routinely performed as part of instrument usage.
- Periodic calibration, which is performed at prescribed intervals for equipment such as water-level indicators, pressure recording devices, and thermometers. In general, equipment that can be calibrated periodically is relatively stable in performance.

Preventive maintenance is an organized and documented program of equipment cleaning, lubricating, reconditioning, adjusting, and/or testing intended to maintain proper performance, prevent equipment from failing during use, and maintain reliability.

1. Calibration Procedures

Documented procedures must be used for calibrating measuring and test equipment and reference equipment. Procedures such as those published by ASTM International (formerly known as the American Society for Testing and Materials), U.S. Environmental Protection Agency (USEPA), or procedures provided by manufacturers will be used whenever possible.

Where pre-established procedures are not available, procedures will be developed. Factors such as the type of equipment, stability characteristics of the equipment, required accuracy and precision, and the effect of error on the quantities measured must be considered. Calibration procedures must include:

- Type of equipment to be calibrated
- Reference equipment and standards to be used
- Calibration method and specific procedure
- Acceptance tolerances
- Frequency of calibration
- Data recording form.

2. Equipment Identification

Measuring and test equipment owned by Arcadis must be uniquely identified using the manufacturer's serial number, a calibration system identification number, or an inventory control tag number. This identification must be attached to the equipment. In addition to the identification number, equipment requiring periodic calibration must bear a label indicating when the next calibration is due. Equipment that is rented or leased for the purposes of measuring and testing must also be uniquely identified.

Personnel are responsible for verifying calibration status from due date labels or instrument records prior to using the equipment. Measuring and test equipment that is not properly calibrated must not be used.

3. Calibration Frequency

Measuring and test equipment and reference equipment will be calibrated at prescribed intervals and/or as part of operational use. The calibration frequency will depend on the type of equipment, inherent stability, manufacturer's recommendations, intended use, effect of error on the measurement process, and experience. Calibration frequencies may be defined in project-specific plans or in calibration procedures. The CPM or Field Supervisor is responsible for specifying the procedures to be followed to meet project data quality objectives.

Scheduled periodic calibration may not be performed for infrequently used equipment; such equipment will be calibrated on an "as needed" basis prior to use, and then at the required frequencies for the duration of its use.

Field equipment will require an operational check per the applicable procedure and or the equipment manual prior to use, and then again at the end of the working day. Pre-use calibration should be completed under conditions of anticipated use (e.g., temperature, humidity, and atmospheric pressure) if these parameters may influence results.

4. Reference Equipment and Standards

Whenever possible, equipment must be calibrated using reference equipment (i.e., physical standards) and chemical and radioactive standards having known relationships to nationally recognized standards (e.g., National Institute of Standards and Technology [NIST]) or accepted values of natural physical constants. If national standards or constants do not exist, the basis for the calibration must be documented.

Physical standards may include calibration weights, certified thermometers, standard measurement tapes, gauge blocks, and reference gauges. These are generally used for periodic calibrations. Physical standards must be used only for calibration.

Chemical and radioactive standards may include reagents, solvents, and gases. These may be Standard Reference Materials (SRM) provided by NIST or USEPA, or they may be vendor-certified materials traceable to NIST or USEPA SRMs. Chemical and radioactive standards will primarily be used for operational calibrations.

The date of receipt and expiration date must be clearly labeled on the container of each standard. If calibration standards are transferred to additional containers, these containers must be labeled with the name of the standard, the lot number, and the shelf life. Calibration standards that exceed shelf life must not be used and must be discarded.

If equipment is sent to the manufacturer or calibration laboratory for calibration, adequate documentation must be maintained to establish the calibration method, reference standard source, or traceability to recognized standards.

5. Calibration Failure

Equipment failing calibration or becoming inoperable during use will be removed from service and segregated to prevent inadvertent use or tagged to indicate it is out of service. The equipment must be repaired and properly recalibrated; equipment that cannot be repaired will be replaced.

The results of activities involving equipment that has failed recalibration will be evaluated by the CPM. If the results are adversely affected, the findings of the evaluation will be documented, and appropriate personnel will be notified.

Periodic calibration of measuring and test equipment does not replace the user's responsibility for verifying proper function of equipment. If an equipment malfunction is suspected, the device must be tagged or removed from service, and recalibrated. If it fails recalibration, it must be repaired or replaced.

6. Documentation of Calibration

Records must be maintained for each piece of calibrated measuring and test equipment and each piece of reference equipment. The records must indicate that established calibration procedures have been followed, and that the accuracy of reference chemical and radioactive standards has been verified.

Records for periodically calibrated equipment must include the following minimum information:

- Type and identification number of equipment
- Calibration frequency and acceptance tolerances
- Calibration dates
- Name of individual and organization performing the calibration
- Reference equipment and/or standards used for calibration
- Calibration data
- Certificates or statements of calibration provided by manufacturers and external organizations
- Documentation of calibration acceptance or failure, and of repair of failed equipment.

For equipment requiring calibration, information should be maintained in a project or equipment database regarding the calibration and maintenance history for that equipment. Equipment that does not have a calibration sticker or that has an expired calibration sticker should be tagged inoperable and sent for calibration. The equipment information file should contain periodic calibration files, as well as equipment calibration and maintenance records, calibration data forms, and/or certification of calibration provided by manufacturers or external organizations and notice of equipment calibration failure.

Measuring and test equipment used for field investigations will typically be calibrated as part of operational use. For this equipment, records of the calibrations or checks will be documented as part of the test data (e.g., in the field notebook or on a Field Activity Log). Equipment-specific forms may also be developed. These records should include information similar to that required for periodically calibrated equipment. Documentation related to malfunctioning equipment or equipment that fails calibration should also be included in the individual equipment file.

Calibration files for equipment requiring periodic calibration should be sent with equipment that is transferred to allow a continuously updated record to be maintained. Recalibration of sensitive equipment should be performed following the transfer.

When measuring and test equipment is rented or leased, procurement documents must specify that a current certificate of calibration must accompany the equipment. This certificate must be maintained with the project documentation calibration records.

7. Operational Checks

Certain equipment may require periodic operability tests or checks to verify that operating systems are within the allowed range. These tests are in addition to formal calibration. Like calibrations, these tests will be performed at specified frequencies, or as part of operational use using reference equipment and standards.

If an instrument fails an operability test, and corrective action cannot bring the instrument into tolerance, it must be removed from service and segregated to prevent inadvertent use or tagged to indicate it is out of service.

Such equipment will be repaired and/or recalibrated.

Operability tests will generally be performed in conjunction with data acquisition. Information recorded must include:

- Type and identification number of equipment (e.g., model and serial numbers)
- Test date
- Name of individual and organization performing the test
- Reference equipment and standards used
- Test data
- Documentation of acceptance or failure.

Documentation may be in the field notebook or on a Field Activity Log.

8. Preventive Maintenance

Preventive maintenance is an organized program of equipment cleaning, lubricating, reconditioning, adjusting, and/or testing intended to maintain proper performance, prevent equipment from failing during use, and maintain reliability. Specific maintenance details may be supplied in project-specific plans. A typical preventive maintenance program includes:

- A listing of the equipment that is included in the program
- The frequency of maintenance (manufacturer's recommendations or previous experience with the equipment)
- Service contracts
- Identification of spare parts
- Items to be checked and specific protocols to be followed
- Documentation of maintenance.

Maintenance records of measuring and test equipment must be maintained at the location that is the host for the equipment. Documentation of subcontractor and Arcadis equipment that is used for an individual project will be included in the project files. Records for multi-project equipment will be maintained by the location that controls the equipment.

Measuring and test equipment must be controlled through the use of sign-out/sign-in records or other suitable method. Equipment that is returned from field use must be free of contamination, packaged in a manner suitable for storage, and returned to its designated area. Support personnel should be notified of performance problems with equipment.[Click to enter text]

- END OF PROCEDURE -

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QP 3.08 – Field Sampling, Measurement, and Observations

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Version Control

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| | 0 | November 3, 2016 | All | QP Issued | QMS |
| | 1 | October 11, 2021 | All | Updated for QMS Relaunch October 12, 2021 | Marc Killingstad David Gerber |
| | 2 | December 2, 2022 | All | Content reviewed by Quality Reviewer. No updates needed at this time. | Marc Killingstad |
| | | | All | Updated document Revision No. and Revision Date on all pages. Changed "Arcadis Environmental Business Line (ENV)" to "Resilience Environment (ENV)" First line, Statement of Policy | Rosario Varrella |

Approval Signatures

| | | |
|-------------------|--|------------|
| Prepared by: | Marc Killingstad | 10/11/2021 |
| | _____ | _____ |
| | Name (Preparer) | Date |
| Quality Reviewer: |  | 12/2/2022 |
| | _____ | _____ |
| | Signature (Quality Reviewer) | Date |
| QMS Approver: |  | 12/2/2022 |
| | _____ | _____ |
| | David Gerber) | Date |

STATEMENT OF POLICY:

It is the Resilience Environment (ENV) policy that field sampling, measurements, and observations must be conducted and documented to facilitate later data interpretation, provide an evidentiary record and to demonstrate that field activities have been performed consistently and in accordance with approved site-specific project planning documents. Site-specific documents describing field sampling activities may include, but are not limited to, work plans, the Project Quality Plan (PQP), Quality Assurance Project Plan (QAPP), Field Sampling Plan (FSP), and Technical Guidance Instructions (TGIs), Quality Procedures (QPs), Health and Safety Plan (HASP), and/or other appropriate project documents associated with the sampling program.

1 Purpose

The objective of this Quality Procedure (QP) is to provide a consistent process for the execution of activities associated with field sampling, measurements, and observations. This QP, while focused on field sampling activities, should be performed in conjunction with QP 3.06 Field Activities Documentation.

2 Responsibilities

Certified Project Manager (CPM) – is responsible for implementation of this QP, including verification that site-specific project planning documents are followed (including approved deviation decisions, if necessary). Although a Field Supervisor may lead the sampling activities, the CPM is ultimately responsible for staff's adherence to this QP.

Quality Consultant – responsible for providing quality assurance and quality control (QA/QC) guidance to the CPM in implementing this procedure. Note that for Federal projects there are specific requirements for the QA officer assigned to a project.

Quality Reviewer – Is responsible for final review of this Quality Procedure (QP). Quality Reviewers may be a Quality Consultant, QMS Document Owner, Technical Solution Leader, Community of Practice Leader, or other qualified subject matter expert (SME).

Project Team Members – project participants who are involved in sampling activities are responsible for compliance with this procedure. Individuals involved in the sampling program will read and adhere to the site-specific project planning documents that direct their field activities.

3 Terms and Conditions

Work Plan – a document that describes proposed project activities.

Quality Assurance Project Plan (QAPP) – a document that prescribes the quality assurance/quality control procedures to be followed. Uniform Federal Policy (UFP)-QAPPs are now frequently required for environmental projects by most federal regulatory agencies. UFP-QAPP includes Worksheets used to document the entire project plan developed following the systematic planning process. For more details on the UFP-QAPP see: <http://www.epa.gov/fedfac/documents/qualityassurance.htm>. Note that if the project QAPP is written following the Uniform Federal Policy (UFP) that it will also contain a description of the sampling rationale and sampling locations as well as quality assurance/quality control requirements. The UFP-QAPP format is designed to capture

the entire systematic planning process. If a UFP-QAPP is written for a project, a separate FSP may not be required unless specified by the particular regulatory agency, client or contract.

Field Sampling Plan (FSP) – a document that describes the procedures and protocols necessary to complete field sampling and data collection activities.

Health and Safety Plan (HASP) – a document that describes the hazards of planned activities and the controls to be implemented to protect site personnel.

Data Quality Objective (DQO) – a statement that specifies the quality of data required to support the purposes and intent of the sampling and analysis activity. DQOs are based on the intended use of the data; as such, different data uses and needs may require different levels of data quality.

Technical Guidance Instruction (TGI) –TGIs may also be created or revised on a program or project specific basis. An TGI library is available on the [Environment Quality Management System SharePoint site](#).

4 Related Documents

- QP 3.06 - Field Activities Documentation
- QP 3.07 - Calibration and Control of Measuring and Test Equipment.

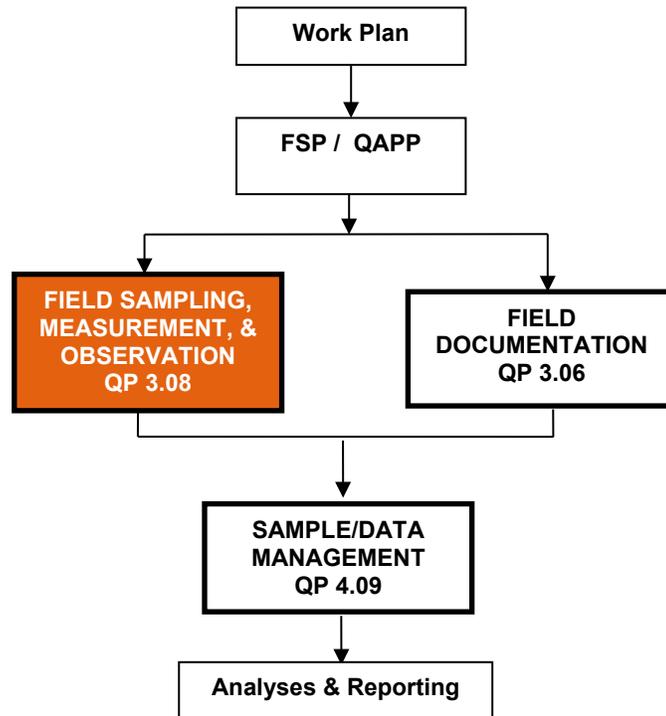
Forms such as purge logs, soil boring logs appropriate for the field sampling activity and observations.

Related documents are available in the [QMS Document Library](#).

5 Description of the Procedure

Sampling and data quality directly affect overall project success because sampling and other field activities are a critical and fundamental component of projects. Errors, mistakes, missed communications, or out-of-scope or out-of-compliance actions may have an adverse effect on a project. Because field conditions cannot be anticipated absolutely, procedures for a particular sampling program must include a formal process for making decisions and obtaining appropriate approvals for deviations necessitated by conditions. Within this context, the basic procedures and requirements for sampling and other field measurements and observations are outlined in the project documents (FSP, QAAP or Work Plan) and the associated procedures. Sample collection, data collection, and drilling locations should be documented (i.e., tape measurements with respect to site features, GPS measurements, and or survey by a licensed land surveyor). It is also recommended taking to the field site maps showing pre-existing and proposed sampling, drilling, data collection, and well locations. It is also recommended the field staff be aware of previous data measurements so field staff can note and communicate any significant changes.

The following flow chart provides the major components of a typical field characterization program and highlights where field sampling activities fit in.



1. Field Sampling, Measurement, and Observation Activities

Field operations are conducted to provide reliable information, data, and/or samples that meet the project and data quality objectives. It is essential that field sampling, measurement, and observation activities begin with the field team having a detailed familiarity with appropriate site-specific project planning documents, most notably the FSP. Time should be scheduled and budgeted for the field team members to review the plan(s) and ask questions. With a good understanding of what samples or data are to be collected, as well as where and why they are to be collected, field personnel will perform the following activities during the implementation of field characterization activities.

1.1 Briefing and Preparation

Before field activity begins, a kick-off meeting should be held to ensure that the project team understands the project objectives and the procedures that will be followed. The CPM and appropriate project office (including relevant technical lead(s)) and field personnel (e.g., Field Supervisor, Crew Leader, or entire crew) should engage in a briefing via telephone discussion or in person to review (in summary fashion) the following:

- Project objectives and project plans
- DQOs
- Sampling locations
- Applicable TGIs for the proposed activities
- Chain of command
- HASP (including site-specific Health and Safety concerns)
- Provisions for addressing deviations
- Communication plan

- Other special circumstances or information critical to the success of the sampling event and integrity of the data and documentation.

When possible, the CPM and/or Field Supervisor should perform a reconnaissance site visit prior to initiation of the sampling or other field activities to review sample locations and consider health and safety or other logistical challenges the site may present. The CPM must also ensure that any utility clearance requirements have been met. Finally, the CPM and/or Field Supervisor must verify that the necessary subcontracts, notifications, and approvals are in place, including coordination with client personnel, agency oversight personnel, access to private or public property (i.e., legal), and coordination with utility companies/agencies regarding the potential of buried, overhead, or other sensitive infrastructure that may affect project implementation and/or health and safety.

1.2 Standard Operating Procedures and Technical Guidance Instructions

Applicable TGIs must be followed to ensure consistency and quality in method and resulting data. A TGI library is available in the Environment Quality Management System SharePoint site. TGIs may also be created or revised on a program or project specific basis. Deviation from established procedure(s) during a data collection activity must be documented. Where plans and TGIs allow discretion (do A or B) or choice (exact sampling location) these decisions should be documented in the field notebook. In cases where the integrity of the data being collected may be jeopardized, field personnel must consider stopping associated work activities until the CPM or other project authority can be consulted as to what corrective action is warranted before work can recommence. Follow the hierarchy of regulatory, client, ENV in selecting and modifying methods and procedures.

1.3 Equipment and Instrumentation

The site-specific project planning documents will be reviewed to identify the types of equipment, instrumentation, and supplies that are needed for the sampling, measurement, observation, or other data collection activities. The selected equipment and instrumentation will meet the requirements of the specifications, methods, and procedures provided in the FSP, QAPP, HASP, or other planning document(s). Further, the Field Supervisor/Crew Leader is responsible for verifying that the equipment and instrumentation are in good working order, clean and, if necessary, properly calibrated and maintained before, during, and after use in the field. (See QP 3.07 Calibration and Control of Measuring and Test Equipment).

1.4 Physical Sample Management

It is extremely important that proper procedures be followed in the sample identification system employed for collected samples, the chain-of-custody procedures, and the manner in which the samples are tracked from collection point, through handling and shipment, and to receipt by the laboratory (including sampling techniques, sample volumes, holding times, preservation, packaging, and shipping procedures). Field personnel are responsible for obtaining the proper number and type of quality control samples, including but not necessarily limited to trip blanks, duplicates, matrix spikes, matrix spike duplicates, and equipment rinse blanks (these requirements should be specified in the site-specific project planning documents and reviewed prior to commencing the field program). These procedures are specified in the Work Plans, FSP, QAPP, and or HASP, or other project planning documents and shall be reviewed by field personnel prior to initiation of field activities. Deviation from established procedures could impact the integrity of the sample or activity; and must be justified, approved by the CPM, and be appropriately documented.

1.5 Qualitative Data Management

Based on requirements specified in site-specific project planning documents, field activities should adhere to applicable TGIs and be carried out in a consistent manner that is well documented in accordance with QP 3.06 Field Activities Documentation. This includes care in making and recording accurate and precise measurements and observations in a timely manner.

1.6 Decontamination and Investigation-Derived Waste

Field personnel will review and be familiar with required decontamination procedures, including those for cleaning field equipment, proper storage of cleaned field equipment, and for properly disposing of waste generated from decontamination procedures. If decontamination is conducted on site, the activities will be performed in a designated, controlled location that will not impact collected samples. Decontamination activities will be appropriately documented in the field notes, following the protocols specified in the FSP/QAPP or TGI and QP 3.06 Field Activities Documentation.

It is important to note that decontamination includes personal protective equipment as well as vehicles and equipment. It is critical that equipment used in one area not serve as a source of contamination of another. This may include weeds or affected soil/water (e.g., carried-in tires or equipment) that could be transported outside the designated work area. Work in surface waters potentially supporting amphibians and other ecological resources may require specific decontamination procedures between sampling events even if no pollutants are anticipated in the waters.

Field personnel will review and be familiar with required procedures for management of investigation-derived waste (IDW) generated as part of the proposed field activities including information/protocols for tracking, storing, labeling, inspecting, sampling, and shipping/disposing of all IDW generated from the proposed field activities. Wastes generated in the field will be collected, stored, and properly disposed in accordance with FSP/QAPP protocols.

1.7 Corrective Action

The CPM and field personnel will be familiar with site-specific project planning procedures designed to address deficiencies or deviations quickly and efficiently, so as not to unnecessarily hold up progress or compromise the integrity of the field effort. Based on the procedures established in the site-specific project planning documents, specific steps are taken as soon as a potential problem is identified. At a minimum, deficiencies or deviations must be reported to the CPM (through pre-established chain of command) and then fully documented to include the nature of the problem, the corrective action taken, and the person(s) responsible for correcting or otherwise addressing the problem. Site-specific project planning documents should contain site-specific corrective action procedures.

- END OF PROCEDURE -

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TGI – Soil Description

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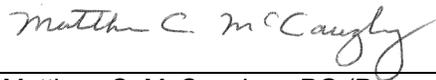
Version Control

| Issue | Revision No. | Date Issued | Page No. | Description | Reviewed By |
|-------|--------------|-------------------|----------|--|---------------------------------|
| | 0 | May 20, 2008 | 17 | Original SOP | Joe Quinnan Joel Hunt |
| | 1 | September 2016 | 15 | Updated to TGI | Nick Welty Patrick Curry |
| | 2 | February 16, 2018 | 15 | Updated descriptions, attachments and references in text | Nick Welty Patrick Curry |
| | 3 | April 15, 2022 | | Minor description edits, intro of grain-size K analysis, revised boring log template | Matt McCaughey Patrick Curry |
| | 4 | June 5, 2023 | | Annual review completed by SME. | Patrick Curry |

Approval Signatures

Prepared by:

6/5/2023



Matthew C. McCaughey, PG (Preparer)

Date

Reviewed by:

6/5/2023



Patrick Curry, PG (Subject Matter Expert)

Date

1 Introduction

This Arcadis Technical Guidance Instruction (TGI) describes proper soil description procedures based on visual inspection and testing of soil cores and samples. This document has been developed to emphasize field observation and documentation of details required to:

- Make hydrostratigraphic interpretations guided by depositional environment/geologic settings
- Provide information needed to understand the distribution of constituents of concern; properly design wells, piezometers, and/or additional field investigations; and develop appropriate remedial strategies.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

This TGI should be followed for unconsolidated material unless there is an established client-required specific procedure or regulatory-required specific procedure. In cases where there is a required specific procedure, it should be followed and should be referenced and/or provided as an appendix to reports that include soil classifications and/or boring logs. When following a required non-Arcadis procedure, additional information required by this TGI should be included in field notes with client approval.

This TGI incorporates elements from various standard systems such as ASTM D2488-06, Unified Soil Classification System, Burmister and Udden Wentworth. However, none of these standard systems focus specifically on contaminant hydrogeology and remedial design. Therefore, although each of these systems contain valuable guidance and information related to correct descriptions, strict application of these systems can omit information critical to our clients and the projects that we perform.

This TGI includes the following attachments:

- **Attachment A** – Field Soil Description Guide
- **Attachment B** – Particle Size System Comparison
- **Attachment C** – Description of Logging Terms
- **Attachment D** – Blank Boring Log
- **Attachment E** – Completed Boring Log

This TGI does not address details of health and safety; drilling method selection; boring log preparation; sample collection; or laboratory analysis. Refer to other Arcadis procedure, guidance, and instructional documents, the project work plans including the quality assurance project plan, sampling plan, and health and safety plan (HASP), as appropriate.

4 Personnel Qualifications

Soil descriptions should only be performed by Arcadis personnel or authorized sub-contractors with a degree in geology or a geology-related discipline. Field personnel will complete training on the Arcadis soil description TGI in the office and/or in the field under the guidance of an experienced field geologist with at least 2 years of prior experience applying the Arcadis soil description method.

5 Equipment List

The following equipment should be taken to the field to facilitate soil descriptions:

- Field book, field forms or digital devices to record soil descriptions
- Field book for supplemental notes
- This TGI for Soil Descriptions and any project-specific procedure, guidance, and/or instructional documents (if required)
- Field card showing Wentworth scale
- Munsell® soil color chart
- Tape measure divided into tenths of a foot
- Stainless steel knife or spatula
- Hand lens
- Water squirt bottle
- 4-ounce glass jars with lids (for collecting soil core samples)
- Personal protective equipment (PPE), as required by the HASP
- Digital camera

- Folding table

6 Cautions

Drilling and drilling-related hazards including subsurface utilities are discussed in other procedure documents and site-specific HASPs and are not discussed herein.

Soil samples may contain hazardous substances that can result in exposure to persons describing soils. Routes for exposure may include dermal contact, inhalation and ingestion. Refer to the project specific HASP for guidance in these situations.

7 Health and Safety Considerations

Field activities associated with soil sampling and description will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities. Know what hazardous substances may be present in the soil and understand their hazards. Always avoid the temptation to touch soils with bare hands, detect odors by placing soils close to your nose, or tasting soils.

8 Procedure

8.1 General Procedures

- Select the appropriate sampling method to obtain representative samples in accordance with the selected sub-surface exploration method, e.g., split-spoon or Shelby sample for hollow-stem drilling, acetate sleeves for direct push, bagged core for sonic drilling, etc.
- Proceed with field activities in required sequence. Although completion of soil descriptions is often not the first activity after opening sampler, identification of stratigraphic changes is often necessary to select appropriate intervals for field screening and/or selection of laboratory samples.
- Set up boring log field sheet.
 - Determine the proper units of measure. Drillers in both the US and Canada generally work in feet due to equipment specifications. Field geologists typically record drilling depths, core recovery, and sample intervals in feet and grain size in millimeters
 - Use the Arcadis standard boring log form (**Attachment D**). *Note that as of April 2022, several digital logging applications are available through the FieldNow™ program and the Fulcrum app. A future revision of this TGI, likely in early 2023, will emphasize digital logging methods and field boring log forms will no longer be acceptable. FieldNow is discussed further in Section 10.*
 - The boring log template includes a graphic log of the primary soil texture to support quick visual evaluation of grain size. The purpose of the graphic log is to quickly assess relative soil permeability. Note, for poorly sorted soils (e.g., glacial till), the principal component may not correlate to permeability of the sample. In this case, the geologist should use best judgement to graph overall soil type consistent with relative soil permeability. For example, for a dense sand/silt/clay till, the graphic log would reflect the silt/clay, rather than sand.

- Record depths along the left-hand side at a standard scale to aid in the use of this tool.
- Examine each soil core (this is different than examining each sample selected for laboratory analysis) and record the soil conditions in accordance with guidelines provided in Section 8.2.
- At the end of the boring, record the amount of drilling fluid used (if applicable) and the total depth logged.
- At a minimum, a written or digital boring log should be prepared with the following information:
 - Describe type of surface material (asphalt, grass, topsoil, gravel, etc.)
 - Describe the type of fill or non-native soils and estimated depth to native soils
 - Record sample intervals (soil cores, environmental and/or geotechnical samples)
 - Describe soil conditions in accordance with this TGI
 - Record moisture content and estimated depth to water table or saturated zone
 - Record the total depth and document why drilling was stopped (refusal, target depth achieved, etc.)

8.2 Soil Description Procedures

The standard soil description order is presented below.

- Depth
- PRIMARY TEXTURE
- Principal and Minor Components with Descriptors
 - % Modifiers and grain size fraction
 - Angularity for very coarse sand and larger particles
 - Consistency or Density
 - Plasticity for silt and clay
 - Dilatancy for silt and silt-sand mixtures
- Sorting
- Moisture Content
- Color
- Notes

Depth. To measure and record the depth below ground surface (bgs) of top and bottom of each stratum, the following information should be recorded.

- Measured depth to the top and bottom of sampled interval. Use starting depth of sample based upon measured tool length information and the length of sample interval.
- Length of sample recovered, not including slough (material that has fallen into hole from previous interval), expressed as fraction with length of recovered sample as numerator over length of sampled interval as denominator (e.g., 36/60 for 36 inches recovered from 5-ft [60-inch] sampling interval).
- Thickness of each stratum measured sequentially from the top of recovery to the bottom of recovery.
- Any observations of sample condition or drilling activity that would help identify whether there was loss from the top of the sampling interval, loss from the bottom of the sampling interval, or compression of the sampling interval. Examples: 14/24, gravel in nose of spoon; or 36/60 bottom 12 inches of core empty.

Determination of Components. Obtain a representative sample of soil from a single stratum. If multiple strata are present in a single sample interval, each stratum should be described separately. More specifically, if the sample is from a 2-foot-long split-spoon where strata of coarse sand, fine sand and clay are present, then the resultant description should be of the three individual strata unless a combined description can clearly describe the interbedded nature of the three strata. Example: SAND, fine; with interbedded lenses of Silt and Clay, ranging between 1 and 3 inches thick.

Identify principal component and express volume estimates for minor components on logs using the following standard modifiers.

| Modifier | Percent of Total Sample (by volume) |
|----------|-------------------------------------|
| and | 36 – 50 |
| some | 21 - 35 |
| little | 10 - 20 |
| trace | <10 |

Determination of components is based on using the Udden-Wentworth particle size classification (see below) and measurement of the average grain size diameter. Each size class differs from the next larger class by a constant ratio of ½. Due to visual limitations, the finer classifications of Wentworth’s scale cannot be distinguished in the field and the subgroups are not included. Visual determinations in the field should be made carefully by comparing the sample to the Soil Description Field Guide (**Attachment A**) that shows Udden-Wentworth scale or by measuring with a ruler.

The following table summarized the modified Udden-Wentworth Scale for grain size classification. Note that gravel is a size category encompassing the granule, pebble, cobble, and boulder size classes.

| Udden-Wentworth Scale (Modified by Arcadis, 2008) | | | | |
|---|-------------------|-------------|-------------|------------------|
| Size Category | Size Class | Millimeters | Inches | Standard Sieve # |
| Gravel (Cobble) | Boulder | 256 – 4096 | 10.08+ | |
| | Large cobble | 128 - 256 | 5.04 -10.08 | |
| | Small cobble | 64 - 128 | 2.52 – 5.04 | |
| Gravel (Pebble) | Very large pebble | 32 – 64 | 0.16 - 2.52 | |
| | Large pebble | 16 – 32 | 0.63 – 1.26 | |
| | Medium pebble | 8 – 16 | 0.31 – 0.63 | |
| | Small pebble | 4 – 8 | 0.16 – 0.31 | No. 5 + |
| | Granule | 2 – 4 | 0.08 – 0.16 | No.5 – No.10 |

| | | | | |
|-------|-------------------------------|----------------|------------------|---|
| Sand | Very coarse sand | 1 -2 | 0.04 – 0.08 | No.10 – No.18 |
| | Coarse sand | ½ - 1 | 0.02 – 0.04 | No.18 - No.35 |
| | Medium sand | ¼ - ½ | 0.01 – 0.02 | No.35 - No.60 |
| | Fine sand | 1/8 - ¼ | 0.005 – 0.1 | No.60 - No.120 |
| | Very fine sand | 1/16 – 1/8 | 0.002 – 0.005 | No. 120 – No. 230 |
| Fines | Silt (subgroups not included) | 1/256 – 1/16 | 0.0002 – 0.002 | Not applicable (analyze by pipette or hydrometer) |
| | Clay (subgroups not included) | 1/2048 – 1/256 | 0.00002 – 0.0002 | |

Identify components as follows. Remove particles greater than very large pebbles (64-mm diameter) from the soil sample. Record the volume estimate of the greater than very large pebbles. Examine the sample fraction of very large pebbles and smaller particles and estimate the volume percentage of the pebbles, granules, sand, silt and clay. Use the jar method, visual method, and/or wash method (Appendix X4 of ASTM D2488) to estimate the volume percentages of each category.

Sieve and hydrometer grain-size analysis can be used to vet the visual description, as well as used to estimate hydraulic conductivity. Lab or field sieve analysis is advisable to characterize the variability and facies trends within each hydrostratigraphic unit. It is recommended that sieve-hydrometer analysis be performed on representative samples from each soil type to estimate the fraction of each grain size category using ASTM D422 Standard Test Method for Particle-Size Analysis of Soils. If desired sieve sizes can be specified to follow the Udden-Wentworth classification (U.S. Standard sieve sizes 6; 12; 20; 40; 70; 140; and 270) to retain pebbles; granules; very coarse sand; coarse sand; medium sand; fine sand; and very fine sand, respectively.

Several empirical formulas provide a reliable means of estimating hydraulic conductivity (K) from grain-size distribution data, provided that the formation does not contain abundant fines that result in cohesive or plastic behavior or include cobble-sized grains (Payne et al. 2008). Grain-size analysis can help bracket the permeability of hydrostratigraphic units (HSUs) and identify order-of-magnitude spatial variations in K. Arcadis has completed modifications to the Excel-based program HydroGeoSieveXL (Devlin 2015) to process sieve data quickly and estimate K. The tool calculates estimated K values from grain-size data using 15 different empirical formulas. A decision matrix then selects which of the formulas is relevant for the soil type and calculates an average K.

Principal Component. The principal component is the size fraction or range of size fractions containing the majority of the volume. Examples: the principal component in a sample that contained 55% small to medium pebbles would be “PEBBLES, small to medium”; or the principal component in a sample that was 20% fine sand, 30% medium sand and 25% coarse sand would be “SAND, fine to coarse” or for a sample that was 40% silt and 45% clay the principal component would be “CLAY and SILT”.

The boring log form (**Appendix D**) includes a graphic log to visually illustrate a relative estimate of soil permeability. To use the graphic log, place an ‘X’ or shade the appropriate column for the primary soil texture. If the soils have a high percentage of a secondary soil texture (i.e., when the ‘and’ modifier is used), it’s acceptable to mark off the appropriate column for the secondary soil texture in this instance. However, care should be used to avoid marking off the columns for other minor soil textures because doing so will make it difficult to determine the relative soil permeability of the poorly sorted soils.

As noted above, for poorly sorted soils such as glacial till, the principal component may not correlate to permeability of the sample. In this case, the geologist should use best judgement to graph overall soil type consistent with relative soil permeability.

Minor Component(s). The minor component(s) are the size fraction(s) containing less than 50% volume. Example: the identified components are estimated to be 60% medium sand to granules, 25% silt and clay; 15% pebbles – there are two identified minor components: silt and clay; and pebbles.

Include a standard modifier to indicate percentage of minor components (see particle size table) and the same descriptors that would be used for a principal component. An example of minor constituents with modifiers include: some silt and clay, low plasticity; little medium to large pebbles, sub-round.

8.2.1 Secondary Descriptors

The following are the descriptors used outside of the principal and minor components. Note that plasticity should be provided as a descriptor for clay and clay mixtures. Dilatancy should be provided for silt and silt mixtures. Angularity should be provided as a descriptor for pebbles and coarse sand.

Angularity. Describe the angularity for very coarse sand and larger particles in accordance with the table below (ASTM D-2488-06). Figures showing examples of angularity are available in ASTM D-2488-06 and the Arcadis Soil Description Field Guide (**Appendix B**).

| Description | Criteria |
|-------------|--|
| Angular | Particles have sharp edges and relatively plane sides with unpolished surfaces |
| Sub-Angular | Particles are like angular description but have rounded edges |
| Sub-Rounded | Particles have nearly plane sides but have well-rounded corners and edges |
| Rounded | Particles have smoothly curved sides and no edges. |

Plasticity. Describe the plasticity for silt and clay based on observations made during the following test method (ASTM D-2488-06).

- As in the dilatancy test (described below), select enough material to mold into a ball about ½ inch (12 mm) in diameter. Mold the material, adding water, if necessary, until it has a soft, but not sticky, consistency.
- Shape the test specimen into an elongated pat and roll by hand on a smooth surface or between the palms into a thread about 1/8 inch (3 mm) in diameter. If the sample is too wet to roll easily, it should be spread into a thin layer and allowed to lose some water by evaporation. Fold the sample threads and reroll repeatedly until the thread crumbles at a diameter of about 1/8 inch. The thread will crumble when the soil is near the plastic limit.

| Description | Criteria |
|-------------|--|
| Non-plastic | A 1/8-inch (3 mm) thread cannot be rolled at any water content. |
| Low | The thread can barely be rolled, and the lump cannot be formed when drier than the plastic limit. |
| Medium | The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be rerolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit. |
| High | It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit. |

Dilatancy. Describe the dilatancy for silt and silt-sand mixtures using the following field test method (ASTM D-2488-06).

- From the specimen, select enough material to mold into a ball about ½ inch (12 mm) in diameter. Mold the material adding water, if necessary, until it has a soft, but not sticky, consistency.
- Smooth the ball in the palm of one hand with a small spatula.
- Shake horizontally, striking the side of the hand vigorously with the other hand several times.
- Note the reaction of water appearing on the surface of the soil.
- Squeeze the sample by closing the hand or pinching the soil between the fingers, and note the reaction as none, slow, or rapid in accordance with the table below. The reaction is the speed with which water appears while shaking and disappears while squeezing.

| Description | Criteria |
|-------------|---|
| None | No visible change in the specimen |
| Slow | Water appears slowly on the surface of the specimen during shaking and does not disappear or disappears slowly upon squeezing |
| Rapid | Water appears quickly on the surface of the specimen during shaking and disappears quickly upon squeezing |

Note that silt and silt-sand mixtures will be non-plastic and display dilatancy. Clay mixtures will have some degree of plasticity but do not typically react to dilatancy testing. Therefore, the tests outlined above can be used to differentiate between silt-dominated and clay-dominated soils.

Sorting. Sorting is the opposite of grading, which is a commonly used term in the USCS or ASTM methods to describe the uniformity of the particle size distribution in a sample. Well-sorted samples are poorly graded and poorly sorted samples are well graded. Arcadis prefers the use of sorting for particle size distributions and grading to describe particle size distribution trends in the vertical profile of a sample or hydrostratigraphic unit because of

the relationship between sorting and the energy of the depositional process. For soils with sand-sized or larger particles, sorting should be determined as follows:

| Description | Criteria |
|---------------|--|
| Well Sorted | the range of particle sizes is limited (e.g., the sample is comprised of predominantly one or two grain sizes) |
| Poorly Sorted | A wide range of particle sizes are present |

You can also use sieve analysis to estimate sorting from a sedimentological perspective; sorting is the statistical equivalent of standard deviation. Smaller standard deviations correspond to higher degree of sorting (see Remediation Hydraulics, 2008).

Consistency or Density. This can be determined by standard penetration test (SPT) blow counts (ASTM D-1586) obtained when using hollow-stem auger drilling methods and a split spoon sampling device. Otherwise, some field tests are available as outlined below. When drilling with hollow-stem augers and split-spoon sampling, the SPT blow counts and N-value is used to estimate density. The N-value is the blows per foot for the 6” to 18” interval. For example, for a 24-inch split spoon soil core, the recorded blows per 6-inch interval are: 4/6/9/22. Since the second interval is 6” to 12”, the third interval is 12” to 18”, the N value is 6+9, or 15. Fifty blow counts for less than 6 inches is considered refusal. In recent years, more common drilling methods include rotary-sonic or direct push. When blow counts are not available, density is determined using a thumb test. Note however, the thumb test only applies to fine-grained soils.

Fine-grained soil – Consistency

| Description | Criteria | Blow Counts (6-12 to 12-18-inch split spoon interval) |
|--------------|---|---|
| Very soft | Easily penetrated several inches by thumb | N-value < 2 |
| Soft | Easily penetrated one inch by thumb | N-value 2-4 |
| Medium Stiff | Indented about ½ inch with much effort | N-value 5-8 |
| Stiff | Indented with ¼ inch with great effort | N-value 9-15 |
| Very Stiff | Readily indented by thumbnail | N-value 16-30 |
| Hard | Indented by thumbnail with difficulty | N-value > than 30 |

Coarse-grained soil – Density

| Description | Criteria | Blow Counts (6-12 to 12-18-inch split spoon interval) |
|--------------|--|---|
| Very loose | Density classification of coarse-grained soils is only required when blow counts from standard penetration tests are performed during hollow-stem auger drilling | N-value 1- 4 |
| Loose | | N-value 5-10 |
| Medium dense | | N-value 11-30 |
| Dense | | N-value 31- 50 |
| Very dense | | N-value >50 |

Moisture Content. Moisture content should be described for each soil sample in accordance with the table below (percentages should not be used unless determined in the laboratory). *Note that some drilling methods (e.g., sonic) can compress and dry out the sample during drilling. Therefore, it can be difficult to determine if a sample is saturated, or merely moist. In this case, care should be taken to try and determine a static water level within the borehole by measuring depth to water through the drill casing, if possible.*

| Description | Criteria |
|-------------|--|
| Dry | Absence of moisture, dry to touch, dusty |
| Moist | Damp but no visible water |
| Wet | Visibly free water |

Color. Color should be described using simple basic terminology and modifiers based on the Munsell system. Munsell alpha-numeric codes are required for all samples. If the sample contains layers or patches of varying colors this should be noted, and all representative colors should be described. The colors should be described for moist samples. If the sample is dry, it should be wetted prior to comparing the sample to the Munsell chart.

Notes. Additional comments should be made where observed and should be presented as notes with reference to a specific depth interval(s) to which they apply. Some of the significant information that may be observed includes the following.

- Odor - You should not make an effort to smell samples by placing near your nose since this can result in unnecessary exposure to hazardous materials. However, odors should be noted if they are detected during the normal sampling procedures. Odors should be based upon descriptors such as those used in NIOSH “Pocket Guide to Chemical Hazards”, e.g., “pungent” or “sweet” and should not indicate specific chemicals such as “phenol-like” odor or “BTEX” odor.
- Structure
- Bedding planes (laminated, banded, geologic contacts).
- Presence of roots, root holes, organic material, man-made materials, minerals, etc.
- Mineralogy

- Cementation
- NAPL presence/characteristics, including sheen (based on client-specific guidance).
- Reaction with HCl - typically only used for special soil conditions, such as caliche environments.
- Origin, if known (Lacustrine; Fill; etc.).

8.3 Example of Soil Descriptions

The standard generic description order is presented below.

- Depth
- PRIMARY TEXTURE
- Principal and Minor Components with Descriptors
 - % Modifiers and grain size fraction
 - Angularity for very coarse sand and larger particles
 - Consistency or Density
 - Plasticity for silt and clay
 - Dilatancy for silt and silt-sand mixtures
- Sorting
- Moisture Content
- Color
- Notes



10-15 feet CLAY, trace silt, trace small to very large pebbles, subround to subangular up to 2" diameter; medium to high plasticity, stiff, moist, dark grayish brown (10YR 4/2). NOTE: Lacustrine; laminated 0.1 to 0.2" thick, laminations brownish yellow (10YR 4/3).



10 -15 feet SAND, medium to very coarse, little granules to medium pebbles, subround to subangular, trace silt; poorly sorted, wet, grayish brown (10YR5/2).

Unlike the first example where a density of cohesive soils could be estimated, this rotary-sonic sand and pebble sample was disturbed during drilling (due to vibrations in a loose sand and pebble matrix) so no density description could be provided. Neither sample had noticeable odor so odor comments were not included.

9 Waste Management

Project-specific requirements should be identified and followed. The following procedures, or similar waste management procedures are generally required.

Water generated during cleaning procedures will be collected and contained onsite in appropriate containers for future analysis and appropriate disposal. PPE (such as gloves, disposable clothing, and other disposable equipment) resulting from personnel cleaning procedures and soil sampling/handling activities will be placed in plastic bags. These bags will be transferred into appropriately labeled 55-gallon drums or a covered roll-off box for appropriate disposal.

Soil materials will be placed in sealed 55-gallon steel drums or covered roll-off boxes and stored in a secured area. Once full, the material will be analyzed to determine the appropriate disposal method.

10 Data Recording and Management

10.1 Digital Data Collection Process Overview

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

10.2 Digital Data Collection Tools for Soil Descriptions

Arcadis is transitioning from the use of paper forms to a digital soil description logging process using web-based FieldNow applications accessible on field tablets and smart phones. Company-wide roll out of a FieldNow application for soil descriptions is targeted by the end of 2022.

Paper forms are included in Revision 3 (April 2022) of this Soil Description TGI. Specifically, a blank boring log and completed boring log are provided in **Attachment D** and **Attachment E**. Additional guidance and examples of the digital data collection tools for soil descriptions will be provided in the next revision to this TGI.

10.3 Additional Guidance

The general logging scheme for soil descriptions is described in this document. Depending on project data quality objectives, specific soil description parameters that are not applicable to project goals may be omitted at the project manager's discretion. In any case, use of consistent procedures is required.

Completed logs and/or logbook will be maintained in the task/project field records file. Digital photographs of typical soil types observed at the site and any unusual features should be obtained whenever possible. Photographs should include a ruler or common object for scale. Photo location, depth and orientation must be recorded in the daily log or logbook and a label showing this information in the photo is useful.

For projects involving soil logging and soil sampling, the soil sample should be recorded on the Arcadis boring log form and the field logbook based on Data Quality Objectives for the task/project.

11 Quality Assurance

Soil descriptions should be completed only by appropriately trained personnel. Descriptions should be reviewed by an experienced field geologist for content, format and consistency. Edited boring logs should be reviewed by the original author to assure that content has not changed.

12 References

- ASTM D-1586, Test Method for Penetration Test and Split-Barrel Sampling of Soils.
- ASTM D-2488-00, Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)
- ASTM D422, 63rd Edition, 1972 - Standard Test Method for Particle-Size Analysis of Soils.
- Devlin, J.F. 2015. HydroGeoSieve XL: an Excel-based tool to estimate hydraulic conductivity from grain-size analysis. Hydrogeology Journal, DOI 10.1007/s10040-015-1255-0.
- Folk, Robert L. 1980. Petrology of Sedimentary Rocks, p. 1-48.
- Payne, F. C., Quinnan, J. A., & Potter, S. T. 2008. Remediation Hydraulics. Boca Raton: FL: CRC Press.
- United States Bureau of Reclamation. Engineering Geology Field Manual. United States Department of Interior, Bureau of Reclamation. <http://www.usbr.gov/pmts/geology/fieldmap.htm>.
- Munsell® Color Chart – available from Forestry Suppliers, Inc.- Item 77341 “Munsell® Color Soil Color Charts. Field Gauge Card that Shows Udden-Wentworth scale – available from Forestry Suppliers, Inc. – Item 77332 “Sand Grain Sizing Folder.”
- NIOSH Pocket Guide to Chemical Hazards.

Attachment A

Soil Field Reference Guide

The purpose of this attachment is to present a field reference guide for use during soil logging. Field staff are encouraged to bring a laminated copy of this reference guide into the job site.



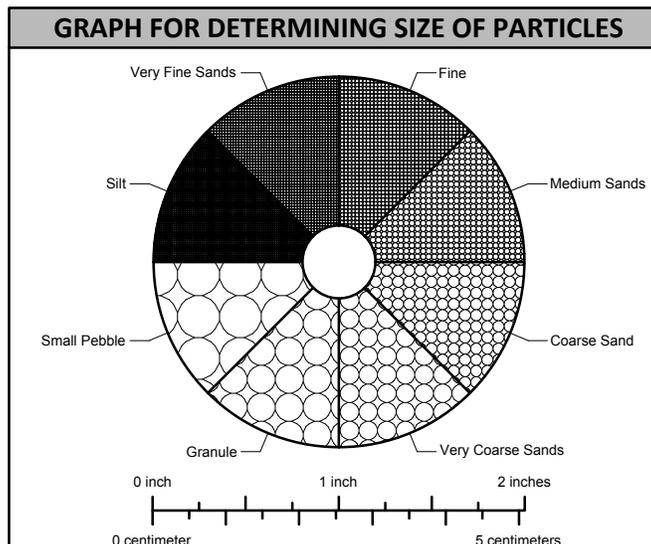
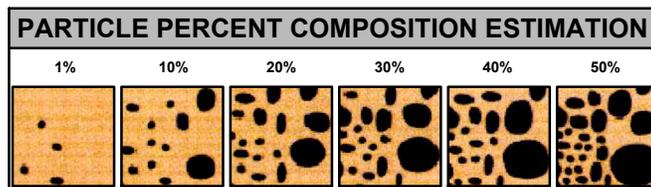
| FINE-GRAINED SOILS | |
|-----------------------------------|--|
| Description | Criteria |
| Descriptor - Plasticity | |
| Nonplastic | A 1/8-inch (3 mm) thread cannot be rolled at any water content. |
| Low | The thread can barely be rolled, and the lump cannot be formed when drier than the plastic limit. |
| Medium | The thread is easy to roll and not much time is required to reach the plastic limit. The thread cannot be re-rolled after reaching the plastic limit. The lump crumbles when drier than the plastic limit. |
| High | It takes considerable time rolling and kneading to reach the plastic limit. The thread can be rolled several times after reaching the plastic limit. The lump can be formed without crumbling when drier than the plastic limit. |
| Descriptor - Dilatancy | |
| No Dilatancy | No visible change when shaken or squeezed. |
| Slow | Water appears slowly on the surface of soil during shaking and does not disappear or disappears slowly when squeezed. |
| Rapid | Water appears quickly on surface of soil during shaking and disappears quickly when squeezed. |
| Minor Components with Descriptors | |
| Moisture | |
| Dry | Absence of moisture, dry to touch, dusty. |
| Moist | Damp but no visible water. |
| Wet | Visible free water; soil is usually below the water table. (Saturated) |
| Consistency | |
| Very soft | N-value < 2 or easily penetrated several inches by thumb. |
| Soft | N-value 2-4 or easily penetrated 1 inch by thumb. |
| Medium stiff | N-value 5-8 or indented about 1/2 inch by thumb with great effort. |
| Stiff | N-value 9-15 or indented about 1/4 inch by thumb with great effort. |
| Very stiff | N-value 16-30 or readily indented by thumb nail. |
| Hard | N-value > than 30 or indented by thumbnail with difficulty. |
| Color using Munsell | |
| Geologic Origin (if known) | |
| Other | |

| DESCRIPTION ORDER |
|---|
| <p>Depth Interval PRIMARY TEXTURE (e.g., SAND) Principal and Minor Components with Descriptors:</p> <ul style="list-style-type: none"> • % Modifiers and grain size fraction • Angularity coarse sand and larger • Consistency or Density • Plasticity for silt and clay • Dilatancy for silt and silt-sand <p>Sorting for granular sediments Moisture Content Color Other NOTES</p> |

| MINOR COMPONENTS % MODIFIERS | |
|------------------------------|-------------------------------------|
| Modifier | Percent of Total Sample (by volume) |
| and | 36 - 50 |
| some | 21 - 35 |
| little | 10 - 20 |
| trace | <10 |

| FOR COARSE-GRAINED SOILS | |
|--|--|
| Description | Criteria |
| Descriptor - Angularity | |
| Angular | Particles have sharp edges and relatively planar sides with unpolished surfaces. |
| Subangular | Particles are similar to angular but have rounded edges. |
| Subround | Particles have nearly planar sides but have well-rounded corners and edges. |
| Round | Particles have smoothly curved sides and no edges. |
| Minor Components with Descriptors | |
| Sorting Cu= d60/d10 | |
| Well Sorted | Near uniform grain-size distribution Cu= 1 to 3. |
| Poorly Sorted | Wide range of grain size Cu= 4 to 6. |
| Moisture | |
| Dry | Absence of moisture, dry to touch, dusty. |
| Moist | Damp but no visible water. |
| Wet | Visible free water; soil is usually below the water table. (Saturated) |
| Density | |
| Very loose | N-value 1 - 4 |
| Loose | N-value 5 - 10 |
| Medium Dense | N-value 11 - 30 |
| Dense | N-value 31 - 50 |
| Very dense | N-value >50 |
| Color using Munsell | |
| Geologic Origin (if known) | |
| Other | |
| Cementation | |
| Weak Cementation | Crumbles or breaks with handling or little finger pressure. |
| Moderate Cementation | Crumbles or breaks with considerable finger pressure. |
| Strong Cementation | Will not crumble with finger pressure. |
| Reaction with Dilute HCl Solution (10%) | |
| No Reaction | No visible reaction. |
| Weak Reaction | Some reaction, with bubbles forming slowly. |
| Strong Reaction | Violent reaction, with bubbles forming immediately. |

| UDDEN-WENTWORTH SCALE | | | |
|---|----------------|-------------------|----------------------------------|
| Fraction | Sieve Size | Grain Size | Approximate Scale |
| Boulder | | 256 - 4096 mm | Larger than volleyball |
| Large Cobble | | 128 - 256 mm | Softball to volleyball |
| Small Cobble | | 64 - 128 mm | Pool ball to softball |
| Very Large Pebble | | 32 - 64 mm | Pinball to pool ball |
| Large Pebble | | 16 - 32 mm | Dime size to pinball |
| Medium Pebble | | 8 - 16 mm | Pencil eraser to dime size |
| Small Pebble | No. 5+ | 4 - 8 mm | Pea size to pencil eraser |
| Granule | No. 10 - 5 | 2 - 4 mm | Rock salt to pea size |
| Very Coarse Sand | No. 18 - 10 | 1 - 2 mm | See field gauge card |
| Coarse Sand | No. 35 - 18 | 0.5 - 1 mm | See field gauge card |
| Medium Sand | No. 60 - 35 | 0.25 - 0.5 mm | See field gauge card |
| Fine Sand | No. 120 - 60 | 0.125 - 0.25 mm | See field gauge card |
| Very Fine Sand | No. 230 - 120 | 0.0625 - 0.125 mm | See field gauge card |
| Silt and Clay. See SOP for description of fines | Not Applicable | <0.0625 mm | Analyze by pipette or hydrometer |



EXAMPLE OF SOIL DESCRIPTION AND PHOTO

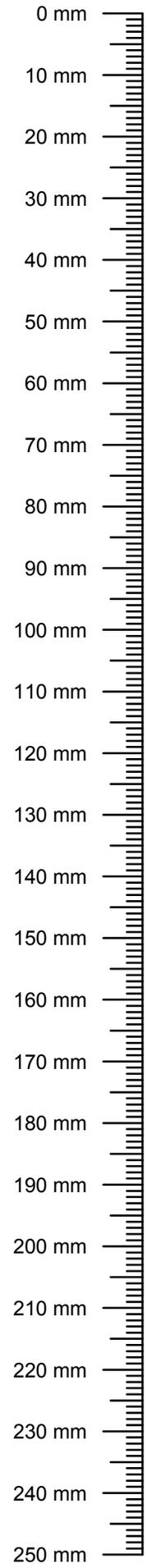
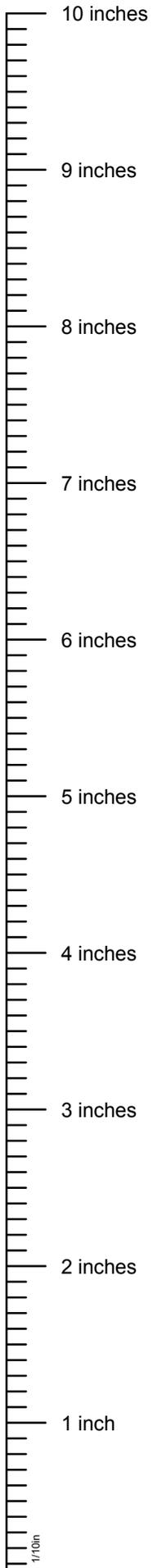
10-15 feet CLAY, trace silt, trace small to very large pebbles, subround to subangular up to 2" diameter; medium to high plasticity, stiff, moist, dark grayish brown (10YR 4/2). NOTE: Lacustrine; laminated 0.1 to 0.2" thick, laminations brownish yellow (10YR 4/3).

16M 02-04
15 ← 10

EXAMPLE OF SOIL DESCRIPTION AND PHOTO

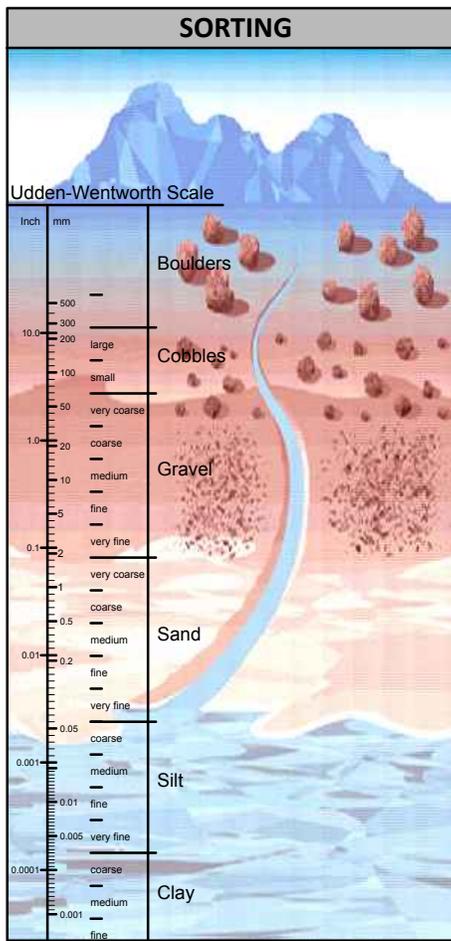
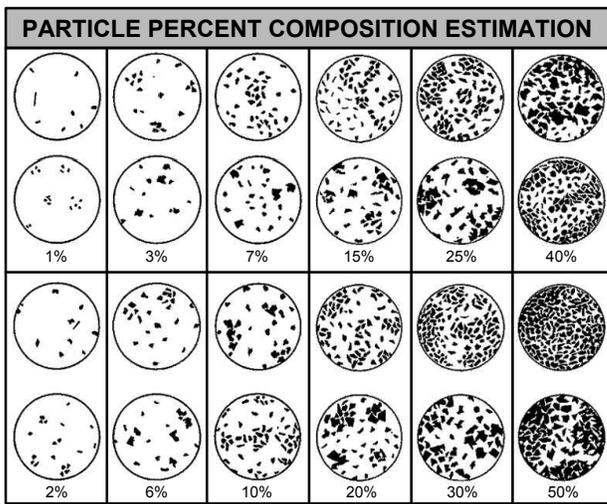
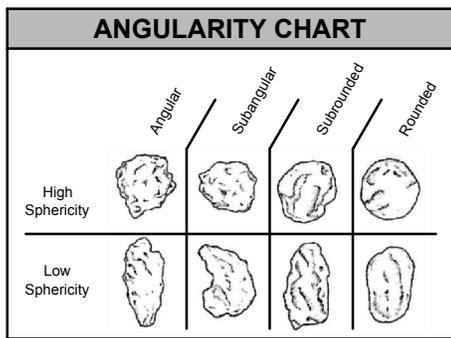
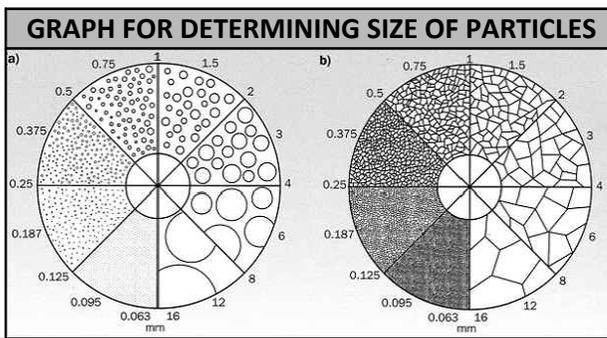
10 - 15 feet SAND, medium to very coarse, little granules to medium pebbles, subround to subangular, trace silt; poorly sorted, wet, grayish brown (10YR 5/2).

16M 01-04
15 ← 10



| VARIATIONS IN SOIL STRATIGRAPHY | |
|---------------------------------|--|
| Term | Thickness of Configuration |
| Parting | 0 - to 1/16-inch thickness. |
| Seam | 1/16 - to 1/2-inch thickness. |
| Layer | 1/2 - to 12-inch thickness. |
| Stratum | > 12-inch thickness. |
| Pocket | Small erratic deposit, usually less than 1 foot in size. |
| Varved Clay | Alternating seams or layers of sand, silt, and clay (laminated). |
| Occasional | ≤ 1 foot thick. |
| Frequent | > 1 foot thick. |

| SOIL STRUCTURE DESCRIPTIONS | |
|-----------------------------|--|
| Term | Description |
| Homogeneous | Same color and appearance throughout. |
| Laminated | Alternating layers < 1/4 inch thick. |
| Stratified | Alternating layers ≥ 1/4 inch thick. |
| Lensed | Inclusions of small pockets of different materials, such as lenses of sand scattered through a mass of clay; note thickness. |
| Blocky | Cohesive soil can be broken down into small angular lumps, which resist further breakdown. |
| Fissured | Breaks along definite planes of fracture with little resistance to fracturing. |
| Slickensided | Fracture planes appear to be polished or glossy, sometimes striated. |



| SETTLING TABLE (SILT/CLAY) | | | | | | | |
|----------------------------|------------|------------|------------|------------|------------|------------|------------|
| Diameter of Particle (mm) | <0.625 | <0.031 | <0.016 | <0.008 | <0.004 | <0.002 | <0.0005 |
| Depth of Withdrawal (cm) | 10 | 10 | 10 | 10 | 5 | 5 | 3 |
| Time of Withdrawal | hr:min:sec |
| Temperature (Celsius) | | | | | | | |
| 20 | 00:00:29 | 00:01:55 | 00:07:40 | 00:30:40 | 00:61:19 | 04:05:00 | 37:21:00 |
| 21 | 00:00:28 | 00:01:52 | 00:07:29 | 00:29:58 | 00:59:50 | 04:00:00 | |
| 22 | 00:00:27 | 00:01:50 | 00:07:18 | 00:29:13 | 00:58:22 | 03:54:00 | |
| 23 | 00:00:27 | 00:01:47 | 00:07:08 | 00:28:34 | 00:57:05 | 03:48:00 | |
| 24 | 00:00:26 | 00:01:45 | 00:06:58 | 00:27:52 | 00:55:41 | 03:43:00 | 33:56:00 |
| 25 | 00:00:25 | 00:01:42 | 00:06:48 | 00:27:14 | 00:54:25 | 03:38:00 | |
| 26 | 00:00:25 | 00:01:40 | 00:06:39 | 00:26:38 | 00:53:12 | 03:33:00 | |
| 27 | 00:00:24 | 00:01:38 | 00:06:31 | 00:26:02 | 00:52:02 | 03:28:00 | |
| 28 | 00:00:24 | 00:01:35 | 00:06:22 | 00:25:28 | 00:50:52 | 03:24:00 | 31:00:00 |
| 29 | 00:00:23 | 00:01:33 | 00:06:13 | 00:24:53 | 00:49:42 | 03:10:00 | |
| 30 | 00:00:23 | 00:01:31 | 00:06:06 | 00:24:22 | 00:48:42 | 03:05:00 | |

Attachment B

Particle Size System Comparison

The purpose of this attachment is to illustrate how the Udden-Wentworth particle sizes and descriptive terms compares to other particle size systems.

When in the field, it is a customary practice to compare current soil descriptions to historical soil boring logs for reference purposes. When reviewing boring logs prepared by others, field staff should first note the particle size system used and recognize these particle size systems may differ. This will avoid confusion when cross referencing between historical and new boring logs and when reviewing existing geologic cross-sections.

For example, a well-sorted sand with grain sizes ranging from 1 to 2 mm should be classified as a very coarse sand by the Udden-Wentworth system. As shown in this attachment, the same particle size would be classified as a medium sand by the United Soil Classification System. The later system has fewer particle size grades and in general, is less descriptive than the Udden-Wentworth system.

PARTICLE SIZE SYSTEM COMPARISON

| System Name | Used By | Grain size distribution in millimeters (mm) | | | | | | | | | | | | | |
|-----------------------------------|--------------------------------------|---|-------|---------|------|--------|--------|-----------|---------|---------|--------|----------|--------|---------|-----|
| Udden-Wentworth | Remediation Geologists and Engineers | | | V. Fine | Fine | Medium | Coarse | V. Coarse | Granule | Pebbles | | | | Cobbles | |
| | | CLAY | SILT | SAND | | | | | Small | Medium | Large | V. Large | Small | Large | |
| | | 0.039 | 0.065 | 0.125 | 0.25 | 0.5 | 1 | 2 | 4 | 8 | 16 | 32 | 64 | 128 | 256 |
| | | | 1/16 | 1/8 | 1/4 | 1/2 | | | | | | | | | |
| United Soil Classification System | Geotechnical Engineers | | | Fine | | | Medium | Coarse | Fine | | Coarse | | | | |
| | | CLAY | SILT | SAND | | | | | GRAVEL | | | | COBBLE | | |
| | | | 0.074 | | 0.42 | | 2 | 4.75 | | 19 | | 75 | | 300 | |
| U.S. Dept. of Agriculture | Soil Scientists | | | V. Fine | Fine | Medium | Coarse | V. Coarse | GRAVEL | | | | | | |
| | | CLAY | SILT | SAND | | | | | GRAVEL | | | | | | |
| | | 0.002 | 0.05 | 0.10 | 0.25 | 0.5 | 1 | 2 | | | | | | | 75 |

Remediation Hydraulics 2008, page 195): The Udden-Wentworth scale is preferred "...because the geometric progression of grain-size diameter also reflects relationships that are important when considering the erosion and deposition of sediments during the depositional process. The correlation between increasing grain size and degree of sorting and permeability is the most important, as permeability structure is responsible for the mobile and immobile porosity within aquifer systems. "

Attachment C

Description of Soil Logging Terms

The purpose of this attachment is to concisely define the soil logging terms used when filling out boring logs. During report preparation, project staff could use this sheet as an index placed in front of the completed boring logs. Also, it can serve as a supplemental reference sheet during field activities.

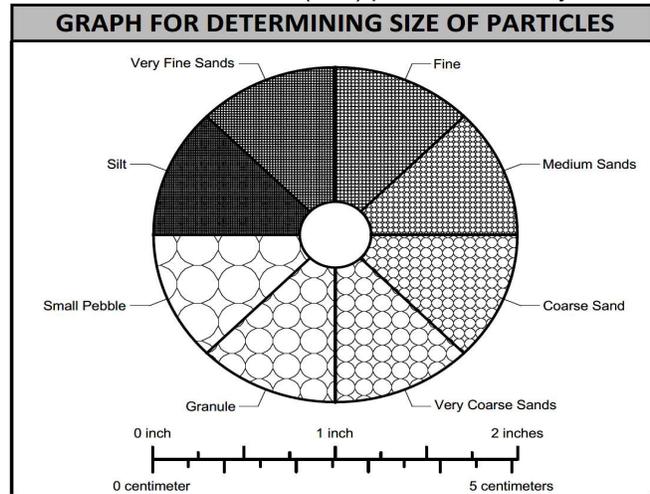
Description of Logging Terms



Note: Soil descriptions based on Arcadis Technical Guidance and Instructions (TGI) procedures. Key terms defined below.

Udden Wentworth Soil Sizes

| | |
|-------------------|------------------|
| Boulder | > 256 mm |
| Large Cobble | 128 to 256 mm |
| Small Cobble | 64 to 128 mm |
| Very Large Pebble | 32 to 64 mm |
| Large Pebble | 16 to 32 mm |
| Medium Pebble | 8 to 16 mm |
| Small Pebble | 4 to 8 mm |
| Granule | 2 to 4 mm |
| Very Coarse Sand | 1 to 2 mm |
| Coarse Sand | 0.5 to 1 mm |
| Medium Sand | 0.25 to 0.5 mm |
| Fine Sand | 0.125 to 0.25 mm |
| Very Fine Sand | 0.062 to 0.12 mm |
| Silt/Clay | <0.065 mm |



Primary Texture (e.g. CLAY, SILT, SAND, GRANULE, PEAT, MUCK, FILL, etc.)

List particle size with the highest percentage per sample interval (e.g. SAND)

Always CAPITALIZE the primary texture

Follow primary texture with a comma followed by grain-size descriptors, etc.

Minor Texture

| | |
|--------|-------------|
| And | (36 to 50%) |
| Some | (21 to 35%) |
| Little | (10 to 20%) |
| Trace | (>10%) |

Angularity

| | |
|-------------|---------------------|
| Angular | Sharp edges |
| Sub-Angular | Rounded edges |
| Sub-Rounded | Well-rounded |
| Rounded | Smooth curved edges |

Sand Density (Blow Counts/ft)

| | |
|--------------|-------|
| Very Loose | 0-4 |
| Loose | 5-10 |
| Medium Dense | 11-30 |
| Dense | 31-50 |
| Very Dense | <50 |

Silt/Clay Consistency (Blow Counts/ft)

| | | |
|--------------|--------|--|
| Very Soft | 0-2, | thumb easily penetrates several inches |
| Soft | 3-4, | thumb easily penetrates one inch |
| Medium Stiff | 5-8, | thumb indents 0.5 in. with much effort |
| Stiff | 9-15, | thumb indents 0.25 in. with great effort |
| Very Stiff | 16-30, | thumbnail is readily intended |

Sorting

| | |
|---------------|-----------------------|
| Well Sorted | 1 to 3 Particle Sizes |
| Poorly Sorted | 4+ Particle Sizes |

Moisture Content

| | |
|-------|--------------------|
| Dry | Dry to touch |
| Moist | No visible water |
| Wet | Visible free water |

Plasticity (for silts and clays)

| | |
|-------------------|---|
| Non-Plastic | 3 mm thread can not be rolled |
| Low Plasticity | 3 mm thread can barely be rolled |
| Medium Plasticity | 3 mm thread can easily and quickly rolled, but not rerolled |
| High Plasticity | 3 mm thread can be rolled slowly, but can be rerolled |

Dilatancy (for silts and silt-sand mixtures)

| | |
|-------|--|
| None | No visible change in the specimen |
| Slow | Water appears slowly during shaking / disappears slowly or not at all upon squeezing |
| Rapid | Water appears quickly during shaking / disappears quickly upon squeezing |

Example Description

10 -15 feet SAND, medium to very coarse, little granules to medium pebbles, subround to subangular, trace silt; poorly sorted, wet, grayish brown (10YR5/2).

Attachment D

Blank Boring Log

The purpose of this attachment is to present a blank field form for use during soil logging. A digital version (Microsoft Excel) of this field form is available from the authors (upon request). If project specific modifications to this boring log template are warranted, please contact the Site Investigation Community of Practice leader for further assistance.

Attachment E

Completed Boring Log

The purpose of this attachment is to provide an example of a completed boring log for reference purposes to field staff. The example provided is for a soil boring completed outside the waste mass of a closed municipal landfill near Baltimore, Maryland. The objective of the drilling program was to determine the depth to groundwater to determine the appropriate depth interval to install a soil gas monitoring well and groundwater monitoring well across the first water-bearing zone. The site geology consists of unconsolidated sediments of the Mid-Atlantic Coastal Plain, specifically the Upper Patapsco formation. These sediments were deposited in a moderate gradient fluvial environment during the Cretaceous period. The landfill was constructed into a regional clay confining unit.

BORING LOG



| | | | | | |
|-------------------------|--------------------------------|------------------------|---------------------------------|--------------------------|------------------------------|
| Boring ID: | <u>MW-08</u> | Project Name: | <u>Acme Landfill</u> | Page: | <u>1 / 1</u> |
| Permit ID: | <u>MD-PG-100</u> | Date Started: | <u>7/18/2018</u> | Ground Elevation: | <u>50.5 ft</u> |
| Site Address: | <u>100 Landfill Road</u> | Date Completed: | <u>7/18/2018</u> | Vertical Datum: | <u>NAVD 88, feet</u> |
| City, State: | <u>Baltimore, Maryland</u> | Total Depth: | <u>35 ft below ground</u> | Northing: | <u>123456.79</u> |
| Drilling Co: | <u>Earth Matters</u> | Depth to Water: | <u>19 ft below ground</u> | Easting: | <u>123456.79</u> |
| Driller: | <u>Rod E. Piper</u> | Hole Diameter: | <u>2-inch</u> | Horizontal Datum: | <u>NAD 83 feet, MD State</u> |
| Drilling Method: | <u>Direct-push/hollow-stem</u> | Core Device: | <u>5-foot macrocore sampler</u> | Prepared by: | <u>Sandy Pebbles</u> |
| Boring Status: | <u>completed as well</u> | Drilling Fluid: | <u>none</u> | Reviewed by: | <u>Clay Brown</u> |

| Drilling Information | | | | Graphical Log for Primary Texture | | | | | | | | Soil Description (Udden-Wentworth System) | Field Notes | | | |
|-------------------------|--------------------|------------------------|-------------------------|-----------------------------------|------|-----------|------|--------|--------|-------------|---------|---|---|--|--|-------------------------|
| Drilling Depth (ft bgs) | Core Interval (ft) | Core Recovery (inches) | VOC Vapor Reading (ppm) | Fines | | Sand | | | | Gravel | | | | Depth Interval (ft), PRIMARY TEXTURE, Principal and Minor Components with Descriptors (% modifiers and grain size fraction, angularity for coarse sand and larger, consistency/density, plasticity for silt and clay, dilatancy for silt/silt-sand); Sorting, Moisture Content, Color. NOTES: <i>Texture Modifiers: Trace (<10%), Little (10 to 20%), Some (21 to 35%), And (36 to 50%)</i> | Driller's Observations, Geologic Formation, Field Screening Results, Sample Interval etc. | |
| | | | | clay | silt | very fine | fine | medium | coarse | very coarse | granule | pebble | cobble | | | boulder |
| 0 to 1 | 0-5 | 43.2/60 | < 1 | | | | | | | | | | | 0-0.5 ft, topsoil with organics | Grass covered area | |
| 1 to 2 | | | < 1 | | | X | | | | | | | | 0.5-5 ft, SAND, fine, trace silt, trace pebble, round; poorly sorted, moist, yellowish brown (7.5 YR 5/8). NOTE: some cementation, does not react with HCl | continuous macro-core logging | |
| 2 to 3 | | | < 1 | | | X | | | | | | | | | | |
| 3 to 4 | | | < 1 | | | X | | | | | | | | | | cemented sand @3.6-4 ft |
| 4 to 5 | | | < 1 | | | X | | | | | | | | | | |
| 5 to 6 | 5-10 | 40.8/60 | < 1 | | | X | X | X | | | | | | 5-10 ft, SAND, fine to coarse, round to subround; well sorted, moist, light to strong brown (7.5 YR 6/4 to 7.5 YR 5/6). | | |
| 6 to 7 | | | < 1 | | | X | X | X | | | | | | | | |
| 7 to 8 | | | < 1 | | | X | X | X | | | | | | | | |
| 8 to 9 | | | < 1 | | | X | X | X | | | | | | | | |
| 9 to 10 | | | < 1 | | | X | X | X | | | | | | | | |
| 10 to 11 | 10-15 | 36/60 | < 1 | | | X | X | X | | | | | | 10-12.5 ft, same as above with trace silt | | |
| 11 to 12 | | | < 1 | | | X | X | X | | | | | | | | |
| 12 to 13 | | | < 1 | | | X | X | X | | | | | | | | |
| 13 to 14 | | | < 1 | | | X | X | X | | | | | | | 12.5 to 15 ft, same as above, color change to pink (7.5 YR 7/3) and reddish yellow (7.5YR 6/8) | |
| 14 to 15 | | | < 1 | | | X | X | X | | | | | | | | |
| 15 to 16 | 15-20 | 55.2/60 | < 1 | | | | X | X | | | | | | 15-18.9 ft, SAND, coarse to very coarse, round to subround; well sorted, moist, strong brown (7.5YR 5/6) to reddish yellow (7.5YR 6/6) | | |
| 16 to 17 | | | < 1 | | | | X | X | | | | | | | | |
| 17 to 18 | | | < 1 | | | X | X | | | | | | | | | |
| 18 to 19 | 20-25 | 36/60 | < 1 | X | X | X | | | | | | | | 18.9-22.7 ft, SAND, very fine to fine, and SILT, coarse to very coarse, poorly sorted, wet, light gray (7.5YR 7/1) | water table encountered @ 18.9 ft | |
| 19 to 20 | | | < 1 | X | X | X | | | | | | | | | | |
| 20 to 21 | | | < 1 | X | X | X | | | | | | | | | | |
| 21 to 22 | | | < 1 | X | X | X | | | | | | | | | | |
| 21 to 23 | | | < 1 | X | X | X | | | | | | | | | | |
| 23 to 24 | 25-30 | 30/60 | < 1 | X | X | | | | | | | | | 22.7-25 ft, CLAY and SILT, high plasticity, soft to stiff at 25 ft, dry to moist, light gray (2/5YR 7/1) w/ red mottling (2.5YR 4/6) | Middle Patapsco Confining Unit | |
| 24 to 25 | | | < 1 | X | X | | | | | | | | | | | |
| 25 to 26 | | | < 1 | X | X | | | | | | | | | | | |
| 26 to 27 | | | < 1 | X | X | | | | | | | | | | | |
| 27 to 28 | | | < 1 | X | X | | | | | | | | | | | |
| 28 to 29 | 30-35 ft | 60/60 | < 1 | X | X | | | | | | | | | 25-31.1 ft, CLAY and SILT, high plasticity, stiff; dry to moist, light gray (2/5YR 7/1) with red mottling (2.5YR 4/6) | | |
| 29 to 30 | | | < 1 | X | X | | | | | | | | | | | |
| 30 to 31 | | | < 1 | X | X | | | | | | | | | | | |
| 31 to 32 | | | < 1 | X | | | | | | | | | | | | |
| 32 to 33 | | | < 1 | X | | | | | | | | | | | | |
| 33 to 34 | < 1 | X | | | | | | | | | | | 31.1-35 ft, SILT, low plasticity, high dilatancy; wet, gray (7.5YR 7/1) | End of direct-push boring @ 35 ft | | |
| 34 to 35 | < 1 | X | | | | | | | | | | | | | | |

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TGI – Monitoring Well Development

Rev: 2

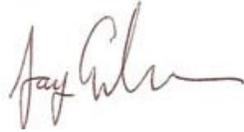
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Version Control

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|-------|--------------|-------------|----------|--|------------------|
| | 0 | 4/24/2017 | All | Re-written as TGI | Marc Killingstad |
| | 1 | 4/12/2022 | All | Updated to new format and some minor content changes | Marc Killingstad |
| | 2 | 4/5/2023 | All | Annual review completed by Marc Killingstad. Updated document revision number and date, version control and signature page. | Marc Killingstad |

Approval Signatures

Prepared by:



4/5/2023

Jay Erickson (Preparer)

Date

Reviewed by:



4/5/2023

Marc Killingstad (Subject Matter Expert)

Date

1 Introduction

This Technical Guidance Instruction (TGI) covers the development of screened wells used for obtaining representative groundwater information and samples from granular aquifers (i.e., monitoring wells).

Note: This TGI only applies to monitoring well development and not remediation (injection/extraction) well development.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

The objectives of monitoring well development are:

1. Repair damage to the borehole wall from drilling that can include clogging, smearing or compaction of aquifer materials;
2. Remove fine-grained sediment from the formation and filter pack that may result in high turbidity levels in groundwater samples;
3. To re-sort formation and filter pack material adjacent to the well screen;

4. To recover any drilling fluids (if used) that may affect the permeability of the formation and filter pack or alter the water quality around the well; and
5. To optimize the well efficiency and hydraulic communication between the well screen and the formation.

Successful monitoring well development is dependent on the following:

1. Hydrostratigraphy – Permeable formations containing primarily sand and gravel are more easily developed due to lower percentages of silt and clay material. Water in permeable formations can be moved in and out of the screen and/or through the formation easier than in less permeable deposits.
2. Well Diameter – Development tooling including brushes, surge blocks, pumps and jetting tools are more readily available for wells 4 inches in diameter and greater.
3. Well Design – Wells with filter packs and screens designed to match the formation through the analysis of formation sieve samples are easier to develop. An important aspect to well design is to minimize the size of the annular space between the formation and well screen. Adequate room must be allowed for the proper installation of well materials, but not too large as to prevent/reduce communication with the surrounding formation.
4. Drilling Methods – Different drilling methods result in varying amount of borehole damage and, therefore, impact the degree to which development will be successful.

Well development methods for monitoring wells include the following:

1. Bailing – Use of a bailer to remove water and sediment from the well casing. This technique does little to remove fines from the filter pack and may lead to bridging of sediment since the flow is in only one direction, toward the well screen. The most effective use of bailing during monitoring well development is in conjunction with other methods (e.g., surging/swabbing) to remove fines accumulated in the monitoring well between cycles of other development methods.
2. Pumping/over pumping – Use of a pump to remove water and sediment from the well casing, over pumping involves pumping the well at a rate that exceeds the design capacity of the well. Similar to bailing, this technique does little to remove fines from the filter pack and may lead to bridging of sediment since the flow is in only one direction, toward the well screen. Small diameter monitoring wells have the additional constraint on pump size and flow rates which further limit the effectiveness of this methodology.
3. Backwashing (rawhiding) – Consists of starting and stopping a pump intermittently to produce rapid pressure changes in a well. This method can produce better results than pumping alone since the procedure involves movement of the water in and out of the screen and formation. However, in many cases the surging action is not rigorous enough to fully develop the well and might be considered the final phase of development after a more rigorous method has been used. Again, small diameter monitoring wells have the additional constraint on pump size and flow rates which further limit the effectiveness of this methodology.
4. Surging/swabbing – Use of a mechanical surge block or swabbing tool to operate like a piston with an up and down motion. The downstroke causes a backwash action that breaks up bridged sediment and the upstroke pulls the dislodged sediment into the well. This method works well for both small and large diameter monitoring wells. Care should be taken on the downstroke so as not to force fines back into the formation, frequent pumping/purging during surging help to keep fines out of the well. Double surge blocks are recommended, and this is typically the most effective method for development of monitoring wells.

5. Jetting – Use of a tool fitted with nozzles that direct streams of water horizontally into well screens at high velocity. Due to the size of the tooling, this method is better suited for wells 4 inches in diameter and larger. The method is also more effective with wire-wrapped/continuous slot screens due to the increased open area. Jetting requires specialized equipment and concurrent pumping to prevent reintroducing fines into the filter pack. Additionally, depending on the configuration of the tool, jetting may require subsequent surging/pumping to remove fines dislodged in the filter pack and formation. Typically, jetting is not a preferred option for new well development but may be effective as part of a re-development/rehabilitation effort.

For most situations, surging/swabbing coupled with bailing or pumping to remove dislodged materials is recommended.

Final well development for properly designed and constructed monitoring wells may begin after the annular seal materials have been installed and allowed to cure, since these wells are designed to retain approximately 90% of the filter pack material. This cure time is typically at least 24 to 48 hours after the sealing materials have been installed.

This TGI is meant to provide a general guide for proper development of newly installed monitoring wells.

A site-specific field implementation plan (FIP) for well installation and development detailing the specific methods and tools is strongly recommended to provide site-specific instruction and guidance.

4 Personnel Qualifications

Generally, Arcadis field personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, and/or state/federal regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as specified in the Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. In addition, Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. The HASP and other documents will identify other training requirements and access control requirements.

The designated Field Manager is responsible for periodic observation of field activities and review of field generated documentation associated with this TGI. The Field Manager is also responsible for implementation of corrective action if problems occur (e.g., retraining personnel, additional review of work plans and TGIs, variances to QC sampling requirements, issuing non-conformances, etc.).

Prior to mobilizing to the field, personnel will review and be thoroughly familiar with relevant site-specific documents including but not limited to the task-specific work plan or field implementation plan (FIP)/field sampling plan/work plan, Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Field personnel assigned to install and develop monitoring wells are responsible for completing their tasks in accordance with the specifications outlined in this TGI and other appropriate and relevant guidelines.

Monitoring well development activities will be performed by persons who have been trained in proper well development procedures under the guidance of an experienced field geologist, engineer, or technician.

5 Equipment List

Required equipment depends on the selected method and should be detailed in the site-specific FIP; however, the following are typically required.

- Approved site-specific Health and Safety Plan (HASP)
- Approved site-specific FIP which will include site map, well construction information/borehole information, and development plan
- Personal protective equipment (PPE) and health and safety equipment, as required by the HASP
- Field notebook and/or smart device (phone or tablet)
- Cleaning/decontamination equipment
 - Non-phosphate laboratory soap (Alconox or equivalent), brushes, clean buckets or clean wash tubs—new buckets or tubs will be purchased if it cannot be determined if the present items are clean
 - Distilled or de-ionized water for equipment decontamination
- Monitoring well keys
- Water-level meter
- Down-hole multiparameter water quality sonde (e.g., YSI)
- Plastic sheeting (e.g., Weatherall Visqueen) to protect all down-hole sampling equipment from contact with potential sources of contamination
- Well development forms/logs
- Well construction logs/diagrams
- Weighted tape (of sufficient length for maximum site depth)
- Turbidity meter
- Camera
- Watch/timing device

6 Cautions

Different USEPA regions and/or state regulatory agencies may stipulate deviations from this document. It is the responsibility of the Project Team (Project Manager and Technical Lead) to be fully aware of the requirements from the applicable regulatory framework.

Prior to beginning field work, the project technical team will ensure that all field logistics (e.g., access issues, health and safety issues, communication network, schedules, etc.) and task objectives are clearly understood by all team members. An internal call with the project technical team to review the FIP/field sampling plan/work plan scope and objectives is strongly recommended prior to mobilization to ensure that the field work will be effectively and efficiently executed.

Where surging is performed to assist in removing fine-grained material from the sand pack, surging must be performed in a gentle manner. Excessive suction could promote fine-grained sediment entry into the outside of the sand pack from the formation.

Avoid using development fluids or materials that could impact groundwater or soil quality or could be incompatible with the subsurface conditions.

In some cases, it may be necessary to add potable water to a well to allow surging and development, especially for new monitoring wells installed in low permeability formations. Before adding potable water to a well, the Certified Project Manager (CPM) and/or Project Hydrogeologist must be notified, and the CPM shall make the decision regarding the appropriateness and applicability of adding potable water to a well during well development procedures. If potable water is to be added to a well as part of development, the potable water source should be sampled and analyzed for constituents of concern, and the results evaluated by the CPM prior to adding the potable water to the well. If potable water is added to a well for development purposes, at the end of development the well will be purged dry to remove the potable water, or if the well no longer goes dry then the well will be purged to remove at least three times the volume of potable water that was added

7 Health and Safety Considerations

Field activities associated with monitoring well development will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities.

Appropriate PPE will be worn at all times in line with the task and the site-specific HASP.

Review all site-specific and procedural hazards as they are provided in the HASP, and review Job Safety Analysis (JSA) documents in the field each day prior to beginning work.

Access to well locations may expose field personnel to hazardous materials such as contaminated groundwater or NAPL (e.g., petroleum hydrocarbons, chlorinated solvents). Other potential hazards include pressurized wells, stinging insects that may inhabit well heads, other biological hazards (e.g., ticks in long grass/weeds around wellhead), and potentially the use of sharp cutting tools (scissors, knife). Open well caps slowly and keep face and body away while allowing to vent any built-up pressure to vent. Only use non-toxic peppermint oil spray for stinging insect nests. Review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives and use appropriate hand protection.

Do not enter confined spaces unless following appropriate confined space entry procedures specified in the HASP.

If thunder or lightning is present, discontinue sampling until 30 minutes have passed after the last occurrence of thunder or lightning.

8 Procedure

As indicated above, for most monitoring wells, gentle surging coupled with bailing or pumping to remove dislodged sediment is recommended.

8.1 Preliminary Well Development

After installation of the primary filter pack around the monitoring well screen, preliminary well development is recommended be performed to ensure that the filter pack settles and does not bridge within the annular space. The preliminary well development steps are as follows:

1. Measure and record depth to water, total depth of well, and depth to top of the sand pack in the annulus.
2. Use steel or weighted bailer to remove any fines that have accumulated in the bottom of the well.
3. Lower an appropriately sized double-surge block into the screened portion of the well on a rigid pipe or high-density tubing and gently cycle up and down to force water in and out of the screen slots and formation. A two-foot throw is recommended (use tape or chalk marks on the pipe or tubing); however, the entire length of well screen must be gently surged.
4. Start above the screen and gently surge over two-foot intervals while working down to the screen bottom.

NOTE: Care must be taken not to surge the well too aggressively at this point as the casing is not well-supported and damage could occur. The objective is to create enough surging action to settle the primary filter pack and provide some preliminary removal of accumulated materials before final development.

NOTE: If possible, ensure that the developer surges the block upward faster than downward to pull the fines out of the filter pack, instead of forcing them back in (and allowing for proper settlement).

5. Monitor the total depth of the well periodically during surging to ensure that we are not pulling excessive amounts of filter pack through the screen and remove any debris accumulated in the well with a weighted bailer or pump.
6. Re-measure the top of the sand in the annulus to see if more sand pack is necessary. Remove any fines that have accumulated out of the well using a submersible pump or weighted bailer.

NOTE: If the monitoring well was drilled using mud rotary drilling methodology or if significant fines were encountered during the well installation, consider adding a commercially available 'mud' dispersant (e.g., AQUA-CLEAR PFD, Nu Well 220, etc.) as part of the preliminary development. This will help to break up the 'skin' along the borehole wall created by either the drilling fluid or smearing during drilling and assist in final development. Follow manufacturer's directions for dosing, and the mixture should be worked through the entire saturated screen interval by gently surging or brushing.

8.2 Final Well Development

After sufficient time has passed to allow for proper curing of the well seal/grout (i.e., 24 to 48 hours), final well development can be performed. Final well development steps are as follows:

1. Don appropriate PPE (as required by the site-specific HASP).
2. Place plastic sheeting around the well.
3. Clean all equipment entering each monitoring well, except for new, disposable materials that have not been previously used.
4. Open the well cover while standing upwind of the well, remove well cap. Insert PID probe approximately 4 to 6 inches into the casing or the well headspace and cover with gloved hand. Record the PID reading in

the field notebook. If the well headspace reading is less than 5 PID units, proceed; if the headspace reading is greater than 5 PID units, screen the air within the breathing zone. If the PID reading in the breathing zone is below 5 PID units, proceed. If the PID reading is above 5 PID units, move upwind from well for 5 minutes to allow the volatiles to dissipate. Repeat the breathing zone test. If the reading is still above 5 PID units, don the appropriate respiratory protection in accordance with the requirements of the HASP. Record all PID readings.

5. Obtain an initial measurement of the depth to water and the total well depth from the reference point at the top of the well casing. Record these measurements in the field logbook. It is recommended to use a weighted tape for the total well depth measurement.
6. The depth to the bottom of the well should be sounded and then compared to the completion form or construction diagram for the well. Any discrepancies should be reported immediately to the CPM and/or Project Hydrogeologist. If sand or sediment is present inside the well, it should first be removed by bailing. Do not insert bailers, pumps, or surge blocks into the well if obstructions, parting of the casing, or other damage to the well is suspected. Instead report the conditions to the CPM and/or Project Hydrogeologist and obtain approval to continue or cease well development activities.

NOTE: If the monitoring well was drilled using mud rotary drilling methodology or if significant fines were encountered during the well installation, it is recommended that a commercially available 'mud' dispersant (e.g., AQUA-CLEAR PFD, Nu Well 220, etc.) be included as part of the final well development to effectively break up the 'skin' along the borehole wall created by either the drilling fluid or smearing during drilling.

Per manufacturer's instructions, the general procedure for adding dispersant is as follows:

- i. Determine volume of water in screen area and double the calculated volume to account for water in gravel pack and formation interface*
 - ii. Once the water volume is determined, calculate the required treatment volume of dispersant need per manufacturer's recommendations*
 - iii. Mix thoroughly before introducing into well*
 - iv. The preferable application method utilizes a tremie line with the product applied into the screened area*
 - v. Mixture should be thoroughly blended in well, then agitated via surging/swabbing/brushing repeatedly (e.g., every two hours) for a period of up to 24 hours*
 - vi. The dispersant should sit for at least 6 to 8 hours or overnight before continuing well development activities*
7. After allowing the dispersant to sit for the required time (if dispersant is used), start the mechanical development by lowering an appropriately sized double-surge block (or similar) into the well on a rigid pipe or high-density tubing.
 - i. Surging should start above the screen to reduce the possibility of "sand-locking" the surge block. Initial surging should be with a long stroke and at a slow rate (20 to 25 strokes per minute)
 - ii. After surging above the screen, the well should be cleaned via bottom-loading bailer, submersible pump, or inertia pump tubing with check valve to the bottom of the well

- iii. Begin surging at the lower end of the screen, gradually working upward, surging in 2-ft intervals until the entire screen has been developed.
 - iv. Surge the well a minimum of 10 throws per 2-ft screen interval.
 - v. Each interval may require several surge cycles to achieve the best development.
 - vi. The entire length of well screen must be surged.
 - vii. Ensure that the developer surges the block upward faster than downward to pull the fines out of the filter pack, instead of forcing them back in (and allowing for proper settlement)
 - viii. measure total depth of the well periodically during surging to ensure that excessive amounts of sediment are not being pulled through the screen. Remove any debris accumulated in the well via simultaneous airlifting (if a combined tool is available) or with bailing/pumping.
8. After completing a cycle of surging, lower a bottom-loading bailer, submersible pump, or inertia pump tubing with check valve to the bottom of the well and gently bounce on the bottom of the well to collect/remove accumulated sediment, if any. Remove and empty the bailer, if used. Repeat until the bailed/pumped water is free of excessive sediment and contact at the bottom of the well feels solid. Alternatively, measurement of the well depth with a weighted tape can be used to verify that sediment and/or silt has been removed to the extent practicable, based on a comparison with the well installation log or previous measurement of total well depth.
9. After surging the well for a minimum of two cycles and removing excess accumulated sediment from the bottom of the well, re-measure the depth-to-water and the total well depth from the reference point at the top of the well casing. Record these measurements in the field log book.
10. Remove formation water by pumping/bailing.
- i. Where pumping is used, measure and record the pre-pumping water level.
 - ii. Operate the pump at a relatively constant rate
 - iii. Measure the pumping rate using a calibrated container and stopwatch, and record the pumping rate in the field log book
 - iv. Measure and record the water level in the well at least once every 5 minutes during pumping
 - v. Record any relevant observations in terms of color, visual level of turbidity, sheen, odors, etc.
 - vi. Pump or bail until termination criteria specified in the site-specific FIP are reached
 - vii. Record the total volume of water purged from the well

NOTE: The FIP may also specify a maximum turbidity requirement for completion of development. Unless otherwise specified the maximum turbidity should be 50 NTUs or less

11. While developing, take periodic water level measurements (at least one every five minutes) to determine if drawdown is occurring and record the measurements on the Well Development Log.
12. While developing, calculate the rate at which water is being removed from the well. Record the volume on the Well Development Log.
13. While developing, water is also periodically collected directly from the well or bailer discharge and readings taken of the indicator parameters: pH, specific conductance, and temperature. Development is

considered complete when the indicator parameters have stabilized (i.e., three consecutive pH, specific conductance, and temperature readings are within tolerances specified in the project work plans or within 10% if not otherwise specified), the extracted water is clear and free of fine sediment and most importantly, when acceptable volume of water has been removed and/or a sufficient amount of surging has been performed.

14. In certain instances, for slow recharging wells, the parameters may not stabilize. In this case, well development is considered complete when minimal amounts of fine-grained sediments are recovered, and an acceptable volume of water has been removed.
15. If the well goes dry, stop pumping or bailing. Note the time that the well went dry. After allowing the well to recover, note the time and depth to water. Resume pumping or bailing when sufficient water has recharged the well.
16. Contain all development water in appropriate containers.
17. When complete, secure the lid back on the well.
18. Place disposable materials in plastic bags for appropriate disposal and decontaminate reusable, downhole pump components and/or bailer

9 Waste Management

Investigation-Derived Waste (IDW), including purge water and decontamination liquids, will be stored on site in appropriately labeled containers and disposed of properly. Disposable materials will be stored and disposed of separately. Containers must be labeled at the time of collection and will include date, location(s), site name, city, state, and description of matrix contained (e.g., water, PPE). Waste will be managed in accordance with the *TGI – Investigation-Derived Waste Handling and Storage*, the procedures identified in the FIP/field sampling plan/work plan or QAPP as well as state-, federal- or client-specific requirements. Be certain that waste containers are properly labeled and documented in the field log.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

All well development activities will be documented on appropriate log forms as well as in a proper field notebook and/or PDA. Additionally, all documents (and photographs) should be scanned and electronically filed in the appropriate project directory for easy access. Pertinent information will include personnel present on site; times of arrival and departure; significant weather conditions; timing of well development activities; development

method(s); observations of purge water color, turbidity, odor, sheen, etc.; purge rate; and water levels before, during, and after pumping.

Management of the original documents from the field will be completed in accordance with the site-specific QAPP. Records generated as a result of this TGI will be controlled and maintained in the project record files in accordance with project requirements.

Development activities will be documented on appropriate field logs as well as in a proper field notebook. All field data will be recorded digitally or with indelible ink. Field forms, logs/notes (including daily field and calibration logs), digital records, and chain-of-custody records will be maintained by the field team lead. Any deviations or omissions from this TGI should be documented.

Initial field logs and forms will be transmitted to the Arcadis CPM and/or Technical Lead at the end of each day unless otherwise directed by the CPM. The field team leader retains copies of the field documentation.

11 Quality Assurance

Quality assurance procedures will be conducted in accordance with the Arcadis Quality Management System or the site-specific QAPP. Refer to the QAPP or FIP/sampling plan/work plan for specific requirements.

12 References

American Society for Testing Materials (ASTM), Designation D5521-05. *Standard Guide for Development of Ground-Water Monitoring Wells in Granular Aquifers*. American Society for Testing Materials. West Conshohocken, Pennsylvania.

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PFAS-SPECIFIC DRILLING AND MONITORING WELL INSTALLATION TECHNICAL GUIDANCE INSTRUCTION

Rev: #3

Rev Date: 4/15/2020



VERSION CONTROL

| Revision No | Revision Date | Page No(s) | Description | Reviewed by |
|-------------|---------------|--------------------------------|---|-----------------|
| 0 | 10/12/2018 | All | Generated from generic Well Installation TGI (Rev 0, April 24, 2017). Revised to be PFAS-specific, provide more instruction on soil sample collection, and only include DPT and Sonic methods | Ankit Gupta |
| 1 | 3/26/2019 | Attachments | Added a restriction against all fluoropolymer materials in Table 2 for sampling; Removed Citranox as an appropriate decon solution in Table 1. Made a correction that Liquinox contains trace levels of 1,4 Dioxane, not Alconox. | Erika Houtz |
| 2 | 12/19/2019 | 5-7, 9-10, 13-15, 18-19, 22-28 | Updated equipment list. Added procedures for single-interval groundwater sampling during drilling activities. Added procedures for hollow stem auger drilling. | Joseph Quinnan |
| 3 | 4/15/2020 | 28-34 | Added procedures for air rotary drilling | Eric Killenbeck |

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APPROVAL SIGNATURES

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1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to any and all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, state-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

This Technical Guidance Instruction (TGI) describes methods for the following at sites impacted by per- and polyfluoroalkyl substances (PFASs):

- Advance soil borings via direct push technology (DPT), rotosonic, hollow stem auger, or air rotary drilling techniques
- Collect single or multiple depth-discrete dry and/or saturated soil samples
- Collect single-interval grab groundwater samples
- Install groundwater monitoring wells in unconsolidated aquifers (as necessary).

This TGI covers specific considerations relevant for PFASs due to their unique chemical and physical properties, low detection limits, and low regulatory standards. A more detailed discussion of general PFAS sampling procedures is provided in PFAS Field Sampling Guidance TGI (Arcadis 2018a).

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If monitoring wells are to be installed upon completion of borehole drilling and soil sampling, it is assumed that the monitoring well has been designed consistent with the approach and methods presented in the American Society of Testing and Materials (ASTM) D5092 – *Standard Practice for Design and Installation of Groundwater Monitoring Wells* (ASTM D5092). This includes sizing of the filter pack and screen slot size, the length of the screen, total depth of the well, material strength and compatibility and surface completion. Typical monitoring wells are constructed of manufactured screen and engineered filter pack and are generally suitable for formations with granular materials having a grain size distribution with up to 50% passing a #200 sieve and up to 20% clay-sized material. Monitoring wells installed in formations finer than this may not be able to produce turbidity free water.

The procedures set out herein are designed to produce standard groundwater monitoring wells suitable for: (1) groundwater sampling; (2) water level measurement; and (3) hydraulic conductivity testing of formation sediments immediately adjacent to the open interval of the well (e.g., slug testing).

This TGI will focus specifically on four drilling methods most likely to be utilized during drilling and soil sampling activities: DPT, rotosonic, hollow stem auger, and air rotary techniques. The drilling method to be used at a given site will be selected based on site-specific consideration of anticipated drilling depths, site or regional geologic knowledge, type of sampling to be conducted, project objectives, and cost.

No oils or grease will be used on equipment introduced into the boring (e.g., drill rod, casing, or sampling tools). No polyvinyl chloride (PVC) glue/cement will be used in constructing or retrofitting monitoring wells that will be used for water-quality monitoring. No coated bentonite pellets will be used in the well drilling or construction process. Specifications of materials to be installed in the borehole will be obtained prior to mobilizing onsite; these materials generally include:

- Well casing (length, material, and diameter);
- Well screen (length, material, diameter, and slot size);
- Bentonite (type, as applicable, chips, non-coated and granular bentonite are acceptable);
- Filter pack (filter pack type and fine sand seal type, as applicable); and
- Grout (type, as applicable).

Well materials will be inspected and, if needed, cleaned or replaced prior to installation.

3 PERSONNEL QUALIFICATIONS

Drilling and soil sampling activities will be performed by persons who have been trained in proper procedures under the guidance of an experienced field geologist, engineer, or technician, with particular emphasis on PFAS sampling procedures outlined in PFAS Field Sampling Guidance TGI (Arcadis 2018a). Field personnel will have undergone in-field training in soil description methods, as described in Soil Description TGI (Arcadis 2018b).

4 EQUIPMENT LIST

The following equipment and materials must be available for borehole advancement, single-interval groundwater sampling, and well construction activities:

- Site plan with proposed sampling locations;
- Relevant work plan (e.g., installation-specific Quality Assurance Project Plan [QAPP] Addendum);
- Site Safety and Health Plan (SSHP);
- Appropriate health and safety equipment, as specified in the SSHP;
- Drilling Equipment:
 - DPT, rotosonic, hollow stem auger, or air rotary drill rig, to be provided by drilling subcontractor. Type to be determined based on site-specific details.
 - Direct push groundwater samplers (e.g., Geoprobe® SP-22 or Geoprobe® SP-16) rotosonic sampling devices (e.g., Cascade Packer Isolation Groundwater Profiler or Geoprobe® SP-60 Sonic Groundwater Sampler), or hollow stem auger/air rotary sampling devices (e.g., pre-packed PVC well screens and PVC riser), to be provided by drilling subcontractor.
 - Traffic cones, delineators, caution tape, and/or fencing as appropriate for securing the work area, if not provided by the drillers.
 - Note: Prior to mobilizing to the site, Arcadis personnel will contact the drilling subcontractor or in-house driller (as appropriate) to confirm that appropriate sampling equipment will be provided in quantities capable of achieving estimated target depths. Specifications of the sampling and well installation equipment are expected to vary by project, so communication with the driller is necessary to ensure that the materials provided will meet the project objectives. Equipment and materials typically provided by the driller could include:
 - Disposable acetate (or Lexan TM) liners (when drilling with direct-push equipment)
 - Appropriate length of drilling rods and tooling
 - Drilling and sampling equipment decontamination materials
 - Decontamination pad materials
 - Well construction materials
 - Drums for investigation derived waste
- Sampling:
 - Appropriate PFAS-free groundwater sampling equipment (e.g., disposable bailers for volumetric sampling, peristaltic pump for shallow groundwater sampling, submersible bladder pump for deeper sampling). Refer to the PFAS Field Sampling Guidance TGI (Arcadis 2018a) and Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells TGI (Arcadis 2016) for necessary equipment.
 - Stainless-steel spatulas, spoons, and trowels.

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- Stainless-steel hand auger with at least 10-ft of extension rods.
- PVC piping of larger diameter than hand auger, if necessary, to keep hand auger borings open.
- Soil logging equipment as specified in the appropriate project documents.
- Dedicated low-density polyethylene (LDPE) plastic sheeting to prevent sample contact with the ground.
- Photoionization detector (PID) or flame ionization detector (FID) with calibration gas.
- 4-gas meter with calibration gas.
- Water level meter with fluorine-free materials (Geotech ET 3/8" with Delrin tip and Buna-N O-ring).
- YSI 6-Series multi-parameter water quality probe or equivalent (e.g., conductivity, temperature, dissolved oxygen, oxidation reduction potential) with flow-through cell.
- Turbidity meter.
- Laboratory provided PFAS-free water for field and equipment blank QC samples.
- Laboratory-provided HPDE PFAS shaker test vials.
- Appropriate sample containers and labels:
 - Laboratory-supplied HDPE sample bottles: see the Poly- and Perfluorinated Alkyl Substances (PFAS) Field Sampling Guidance (Arcadis 2018a) for PFAS-specific considerations.
 - Polyethylene bags (Ziploc® brand only) to hold ice and samples.
 - Appropriate blanks (field reagent blanks supplied by the laboratory).
 - Packing and shipping materials.
 - Chain-of-Custody (COC) Forms; see the Sample Chain of Custody Standard Operating Procedure (SOP) for reference (Arcadis 2017a).
 - Appropriate transport containers (coolers) with ice and appropriate labeling; no blue ice.
- Decontamination/Waste Management:
 - PFAS-free decontamination fluids and equipment:
 - HDPE or PVC brushes and squirt bottles
 - Stainless steel bowl
 - HDPE buckets to hold decontamination fluids
 - Alconox or Liquinox (other detergents prohibited)
 - Distilled or laboratory-supplied deionized water
 - Laboratory provided PFAS-free water

- See the Poly- and Perfluorinated Alkyl Substances (PFAS) Field Sampling Guidance (Arcadis 2018a) or the Groundwater and Soil Sampling Equipment Decontamination TGI (Arcadis 2017b) for additional guidance.
- Portable field hand washing setup.
- Non-hazardous drum labels as required for investigation-derived waste handling: see the Investigation-Derived Waste Handling and Storage TGI for details (Arcadis 2017c).
- Field Notes:
 - Pens, pencils, and/or Sharpies® for writing
 - Appropriate field forms
 - Clipboards, field binders, field notebook, and field note pages that are not waterproof
 - Digital camera
- Other:
 - Field clothing made of cotton or other natural fibers that is well laundered (i.e., washed at least 6 times)
 - Well laundered cotton blankets for covering field vehicle seats
 - PFAS-free sunscreen and insect repellent
 - Garbage bags
 - Paper towels
- Locks and keys for securing the well after installation
- Engineer's tape/measuring wheel

5 CAUTIONS

5.1 Utility Clearance

The appropriate drilling authorities will be contacted and a site visit for public utility line clearance at the proposed boring locations will be conducted at least 72 hours prior to work commencing. As applicable, utility maps will be reviewed during field reconnaissance of the proposed inspection locations to determine if any are co-located with public utility lines. Arcadis will also contract an independent geophysical survey company to verify that proposed boring locations are not co-located with existing underground utility/substructure features, as necessary. Arcadis will clear locations with soft dig methods to assess the presence of underground utilities as necessary. See the Utility Location and Clearance Arcadis Health and Safety Standard (Arcadis 2017d) for reference.

5.2 General Drilling and Well Construction Considerations

Prior to beginning field work, contact the project technical team to ensure that all field logistics (e.g., access issues, health and safety issues, communication network, schedules, etc.) and task objectives are clearly understood by all team members.

Some regulatory agencies require a minimum annular space between the well or permanent casing and the borehole wall. When specified, the minimum clearance is typically 2 inches on all sides (e.g., a 2-inch diameter well requires a 6-inch diameter borehole). In addition, some regulatory agencies have specific requirements regarding grout mixtures. Determine whether the oversight agency has any such requirements prior to finalizing the drilling and well installation plan.

If dense non-aqueous phase liquids (DNAPL) are known or expected to exist at the site, refer to the project specific documents for additional details regarding drilling and well installation to reduce the potential for inadvertent DNAPL remobilization.

Similarly, if light non-aqueous phase liquids (LNAPLs) are known or expected to be present as “perched” layers above the water table, refer to the DNAPL Contingency Plan. Follow the general provisions and concepts in the DNAPL contingency plan during drilling above the water table at known or expected LNAPL sites.

Consider the compatibility between the well materials and the surrounding environment. For example, PVC well materials are not preferred when DNAPL is present. In addition, some groundwater conditions leach metals from stainless steel or are corrosive to metal well materials. If questions arise, contact the CPM and/or project technical lead to discuss.

Specifications of materials used for backfilling the borehole will be obtained, reviewed and approved to meet project quality objectives. Bentonite is not recommended where DNAPLs are likely to be present or in groundwater with high salinity. In these situations, neat cement grout is preferred.

As noted above, coated bentonite pellets will not be used in monitoring well construction, as the coating could impact the water quality in the completed well.

Heat of hydration during neat cement grout curing must be considered to avoid damage to PVC well materials. The annular space for a typical monitoring well is small enough that heat of hydration should not create excessive temperature increases which may damage PVC well material. However, washouts in the borehole can lead to thick accumulations of grout which can produce enough heat during curing to weaken and potentially damage PVC casing. If heat of hydration is a concern, contact the project technical lead to address the issue.

5.3 PFAS-Specific General Sampling Considerations

This section provides a summary of methods and procedures applicable to the collection of environmental samples for field screening or laboratory analysis during PFAS site characterization activities. In general, sampling techniques used for PFAS site characterization are consistent with conventional sampling techniques used in the environmental industry, but special consideration is made regarding PFAS-containing materials and cross-contamination potential. For example, Teflon™ and other fluoropolymer containing materials are found in pumps, tubing, and sample storage containers and therefore should be

avoided (Department of Environment Regulation [DER], Western Australia 2016; New Hampshire Department of Environmental Services [NHDES] 2016). Certain field documentation materials such as waterproof paper or field books, adhesive paper products, and some writing utensils (grouped as non-Sharpie® markers) are also prohibited items during PFAS sampling (DER 2016; NHDES 2016).

New nitrile gloves should be donned before any of the following activities:

- Decontamination of re-usable sampling equipment;
- Contact with sample bottles or PFAS-free water bottles;
- Handling clean sample tubing/down-well equipment or connecting tubing;
- Handling QC samples including field blanks and equipment blanks.

Additionally, new nitrile gloves should also be donned after handling of any non-dedicated sampling equipment; contact with contaminated surfaces; and whenever judged necessary by field personnel.

When in doubt change your gloves.

Prior to initiating field activities, water sources to be used during drilling activities (e.g., roto-sonic drilling, should be sampled to verify those sources are PFAS-free. While not part of the PQAPP, this is considered best practice and should be completed to the extent possible.

Waterproof field books must not be used for field notes. Instead, field notes should be on loose paper on Masonite, plastic, or aluminum clip boards. Other requirements for field notes include:

- Keep field notes, writing implements, and electronic data collection tablets away from samples and sampling materials; and,
- Do not write on sampling bottles unless they are closed.

Tables 1 and 2 in Attachment 1 provide recommendations for PFAS Site Inspection equipment. **Table 1** provides a summary of materials that have been approved for site inspection; this list is expected to grow longer as industry experience increases. **Table 2** provides a summary of field equipment and materials that have available testing information and/or industry knowledge regarding PFAS cross-contamination potential and it is recommended that these materials be prohibited for sample collection. For materials that are suspected of containing PFASs and/or retaining PFASs, these recommendations are considered preliminary and subject to change.

Given the extremely low detection limits associated with PFAS analysis and the many potential sources of trace levels of PFASs, field personnel are typically advised to err on the side of caution by strictly following field wear guidelines and decontamination procedures as specified in the Poly- and Perfluorinated Alkyl Substances (PFAS) Field Sampling Guidance (Arcadis 2018a). **The most important consideration during PFAS related drilling and soil sampling is to prevent contact between sample media and suspect PFAS sources.**

5.4 PFAS-Specific Groundwater Sampling

The potential presence of PFASs in equipment that may come in contact with the target water sample must be evaluated as part of the sample planning process to maintain sample integrity. For example, low-flow sampling with a peristaltic pump should be conducted using silicone or HDPE tubing; Teflon™ tubing is prohibited (DER 2016). If a bladder pump is used to collect samples, the bladder and other internal

parts (e.g., check balls, o-rings, compression fittings) should not be made of Teflon™ either, and bladder and o-rings should be changed between samples (DER 2016).

Note that if high concentrations of PFASs related to Class B firefighting foams are expected in a groundwater sample, it has been recommended to collect and shake a small portion of the sample at the time of sample collection (USACE 2016; Arcadis 2018a). If foaming is noted within the sample, it indicates elevated concentrations of PFASs may be present and the sample should be proactively diluted at the laboratory prior to analysis. The foaming should be noted on the sample chain of custody form. It is recommended to collect sampling equipment blanks following foam observation to confirm the effectiveness of decontamination procedures.

5.5 PFAS-Specific Soil Sampling

Equipment that contacts soil cuttings during sampling activities should be carefully considered and selected. PFAS-containing materials are potentially present in some of the equipment typically used for soil sampling. This includes any lubricants, connections, fittings, etc. used on the cutting shoe on the head of a direct push drill string. Additionally, no materials that pose a cross-contamination risk should be introduced to the bucket of a hand auger. **To minimize the risk of cross-contamination, all hand augering activities (i.e. augering, sample collection, decontamination) should be performed by Arcadis personnel (as opposed to drilling subcontractor) when surface soil samples will be collected.** Each piece of reusable drilling/sampling equipment that comes into direct contact with soil cuttings or groundwater must be inspected before use to confirm that PFAS-containing materials are not present, which could be a source of cross-contamination and cause false positives, and that PFASs will not adhere to the material, which has the potential to cause low bias sample results. If equipment cannot be verified as being PFAS-free and there is a concern that it could potentially introduce contamination, a conservative number of equipment blanks should be collected to confirm that materials in the sample equipment do not cause false positives by introducing PFASs. Other quality assurance methods may be implemented to avoid materials that could result in potential losses associated with PFASs adhering to surfaces. For example, collecting soil samples for laboratory analysis from an “undisturbed” portion of a large diameter soil core is a good practice.

The following additional notes are provided regarding soil sampling materials:

- Where drilling or decontamination water is needed, a sample of the source water must be collected and analyzed for PFAS before drilling begins to ensure that background PFASs will not be introduced. Some water systems may be constructed with PFAS-containing thread and gasket sealants; therefore, an inspection of the source water distribution system may provide an additional level of assurance for identifying a source of PFAS-free water for site inspections.
- It is often standard practice to cover the ends of sample sleeves and protect the sample from potential cross-contamination from the plastic end caps with Teflon™ or other PTFE tape (Geotechnical Services, Inc. 2018); this practice is prohibited for PFAS sample collection (DER 2016).
- Lexan™ liner sleeves are made of polycarbonate and they are not expected to contain PFASs based on review of the Safety Data Sheet (Sabic 2016).

- Acetate (i.e., cellulose acetate butyrate) liners are commonly used as sleeves and are not expected to contain PFASs.
- Studies evaluating the use of stainless steel indicate that PFASs do not strongly sorb to stainless-steel (Obal et al. 2012). Therefore, stainless-steel sleeves and equipment should be acceptable for collection of soil samples for PFAS analysis.

6 HEALTH AND SAFETY CONSIDERATIONS

Field activities associated with drilling, soil sampling, and monitoring well installation will be performed in accordance with the SSHP, a copy of which will be present on site during such activities.

7 PROCEDURE

The procedures for drilling, soil sampling, and installing groundwater monitoring wells (if necessary), are presented below. All field sampling should be completed by a two-person team, with one collecting the samples, and the other handling documentation and providing support. This will help to limit the potential for accidental cross-contamination of the sample media.

7.1 Direct Push Technology (DPT) Method

Direct-push drilling may be used to complete soil borings and install monitoring wells. Examples of this technique include the Diedrich ESP vibratory probe system, GeoProbe®, or AMS Power Probe® dual-tube system. Environmental probe systems typically use a hydraulically operated percussion hammer. Depending on the equipment used, the hammer delivers 140- to 350-foot pounds of energy with each blow. The hammer provides the force needed to penetrate very stiff to medium dense soil formations. The hammer simultaneously advances an outer steel casing that contains a dual-tube liner for sampling soil. The outside diameter (OD) of the outer casing ranges from 1.75 to 2.4 inches and the OD of the inner sampling tube ranges from 1.1 to 1.8 inches. The outer casing isolates shallow layers and permits the unit to continue to probe at depth. The double-rod system provides a borehole that may be tremie-grouted from the bottom up. Alternatively, the inside diameter (ID) of the steel casing provides clearance for the installation of small-diameter (e.g., 0.75- to 1-inch ID) micro-wells. The procedures for drilling, soil sampling, single-interval groundwater sampling, and installing monitoring wells in soil using the direct-push method are described below.

1. Place LDPE plastic sheeting over core/sampling processing area to create a clean working surface, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity.
2. Clear the ground surface of brush, root mat, grass, leaves, or other debris prior to sampling.
3. Decontaminate all non-disposable sampling equipment and tooling that will or may to come into direct contact with soil prior to first use. Disposable sampling equipment must be kept in sealed PFAS-free packaging until it is used.
4. Use stainless-steel hand auger to collect samples from 0 - 5 ft bgs surface interval, if applicable. All hand auguring to collect soil samples will be completed by Arcadis personnel, not the drilling

subcontractor. These samples can be collected either during other Utility Clearance activities (e.g., third party clearance) or immediately prior to drilling.

- a. The sample should be collected manually directly from the hand auger bucket (using stainless steel scoop, spatula, or trowel as necessary) and placed directly into the sample jar, following Steps 7 – 15 below. The sample should not contact the ground or LDPE sheeting.
 - b. If collecting multiple samples from the same boring, after collecting sample from the surface or shallowest depth interval examine the stability of the soil in the boring sidewalls. If sidewalls appear to be at risk of collapsing into the borehole insert a length of PVC pipe into the boring to maintain the opening and prevent collapse prior to augering to the next deeper sampling interval.
5. Use dual tube rod system and collect soil cores in acetate or Lexan™ liners. The cutting shoe and core extractor must be stainless steel with no PFAS-containing materials present (e.g., gaskets, coatings).
 6. After each drilling run, drillers extract and cut open liners and provide to Arcadis personnel for characterization and sampling. Drillers must not touch soil inside of liners during this process. Arcadis personnel decontaminate cutter between uses (see below).
 7. Don a new set of nitrile gloves prior to handling sample core, then characterize soils in accordance with P-04 TGI - Soil Description (Arcadis 2018b). Record descriptions in the field notes, boring logs, and/or tablet/cell phone via Arcadis Fulcrum application. It is also beneficial to photo document the samples. It should be noted that logs collected via tablet or cell phone must be electronically backed up and transferred to a location accessible to other project team members as soon as feasible to retain and protect the field data.
 8. Don a new set of nitrile gloves prior collecting soil samples for analysis. Do not use gloved hands to handle items (e.g., papers, pens, clothes) before collecting samples. Do not touch outside of sample liner with gloved hands.
 9. Collect field samples and any required QC samples from recovered soil cores using a clean stainless-steel trowel and place in clean, labeled bottles supplied by the laboratory for the required analyses. If collecting samples for multiple analyses, collect PFAS samples first. Make sure caps remain on PFAS sample bottles until immediately prior to filling. Caps must remain in the hand of the sampler until replacing on the bottle.
 10. Once the sample has been placed in the bottle, and the bottle cap has been completely tightened, label the sample with sample identification number, date, and time of collection. Labels must be completed only after the caps have been placed back on each bottle. (See P-01 QP#3.06 Field Activities documentation for sample label information).
 11. Place soil sample bottles in a sealed Ziploc® bag, and then into sample coolers. Store PFAS samples in separate cooler from other samples.
 12. Record the label information and time of sampling in the field notes and sampling forms.
 13. Fill out laboratory COC and check against the labels on the sample bottles progressively after each sample is collected.
 14. Decontaminate all reusable sampling equipment between sample intervals and borings as described in Section 10.
 15. Repeat Steps 7 – 14 until all samples have been collected from the boring location.

16. Abandon soil boring to grade in accordance with the site-specific work plan upon completion and before moving to the next boring location. **If single-interval groundwater sample is required, see Section 7.1.1 for procedure. If well is to be installed, see Section 7.1.2 for well construction procedure.**
17. Mark boring location with wooden stake that identifies boring ID for subsequent surveying, as necessary.
18. Manage investigation-derived-waste (IDW) as specified in Section 8 and in accordance with the site-specific work plan.
19. If samples are not shipped the same day as collected, add fresh ice to sample coolers at the end of the day to maintain the temperature between 0 and 6°C. Place ice in sealed Ziplock® bags. Do not use blue ice. Sample coolers must remain in the possession of the sampling team at all times or secured under lock and key until shipment to the laboratory.

7.1.1 DPT Single-Interval Groundwater Sampling

The steps below describe the procedure for collecting a single-interval groundwater sample at a desired sample interval, commonly from the first encountered shallow groundwater. It should be verified that samples are taken below the water table (i.e., not perched water in the vadose zone). Single-interval sampling should be performed with a Geoprobe® SP-22 sampling device or similar (e.g., HydroPunch™). A primary difference with single-interval methods relative to multi-interval vertical aquifer profiling (VAP) sampling is that the sampling screen is driven to the appropriate depth by the drill rig instead of lowering the sampling screen through the drill tools after reaching the appropriate depth. If multi-interval VAP sampling is required, refer to Arcadis TGI for Vertical Aquifer Profiling for PFAS Analysis (Arcadis 2019).

If using a Geoprobe® SP-22 sampling device, the following steps will be followed:

1. Ensure 4-gas meter, YSI 6-Series multi-parameter water quality probe, and turbidity meter are calibrated each morning (see QAPP worksheet #22 and P-09 Calibration and Control of Measuring and Test Equipment in PQAPP Appendix A). Document calibration results on equipment calibration log.
2. Advance Geoprobe® SP-22, equipped with stainless-steel screen, using standard Geoprobe® rods to the target depth interval in accordance with **Section 7.1**.
3. Retract the outer casing to expose the screen for the desired sample interval length, using extension rods to hold the screen in place. The Geoprobe® SP-22 sampling screen can be either 12" or 48". Note: no soil cores will be retrieved using the Geoprobe® SP-22.
4. Go to Step 9.

If using a HydroPunch™ sampling device, the following steps will be followed:

1. Ensure 4-gas meter, YSI 6-Series multi-parameter water quality probe, and turbidity meter are calibrated each morning (see QAPP worksheet #22 and P-09 Calibration and Control of Measuring and Test Equipment in PQAPP Appendix A). Document calibration results on equipment calibration log.
2. The drilling subcontractor will advance the borehole to approximately 2 feet above the depth from which a discrete water sample is to be obtained.

3. The drilling subcontractor will disassemble the HydroPunch™ sampling device according to the manufacturer's instructions to allow the sampler to be decontaminated. The sampler should be completely disassembled, including O-rings and/or check valves.
4. Decontaminate the sampler per instructions in Section 10 as appropriate for the range of groundwater analytes to be sampled for, by washing with laboratory-grade detergent and potable water wash, followed by solvent rinse (if sampling for organics) and final rinse with deionized or distilled water. Check the condition of the O-rings during each cleaning and replace if necessary.
5. The drilling subcontractor will reassemble the decontaminated HydroPunch™ sampling device according to the manufacturer's instructions and lower the device to the bottom of the borehole.
6. The drilling subcontractor will push or drive the HydroPunch™ 5 feet below the bottom of the casing or augers, then retract the sampler 3 feet upward. Subsurface friction will retain the drive point in place, exposing the screen and allowing groundwater to enter the sampling tool.
7. Allow sufficient time to allow the sampler to fill with water. Typically, 30 minutes is sufficient, except in low permeability materials. If sufficient water has not accumulated in approximately 30 minutes, leave the temporary well casing and screen in the borehole to allow for overnight recovery of groundwater for follow-up sampling the next day.
8. Go to Step 9.

Complete the following steps for sample collection:

9. Place LDPE plastic sheeting adjacent to the sample port for use as a clean work area if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity. Do not allow vehicle exhaust to point towards the sample point.
10. Don a new set of nitrile gloves, connect tubing to sampling pump and flow-through cell, and slowly lower tubing and/or pump into well. If possible, use two field personnel to insert tubing/pump into well to avoid contact with surrounding ground surface or other materials that could cause cross-contamination. Insert tubing (peristaltic pump, if depth to water <25 ft bgs), or pump intake (small-diameter bladder pump, if depth to water >25 ft bgs) at the approximate mid-depth of the sampler screen interval.
 - Alternately, a Waterra-type inertial pump can be used to retrieve the water sample. If the formation has low-permeability and enough water is not anticipated in the tooling to allow purging of water, a stainless-steel bailer may be considered (after consulting with Arcadis RL).
11. Purge until water is visually clear of sediment, or for a maximum of 20 minutes before collecting GW samples.
 - Note: for low-permeability formations, collect a grab sample from the screen point sampling device and/or saturated soil sample from the soil core. Leave temporary well casing and screen in the borehole to allow for overnight recovery of groundwater for follow-up sampling the next day.
12. Don a new set of nitrile gloves prior to collecting groundwater sample and each QC sample. Do not use gloved hands to handle items (e.g., papers, pens, clothes, equipment) before collecting samples.

13. Fill sample bottles using labeled HDPE bottles that are supplied by laboratory only. Ensure the cap remains on the bottle until immediately prior to sample collection and gets placed back on the bottle immediately after sample collection. Do not place the cap on any surface; keep in hand opposite of sample collection and do not touch the inside of the cap.
14. If high concentrations of PFASs related to Class B firefighting foams are expected in a groundwater sample (as specified in the QAPP Addendum), collect and shake a small portion of the sample (approximately 10-25 mL) on site. If foaming is observed, document the foaming on the sample log and on the COC to notify laboratory personnel. The “shaker test” vial can then be disposed of as specified in Section 8.
15. Collect QC samples at frequency specified in PQAPP Worksheet #20. QC sample locations to be selected based on consultation with Arcadis RL.
16. Place filled sample bottles in a sealed (Ziploc[®]) bag, record any label information that was not previously filled out (e.g., sample time). Record the label information and time of sampling in the field notes and sampling forms. Place samples into sample coolers. Store PFAS samples in separate cooler from any other types of samples.
17. Fill out laboratory COC and check against the labels on the sample bottles progressively after each sample is collected.
18. Pull the Geoprobe[®] SP-22, HydroPunch[™], or other sampling device back up and decontaminate per instructions in Section 10.

7.1.2 DPT Monitoring Well Construction

1. Upon advancing the borehole to the desired depth, install the well through the inner drill casing. The well will consist of 2-inch ID PVC or stainless-steel slotted screen and blank riser. Screen length and construction will be specified in the Work Plan or discussed with the Arcadis PM.
2. When the monitoring well assembly has been set in place, place a washed silica filter pack in the annular space from the bottom of the boring to a height of 1 to 2 feet above the top of the well screen (following specifications in the Work Plan) using a tremie pipe. The filter pack is placed, and drilling equipment (i.e., rods) extracted in increments until the top of the sand pack is at the appropriate depth. Verify that the expected volume of filter pack matches with the actual amount installed. There can be differences due to irregularities in the borehole. Washout of the borehole will result in the need for greater than calculated well materials. If a difference of more than 10% is noted, consult with the project technical team. The filter pack will be consistent with the screen slot size and the soil particle size in the screened interval, as specified in the Work Plan. The well should be gently surged to prevent filter pack material bridging and to settle the filter pack prior to well seal installation.
 - a. Alternately, a monitoring well assembly with a pre-packed screen can be installed. The monitoring well assembly (i.e., regular PVC or pre-packed) should be discussed and decided prior to beginning field work and specified in the QAPP addendum. Pre-packed filters should be verified as PFAS-free prior to use.

3. A hydrated bentonite seal (a minimum of 2 feet thick) will then be placed in the annular space above the sand pack (alternatively, in some cases a fine sand seal may be installed instead of bentonite—follow the specifications in the Work Plan). Use of a tremie pipe is not required for placement of the bentonite seal (though may be required if a well is very deep and borehole bridging is reasonably anticipated). However, bentonite should be poured into the annular space slowly enough to ensure borehole bridging does not occur. If non-hydrated bentonite is used, the bentonite should be permitted to hydrate in place for a minimum of 30 minutes before proceeding. *No coated bentonite pellets will be used in monitoring well drilling or construction.* PFAS-free water (verified by laboratory analysis of source water) should be added to hydrate the bentonite if the seal is above the water table. Continuously monitor the placement of the sand pack and bentonite with a weighted tape measure.
4. During the extraction of the augers or casing, a cement/bentonite or neat cement grout will be placed in the annular space from the bentonite seal to a depth approximately 2 ft bgs or as specified in the Work Plan. As with the filter pack, it is recommended that seal material be placed with a tremie pipe. Ensure that seal materials are mixed at the proper ratios with PFAS-free water (verified by laboratory analysis of source water) following manufacturer's recommendations.
5. Install the monitoring well completion as specified in the Work Plan. Typical completions are a locking, steel protective casing (extended at least 1.5 feet below grade and 2 feet above grade) over the riser casing set within a neat cement pad at grade. Alternatively, for flush-mount completions, place a steel curb box with a bolt-down lid over the riser casing set within a neat cement pad. In either case, the cement pad will extend approximately 1.5 to 2.0 feet below grade and laterally at least 1 foot in all directions from the protective casing and should slope gently away to promote drainage away from the well.
6. During well installation, record construction details and tabulate materials used in field notebook as well as appropriate field forms.
7. After completing the well installation, lock the well, clean the area, and dispose of materials in accordance with the procedures outlined in Section 8 below.

7.2 Rotasonic Drilling Methods

For sites with deep unconsolidated aquifers or challenging drilling conditions (e.g., presence of dense tills, caliche, cobbles), DPT drilling may not be feasible or cost effective due to limited production rates. In these cases, alternate drilling methods (e.g., rotasonic) are required. Rotasonic drilling produces soil cores that, for the most part, are relatively undisturbed, but note that when drilling in consolidated or finer-grained sediment the vibratory action during core barrel advancement may create secondary fractures or breaks.

1. Place LDPE plastic sheeting over core/sampling processing area to create a clean working surface, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity.

2. Clear the ground surface of brush, root mat, grass, leaves, or other debris prior to sampling.
3. Decontaminate all non-disposable sampling equipment/tooling that will or may come into direct contact with soil prior to first use. Disposable sampling equipment must be kept in sealed PFAS-free packaging until it is used.
4. Use stainless-steel hand auger to collect samples from 0 - 5 ft bgs surface interval, if applicable. All hand augering to collect soil samples will be completed by Arcadis personnel, not the drilling subcontractor. These samples can be collected either during other Utility Clearance activities (e.g., third party clearance) or immediately prior to drilling.
 - a. Hand auger soil sample should be collected manually from the hand auger bucket (using stainless steel scoop, spatula, or trowel as necessary) and placed directly into the sample jar. The sample should not contact the ground or LDPE sheeting.
 - b. If sampling by hand auger, after collecting sample from the surface or shallowest depth interval examine the stability of the soil in the boring sidewalls. If sidewalls appear to be at risk of collapsing into the borehole insert a length of polyvinyl chloride (PVC) pipe into the boring to maintain the opening and prevent collapse prior to augering to the next deeper sampling interval.
5. During rotosonic drilling, drillers extract soil core bags after each drilling run, place the core bag onto LDPE sheeting, and cut open bags so Arcadis personnel can perform characterization and sampling. Arcadis personnel should confirm with drilling subcontractor that core bags are constructed of PFAS-free material. Drillers must not touch soil inside of bags during this process. Arcadis personnel decontaminate cutter between uses (see below).
6. Don a new set of nitrile gloves prior to collecting each sample. Do not use gloved hands to handle papers, pens, clothes, etc., before collecting samples. Do not touch outside of sample bag with gloved hands.
7. During sampling, characterize soils in accordance with P-04 TGI - Soil Description (Arcadis 2018b). Record descriptions in the field notes, boring logs, and/or personal digital assistant (PDA). It is also beneficial to photo document the samples. It should be noted that PDA logs must be electronically backed up and transferred to a location accessible to other project team members as soon as feasible to retain and protect the field data.
8. Collect sample volumes from recovered soil cores using a clean stainless-steel trowel and place in clean, labeled bottles supplied by the laboratory for the required analyses (see sample container list in PQAPP Worksheets #19&30). Make sure caps remain on PFAS sample bottles until immediately prior to filling. Caps must remain in the hand of the sampler until replacing on the bottle.
9. Once the sample has been placed in the bottle, and the bottle cap has been completely tightened, label the sample with sample identification number, date, and time of collection. Labels must be completed only after the caps have been placed back on each bottle. (See P-01 QP#3.06 Field Activities documentation for sample label information).
10. Collect QC samples at frequency specified in PQAPP Worksheet #20. QC sample locations to be selected based on consultation with Arcadis RL.
11. Place soil sample bottles in a sealed Ziploc® bag, and then into sample coolers. Store PFAS samples in separate cooler from other samples.
12. Record the label information and time of sampling in the field notes and sampling forms.

13. Fill out laboratory COC and check against the labels on the sample bottles progressively after each sample is collected.
14. Abandon all soil borings to grade as specified in the QAPP Addendum upon completion and before moving to the next boring location. **If single-interval groundwater sample is required, see Section 7.2.1 for procedure. If well is to be installed, see Section 7.2.2 for well construction procedure.**
15. Mark boring location with wooden stake that identifies boring ID for subsequent surveying, as necessary.
16. Manage investigation-derived-waste (IDW) as specified in site-specific work plan.
17. If samples are not shipped the same day as collected, add fresh ice to sample coolers at the end of the day to maintain the temperature between 0 and 6°C. Place ice in sealed polyethylene bags (Ziplock). Do not use blue ice. See QAPP worksheet #19 and 30 for sample containers, preservation and hold times. Sample coolers must remain in the possession of the sampling team at all times or secured under lock and key until shipment to the laboratory.

7.2.1 Rotosonic Single-Interval Groundwater Sampling

Groundwater profilers can be used to collect single-interval groundwater samples at a desired sample interval, commonly biased towards transport zones determined from soil lithological core, or from the first encountered groundwater. If multi-interval VAP sampling is required, refer to Arcadis TGI for Vertical Aquifer Profiling for PFAS Analysis (Arcadis 2019).

The configuration of individual samplers varies based on their manufacturer and drilling contractor (e.g., Cascade Packer Isolation Groundwater Profiler, Geoprobe® SP-60 Packer Sampler). The overall strategy of rotosonic drilling sampling is consistent with DPT sampling; however, drilling with sonic or some rotary methods requires the introduction of drilling water that can potentially affect the integrity of the groundwater sample. If drilling water is used, a source blank sample will be collected prior to the start of work. If state or local regulations allow, source water can be spiked with non-toxic fluorescent dyes per Arcadis SOP for use of visible tracer in drilling fluid to obtain representative groundwater samples during drilling (Arcadis 2010).

Rotosonic single-interval groundwater sampling will be performed using dual-tube casing. Packer Isolation Groundwater Profilers will be used to conduct groundwater sampling. The biggest advantage of this device is that the groundwater sampling depth interval can be determined based on lithological logs obtained from the same borehole since hydraulic profiling tool is not deployable via rotosonic drilling methods.

The following steps will be followed:

1. Ensure 4-gas meter, YSI 6-Series multi-parameter water quality probe, and turbidity meter are calibrated each morning (see QAPP worksheet #22 and P-09 Calibration and Control of Measuring and Test Equipment in PQAPP Appendix A). Document calibration results on equipment calibration log.
2. Advance dual-tube sonic tooling casing to target depth interval in accordance with **Section 7.2**.
3. Retrieve the soil core and the inner sonic core barrel. Characterize soils in accordance with Arcadis TGI for Soil Descriptions (Arcadis 2018b).

4. Insert the stainless-steel screen and packer assembly (e.g., Cascade Packer Isolation Groundwater Profiler, Geoprobe® SP60 Packer Sampler) to the base of the sonic casing.
5. Extract the outer sonic casing to expose the screen to the formation.
6. Inflate the packer to isolate the screened interval from any water that might be above the packer in the sonic casing.
7. Place LDPE plastic sheeting adjacent to the sample port for use as a clean work area, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity. Do not allow vehicle exhaust to point towards the sample point.
8. Don a new set of nitrile gloves, connect tubing to sampling pump and flow-through cell, and slowly lower tubing and/or pump into well. If possible, use two field personnel to insert tubing/pump into well to avoid contact with surrounding ground surface or other materials that could cause cross-contamination. Insert tubing (peristaltic pump, if depth to water <25 ft bgs), or pump intake (bladder pump, if depth to water >25 ft bgs) at the approximate mid-depth of the sampler screen interval.
9. Purge well until water is visually clear of sediment, or for a maximum of 20 minutes before collecting GW samples.
 - o Note: for low-permeability formations, collect a grab sample from the screen point sampling device and/or saturated soil sample from the soil core. Leave temporary well casing and screen in the borehole to allow for overnight recovery of groundwater for follow-up sampling the next day.
10. Don a new set of nitrile gloves prior to collecting the groundwater sample and each QC sample. Do not use gloved hands to handle items (e.g., papers, pens, clothes, equipment) before collecting samples.
11. Fill sample bottles using labeled HDPE bottles that are supplied by laboratory only. Make sure that the cap remains on the bottle until immediately prior to sample collection and gets placed back on the bottle immediately after sample collection. Do not place the cap on any surface; keep in hand opposite of sample collection and do not touch the inside of the cap.
12. If high concentrations of PFASs related to Class B Firefighting foams are expected in a groundwater sample (as specified in the QAPP Addendum), collect and shake a small portion of the sample (approximately 10-25 mL) on site. If foaming is observed, document the foaming on the sample log and on the COC to notify laboratory personnel. The “shaker test” vial can then be disposed of as IDW as specified in Section 8.
13. Collect QC samples at frequency specified in PQAPP Worksheet #20. QC sample locations to be selected based on consultation with Arcadis RL;
14. Place filled sample bottles in a sealed (Ziploc®) bag, record any label information that was not pre-filled out (e.g., sample time). Record the label information and time of sampling in the field notes and sampling forms. Place samples into sample coolers. Store PFAS samples in separate cooler from any other types of samples.
15. Fill out laboratory COC and check against the labels on the sample bottles progressively after each sample is collected.
16. Deflate the packer.
17. Pull the Packer Isolation Groundwater Profiler back up and decontaminate per instructions in Section 10.

7.2.2 Rotosonic Monitoring Well Construction

1. If it is necessary to install a monitor well into a permeable zone below a confining layer, particularly if the deeper zone is believed to have water quality that differs significantly from the zone above the confining layer, then a telescopic well construction will be considered. In this case, the borehole is advanced approximately 3 to 5 feet into the top of the confining layer, and a permanent casing (typically PVC, black steel or stainless steel) is installed into the socket drilled into the top of the confining layer. The casing is then grouted in place. Grout should be mixed with PFAS-free water (verified by laboratory analysis of source water). The preferred methods of grouting telescoping casings include: pressure-injection grouting using an inflatable packer installed temporarily into the base of the casing, such that grout is injected out the bottom of the casing until it is observed at ground surface outside the casing; displacement-method grouting (also known as the Halliburton method), which entails filling the casing with grout and displacing the grout out the bottom of the casing by pushing a drillable plug, typically made of wood to the bottom of the casing, following by tremie grouting the remainder of the annulus outside the casing; or tremie grouting the annulus surrounding the casing using a tremie pipe installed to the base of the borehole. In all three cases, the casing is grouted to the ground surface, and the grout is allowed to set prior to drilling deeper through the casing. Site-specific criteria and work plans should be created for the completion of non-standard monitoring wells, including telescopic wells.
2. Before installing a screened well, it is important to confirm that the borehole has been advanced into the targeted saturated zone. This is particularly important for wells installed to monitor the water table and/or the shallow saturated zone, as the capillary fringe may cause soils above the water table to appear saturated. If one or more previously installed monitoring wells exist nearby, use the depth to water at such well(s) to estimate the water-table depth at the new borehole location.

To verify that the borehole has been advanced into the saturated zone, it is necessary to measure the water level in the borehole. For boreholes drilled using water (e.g., Rotosonic), monitor the water level in the borehole as it re-equilibrates to the static level. In low-permeability units like clay, fine-grained glacial tills, shale and other bedrock formations, it may be necessary to wait overnight to allow the water level to equilibrate. Document depth to water in the borehole on the appropriate field forms and field notebook. If there are questions concerning the depth of the well/screen interval, consult with the project technical lead or PM prior to finalizing well depth/screen interval. To the extent practicable, ensure that the depth of the well below the apparent water table is deep enough so that the installed well can monitor groundwater year-round, accounting for seasonal water-table fluctuations. When in doubt, err on the side of slightly deeper well installation.

3. Upon completing the borehole to the desired depth, if a screened well construction is desired, install the monitoring well by lowering the screen and solid PVC risers through the augers or casing. Monitoring wells typically will be constructed of 2-inch-diameter (although sometimes 4-inch), flush-threaded PVC or stainless steel slotted or wire wrapped well screen and blank riser

casing. Smaller diameters may be used if multiple wells are to be installed in a single borehole. The screen length will be specified in the Work Plan (or equivalent) based on regulatory requirements and specific monitoring objectives. Monitoring well screens should be limited to 5 to 10 feet long. The screen length will depend on the purpose for the well and the objectives of the groundwater investigation and will (in most cases) be determined prior to the field mobilization.

The slot size and filter pack gradation should be predetermined in the Work Plan (or equivalent) based on site-specific grain-size analysis (sieve analysis) or other geologic considerations or monitoring objectives. Typically, slot sizes for monitoring wells will range from 0.010 inches to 0.020 inches while the filter pack will be 20-40, Morie No. 0, or equivalent. In very fine-grained formations where sample turbidity needs to be minimized, it may be preferred to use a 0.006-inch slot size and 30-65, Morie No. 00, or equivalent filter pack. Alternatively, where monitoring wells are installed in coarse-grained deposits and higher well yield is required, a 0.020-inch slot size and 10-20, Morie No. 1, or equivalent filter pack may be preferred. If the screen slot size and filter pack have not been based on site-specific grain-size analysis, consider collecting soil samples during well installation so future wells can be properly designed.

Alternately, a monitoring well assembly with a pre-packed screen can be installed. The monitoring well assembly (i.e., regular PVC or pre-packed) should be discussed and decided prior to beginning field work and specified in the QAPP addendum. Pre-packed filters should be verified as PFAS-free prior to use.

A blank riser will extend from the top of the screen to approximately 2.5 feet above grade or, if necessary, just below grade where conditions warrant a flush-mounted monitoring well. For wells greater than 50 feet deep, centralizers may be desired to assist in centering the monitoring well in the borehole during construction.

4. When the monitoring well assembly has been set in place, place a washed silica filter pack in the annular space from the bottom of the boring to a height of 1 to 2 feet above the top of the well screen (following specifications in the Work Plan) using a tremie. The filter pack is placed, and drilling equipment extracted in increments until the top of the sand pack is at the appropriate depth. Verify that the expected volume of filter pack matches with the actual amount installed. There can be differences due to irregularities in the borehole. Washout of the borehole will result in the need for greater than calculated well materials. If a difference of more than 10% is noted, consult with the project technical team. The filter pack will be consistent with the screen slot size and the soil particle size in the screened interval, as specified in the Work Plan (or equivalent). The well should be gently surged to prevent filter pack material bridging and to settle the filter pack prior to well seal installation.
5. A hydrated bentonite seal (a minimum of 2 feet thick) will then be placed in the annular space above the sand pack (alternatively, in some cases a fine sand seal may be installed instead of bentonite—follow the specifications in the Work Plan). Use of a tremie pipe is not required for placement of the bentonite seal (though may be required if a well is very deep and borehole

bridging is reasonably anticipated). However, bentonite should be poured into the annular space slowly enough to ensure borehole bridging does not occur. If non-hydrated bentonite is used, the bentonite should be permitted to hydrate in place for a minimum of 30 minutes before proceeding. *No coated bentonite pellets will be used in monitoring well drilling or construction.* PFAS-free water (verified by laboratory analysis of source water) should be added to hydrate the bentonite if the seal is above the water table. Continuously monitor the placement of the sand pack and bentonite with a weighted tape measure.

6. During the extraction of the augers or casing, a cement/bentonite or neat cement grout will be placed in the annular space from the bentonite seal to a depth approximately 2 ft bgs or as specified in the Work Plan (or equivalent). As with the filter pack, it is recommended that seal material be placed with a tremie pipe. Ensure that seal materials are mixed at the proper ratios with PFAS-free water (verified by laboratory analysis of source water) following manufacturer's recommendations.
7. Install the monitoring well completion as specified Work Plan (or equivalent). Typical completions are a locking, steel protective casing (extended at least 1.5 feet below grade and 2 feet above grade) over the riser casing and secure with a neat cement seal. Alternatively, for flush-mount completions, place a steel curb box with a bolt-down lid over the riser casing and secure with a neat cement seal. In either case, the cement seal will extend approximately 1.5 to 2.0 feet below grade and laterally at least 1 foot in all directions from the protective casing and should slope gently away to promote drainage away from the well.
8. Monitoring wells should be labeled using indelible ink or paint with the appropriate designation on both the inner and outer well casings or inside of the curb box lid. Use caution when labeling the well as paint or indelible ink could potentially contain PFAS materials.
9. When an above-grade completion is used, the riser will be sealed using an expandable locking plug and the top of the well will be vented by drilling a small-diameter (1/8 inch) hole near the top of the well casing or through the locking plug, or by cutting a vertical slot in the top of the well casing. When a flush-mount installation is used, the riser will be sealed using an unvented, expandable locking plug.
10. During well installation, record construction details and actual measurements relayed by the drilling contractor and tabulate materials used (e.g., screen and riser footages; bags of bentonite, cement, and sand) in the field notebook as well as appropriate field forms.
11. After completing the well installation, lock the well, clean the area, and dispose of materials in accordance with the procedures outlined in Section 8 below.

7.3 Hollow Stem Auger Drilling Methods

For some sites, direct push drilling may not be feasible and rotasonic drilling may not be cost effective. In these cases, hollow stem auger (HSA) drilling may be required. HSA drilling procedures are similar to

rotasonic drilling, but HSA is not well suited to very coarse materials (e.g. cobbles and boulder) and it produces disturbed soil samples.

1. Place LDPE plastic sheeting over core/sampling processing area to create a clean working surface, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity.
2. Clear the ground surface of brush, root mat, grass, leaves, or other debris prior to sampling.
3. Decontaminate all non-disposable sampling equipment/tooling that will or may come into direct contact with soil prior to first use. Disposable sampling equipment must be kept in sealed PFAS-free packaging until it is used.
4. Use stainless-steel hand auger to collect samples from 0 - 5 ft bgs surface interval, if applicable. All hand augering to collect soil samples will be completed by Arcadis personnel, not the drilling subcontractor. These samples can be collected either during other Utility Clearance activities (e.g., third party clearance) or immediately prior to drilling.
 - a. Hand auger soil sample should be collected manually from the hand auger bucket (using stainless steel scoop, spatula, or trowel as necessary) and placed directly into the sample jar. The sample should not contact the ground or LDPE sheeting.
 - b. If sampling by hand auger, after collecting sample from the surface or shallowest depth interval examine the stability of the soil in the boring sidewalls. If sidewalls appear to be at risk of collapsing into the borehole insert a length of polyvinyl chloride (PVC) pipe into the boring to maintain the opening and prevent collapse prior to augering to the next deeper sampling interval.
5. During HSA drilling, soil cuttings will accumulate around the outside of the borehole. Periodically the drillers will shovel cuttings into containers for disposal. Arcadis personnel can also perform characterization and sampling directly from these cuttings.
6. Drillers must not touch soil that will be collected for sampling. Arcadis personnel will use a stainless-steel spatula, spoon, or trowel to collect soil direct from the cuttings pile at the desired sample depth. Arcadis personnel decontaminate spatula, spoon, or trowel between uses in accordance with Section 10.
7. Don a new set of nitrile gloves prior to collecting each sample. Do not use gloved hands to handle papers, pens, clothes, etc., before collecting samples. Do not touch outside of sample bag with gloved hands.
8. During sampling, characterize soils in accordance with P-04 TGI - Soil Description (Arcadis 2018b). Record descriptions in the field notes, boring logs, and/or personal digital assistant (PDA). It is also beneficial to photo document the samples. It should be noted that PDA logs must be electronically backed up and transferred to a location accessible to other project team members as soon as feasible to retain and protect the field data.
9. Collect sample volumes from cuttings pile using a clean stainless-steel trowel and place in clean, labeled bottles supplied by the laboratory for the required analyses (see sample container list in PQAPP Worksheets #19&30). Make sure caps remain on PFAS sample bottles until immediately prior to filling. Caps must remain in the hand of the sampler until replacing on the bottle.
10. Once the sample has been placed in the bottle, and the bottle cap has been completely tightened, label the sample with sample identification number, date, and time of collection. Labels must be completed only after the caps have been placed back on each bottle. (See P-01 QP#3.06 Field Activities documentation for sample label information).

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11. Collect QC samples at frequency specified in PQAPP Worksheet #20. QC sample locations to be selected based on consultation with Arcadis RL.
12. Place soil sample bottles in a sealed Ziploc® bag, and then into sample coolers. Store PFAS samples in separate cooler from other samples.
13. Record the label information and time of sampling in the field notes and sampling forms.
14. Fill out laboratory COC and check against the labels on the sample bottles progressively after each sample is collected.
15. Abandon all soil borings to grade as specified in the QAPP Addendum upon completion and before moving to the next boring location. **If single-interval groundwater sample is required, see Section 7.3.1 for procedure. If well is to be installed, see Section 7.3.2 for well construction procedure.**
16. Mark boring location with wooden stake that identifies boring ID for subsequent surveying, as necessary.
17. Manage investigation-derived-waste (IDW) as specified in site-specific work plan.
18. If samples are not shipped the same day as collected, add fresh ice to sample coolers at the end of the day to maintain the temperature between 0 and 6°C. Place ice in sealed polyethylene bags (Ziplock). Do not use blue ice. See QAPP worksheet #19 and 30 for sample containers, preservation and hold times. Sample coolers must remain in the possession of the sampling team at all times or secured under lock and key until shipment to the laboratory.

7.3.1 Hollow Stem Auger Single-Interval Groundwater Sampling

Unlike direct push and roto sonic methods, there are limited single-interval groundwater sampling devices for HSA drilling. Commonly, groundwater samples will be collected with pre-packed PFAS-free temporary PVC well screen and PVC riser. This method is most appropriate for sampling the first encountered groundwater. Because soil samples are disturbed, it is often not possible to target specific intervals based on transport properties.

The following steps will be followed to collect the groundwater sample:

1. Ensure 4-gas meter, YSI 6-Series multi-parameter water quality probe, and turbidity meter are calibrated each morning (see QAPP worksheet #22 and P-09 Calibration and Control of Measuring and Test Equipment in PQAPP Appendix A). Document calibration results on equipment calibration log.
2. Advance augers until groundwater is encountered.
3. Arcadis will continuously characterize soil and/or rock cuttings.
4. When groundwater is encountered, the water level in the borehole will be allowed to reach static condition. If a grab groundwater sample is required, a pre-packed PFAS-free temporary PVC well screen will be lowered to the desired sample depth. Then, the augers will be raised to above the temporary well screen to expose the screen.
5. Place LDPE plastic sheeting adjacent to the sample port for use as a clean work area, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity. Do not allow vehicle exhaust to point towards the sample point.

6. Don a new set of nitrile gloves, connect tubing to sampling pump and flow-through cell and slowly lower tubing and/or pump into well. If possible, use two field personnel to insert tubing/pump into well to avoid contact with surrounding ground surface or other materials that could cause cross-contamination. Insert tubing (peristaltic pump, if depth to water <25 ft bgs), or pump intake (small-diameter bladder pump, if depth to water >25 ft bgs) at the approximate mid-depth of the sampler screen interval.
 - a. Alternately, a Waterra-type inertial pump can be used to retrieve the water sample. If the formation has low-permeability and enough water is not anticipated in the tooling to allow purging of water, a stainless-steel bailer may be considered (after consulting with Arcadis RL).
7. Purge until water is visually clear of sediment, or for a maximum of 20 minutes before collecting GW samples.
 - a. Note: for low-permeability formations, collect a grab sample from the screen point sampling device and/or saturated soil sample from the soil core. Leave temporary well casing and screen in the borehole to allow for overnight recovery of groundwater for follow-up sampling the next day.
8. Don a new set of nitrile gloves prior to collecting the groundwater sample and each QC sample. Do not use gloved hands to handle items (e.g., papers, pens, clothes, equipment) before collecting samples.
9. Fill sample bottles using labelled HDPE bottles that are supplied by laboratory only. Make sure that the cap remains on the bottle until immediately prior to sample collection and gets placed back on the bottle immediately after sample collection. Do not place the cap on any surface; keep in hand opposite of sample collection and do not touch the inside of the cap.
10. If high concentrations of PFASs related to Class B Firefighting foams are expected in a groundwater sample (as specified in the QAPP Addendum), collect and shake a small portion of the sample (approximately 10-25 mL) on site. If foaming is observed, document the foaming on the sample log and on the COC to notify laboratory personnel. The “shaker test” vial can then be disposed of as IDW as specified in Section 8.
11. Collect QC samples at frequency specified in PQAPP Worksheet #20. QC sample locations to be selected based on consultation with Arcadis RL.
12. Place filled sample bottles in a sealed (Ziploc®) bag, record any label information that was not pre-filled out (e.g., sample time). Record the label information and time of sampling in the field notes and sampling forms. Place samples into sample coolers. Store PFAS samples in separate cooler from any other types of samples.
13. Fill out laboratory COC and check against the labels on the sample bottles progressively after each sample is collected.
14. Retrieve the temporary well screen dispose of as specified in Section 8.

7.3.2 Hollow Stem Auger Monitoring Well Construction

HAS well construction methods are the same as when using rotasonic drilling methods and are outlined below.

1. If it is necessary to install a monitor well into a permeable zone below a confining layer, particularly if the deeper zone is believed to have water quality that differs significantly from the

zone above the confining layer, then a telescopic well construction will be considered. In this case, the borehole is advanced approximately 3 to 5 feet into the top of the confining layer, and a permanent casing (typically PVC, black steel or stainless steel) is installed into the socket drilled into the top of the confining layer. The casing is then grouted in place. Grout should be mixed with PFAS-free water (verified by laboratory analysis of source water). The preferred methods of grouting telescoping casings include: pressure-injection grouting using an inflatable packer installed temporarily into the base of the casing, such that grout is injected out the bottom of the casing until it is observed at ground surface outside the casing; displacement-method grouting (also known as the Halliburton method), which entails filling the casing with grout and displacing the grout out the bottom of the casing by pushing a drillable plug, typically made of wood to the bottom of the casing, following by tremie grouting the remainder of the annulus outside the casing; or tremie grouting the annulus surrounding the casing using a tremie pipe installed to the base of the borehole. In all three cases, the casing is grouted to the ground surface, and the grout is allowed to set prior to drilling deeper through the casing. Site-specific criteria and work plans should be created for the completion of non-standard monitoring wells, including telescopic wells.

2. Before installing a screened well, it is important to confirm that the borehole has been advanced into the targeted saturated zone. This is particularly important for wells installed to monitor the water table and/or the shallow saturated zone, as the capillary fringe may cause soils above the water table to appear saturated. If one or more previously installed monitoring wells exist nearby, use the depth to water at such well(s) to estimate the water-table depth at the new borehole location.

To verify that the borehole has been advanced into the saturated zone, it is necessary to measure the water level in the borehole. For boreholes drilled using water (e.g., Rotosonic), monitor the water level in the borehole as it re-equilibrates to the static level. In low-permeability units like clay, fine-grained glacial tills, shale and other bedrock formations, it may be necessary to wait overnight to allow the water level to equilibrate. Document depth to water in the borehole on the appropriate field forms and field notebook. If there are questions concerning the depth of the well/screen interval, consult with the project technical lead or PM prior to finalizing well depth/screen interval. To the extent practicable, ensure that the depth of the well below the apparent water table is deep enough so that the installed well can monitor groundwater year-round, accounting for seasonal water-table fluctuations. When in doubt, err on the side of slightly deeper well installation.

3. Upon completing the borehole to the desired depth, if a screened well construction is desired, install the monitoring well by lowering the screen and solid PVC risers through the augers or casing. Monitoring wells typically will be constructed of 2-inch-diameter (although sometimes 4-inch), flush-threaded PVC or stainless steel slotted or wire wrapped well screen and blank riser casing. Smaller diameters may be used if multiple wells are to be installed in a single borehole. The screen length will be specified in the Work Plan (or equivalent) based on regulatory requirements and specific monitoring objectives. Monitoring well screens should be limited to 5 to

10 feet long. The screen length will depend on the purpose for the well and the objectives of the groundwater investigation and will (in most cases) be determined prior to the field mobilization.

The slot size and filter pack gradation should be predetermined in the Work Plan (or equivalent) based on site-specific grain-size analysis (sieve analysis) or other geologic considerations or monitoring objectives. Typically, slot sizes for monitoring wells will range from 0.010 inches to 0.020 inches while the filter pack will be 20-40, Morie No. 0, or equivalent. In very fine-grained formations where sample turbidity needs to be minimized, it may be preferred to use a 0.006-inch slot size and 30-65, Morie No. 00, or equivalent filter pack. Alternatively, where monitoring wells are installed in coarse-grained deposits and higher well yield is required, a 0.020-inch slot size and 10-20, Morie No. 1, or equivalent filter pack may be preferred. If the screen slot size and filter pack have not been based on site-specific grain-size analysis, consider collecting soil samples during well installation so future wells can be properly designed.

Alternately, a monitoring well assembly with a pre-packed screen can be installed. The monitoring well assembly (i.e., regular PVC or pre-packed) should be discussed and decided prior to beginning field work and specified in the QAPP addendum. Pre-packed filters should be verified as PFAS-free prior to use.

A blank riser will extend from the top of the screen to approximately 2.5 feet above grade or, if necessary, just below grade where conditions warrant a flush-mounted monitoring well. For wells greater than 50 feet deep, centralizers may be desired to assist in centering the monitoring well in the borehole during construction.

4. When the monitoring well assembly has been set in place, place a washed silica filter pack in the annular space from the bottom of the boring to a height of 1 to 2 feet above the top of the well screen (following specifications in the Work Plan) using a tremie. The filter pack is placed, and drilling equipment extracted in increments until the top of the sand pack is at the appropriate depth. Verify that the expected volume of filter pack matches with the actual amount installed. There can be differences due to irregularities in the borehole. Washout of the borehole will result in the need for greater than calculated well materials. If a difference of more than 10% is noted, consult with the project technical team. The filter pack will be consistent with the screen slot size and the soil particle size in the screened interval, as specified in the Work Plan (or equivalent). The well should be gently surged to prevent filter pack material bridging and to settle the filter pack prior to well seal installation.
5. A hydrated bentonite seal (a minimum of 2 feet thick) will then be placed in the annular space above the sand pack (alternatively, in some cases a fine sand seal may be installed instead of bentonite—follow the specifications in the Work Plan). Use of a tremie pipe is not required for placement of the bentonite seal (though may be required if a well is very deep and borehole bridging is reasonably anticipated). However, bentonite should be poured into the annular space slowly enough to ensure borehole bridging does not occur. If non-hydrated bentonite is used, the bentonite should be permitted to hydrate in place for a minimum of 30 minutes before proceeding.

No coated bentonite pellets will be used in monitoring well drilling or construction. PFAS-free water (verified by laboratory analysis of source water) should be added to hydrate the bentonite if the seal is above the water table. Continuously monitor the placement of the sand pack and bentonite with a weighted tape measure.

6. During the extraction of the augers or casing, a cement/bentonite or neat cement grout will be placed in the annular space from the bentonite seal to a depth approximately 2 ft bgs or as specified in the Work Plan (or equivalent). As with the filter pack, it is recommended that seal material be placed with a tremie pipe. Ensure that seal materials are mixed at the proper ratios with PFAS-free water (verified by laboratory analysis of source water) following manufacturer's recommendations.
7. Install the monitoring well completion as specified Work Plan (or equivalent). Typical completions are a locking, steel protective casing (extended at least 1.5 feet below grade and 2 feet above grade) over the riser casing and secure with a neat cement seal. Alternatively, for flush-mount completions, place a steel curb box with a bolt-down lid over the riser casing and secure with a neat cement seal. In either case, the cement seal will extend approximately 1.5 to 2.0 feet below grade and laterally at least 1 foot in all directions from the protective casing and should slope gently away to promote drainage away from the well.
8. Monitoring wells should be labeled using indelible ink or paint with the appropriate designation on both the inner and outer well casings or inside of the curb box lid. Use caution when labeling the well as paint or indelible ink could potentially contain PFAS materials.
9. When an above-grade completion is used, the riser will be sealed using an expandable locking plug and the top of the well will be vented by drilling a small-diameter (1/8 inch) hole near the top of the well casing or through the locking plug, or by cutting a vertical slot in the top of the well casing. When a flush-mount installation is used, the riser will be sealed using an unvented, expandable locking plug.
10. During well installation, record construction details and actual measurements relayed by the drilling contractor and tabulate materials used (e.g., screen and riser footages; bags of bentonite, cement, and sand) in the field notebook as well as appropriate field forms.
11. After completing the well installation, lock the well, clean the area, and dispose of materials in accordance with the procedures outlined in Section 8 below.

7.4 Air Rotary Drilling Methods

For sites that require bedrock investigations but no collection of undisturbed rock cores, air rotary drilling may be required. Air rotary drilling procedures are similar to HSA. It produces disturbed soil and rock samples that are lifted to the surface by blowing compressed air into the borehole. Air rotary drilling is not well suited for situations that require high-resolution bedrock logging, but can be an effective way to generally characterize bedrock lithology and sample bedrock groundwater.

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1. Place LDPE plastic sheeting over sampling processing area to create a clean working surface, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity.
2. Clear the ground surface of brush, root mat, grass, leaves, or other debris prior to sampling.
3. Decontaminate all non-disposable sampling equipment/tooling that will or may to come into direct contact with soil prior to first use. Disposable sampling equipment must be kept in sealed PFAS-free packaging until it is used.
4. Use stainless-steel hand auger to collect samples from 0 - 5 ft bgs surface interval, if applicable. All hand augering to collect soil samples will be completed by Arcadis personnel, not the drilling subcontractor. These samples can be collected either during other Utility Clearance activities (e.g., third party clearance) or immediately prior to drilling.
 - a. Hand auger soil sample should be collected manually from the hand auger bucket (using stainless steel scoop, spatula, or trowel as necessary) and placed directly into the sample jar. The sample should not contact the ground or LDPE sheeting.
 - b. If sampling by hand auger, after collecting sample from the surface or shallowest depth interval examine the stability of the soil in the boring sidewalls. If sidewalls appear to be at risk of collapsing into the borehole insert a length of polyvinyl chloride (PVC) pipe into the boring to maintain the opening and prevent collapse prior to augering to the next deeper sampling interval.
5. During air rotary drilling, soil and rock cuttings will accumulate around the outside of the borehole. Periodically the drillers will shovel cuttings into containers for disposal. Arcadis personnel can also perform characterization and sampling directly from these cuttings.
6. Drillers must not touch soil that will be collected for sampling. Arcadis personnel will use a stainless-steel spatula, spoon, or trowel to collect soil direct from the cuttings pile at the desired sample depth. Arcadis personnel decontaminate spatula, spoon, or trowel between uses in accordance with Section 10.
7. Don a new set of nitrile gloves prior to collecting each sample. Do not use gloved hands to handle papers, pens, clothes, etc., before collecting samples. Do not touch outside of sample bag with gloved hands.
8. During sampling, characterize soils in accordance with P-04 TGI - Soil Description (Arcadis 2018b). Record descriptions in the field notes, boring logs, and/or personal digital assistant (PDA). It is also beneficial to photo document the samples. It should be noted that PDA logs must be electronically backed up and transferred to a location accessible to other project team members as soon as feasible to retain and protect the field data.
9. Collect sample volumes from cuttings pile using a clean stainless-steel trowel and place in clean, labeled bottles supplied by the laboratory for the required analyses (see sample container list in PQAPP Worksheets #19&30). Make sure caps remain on PFAS sample bottles until immediately prior to filling. Caps must remain in the hand of the sampler until replacing on the bottle.
10. Once the sample has been placed in the bottle, and the bottle cap has been completely tightened, label the sample with sample identification number, date, and time of collection. Labels must be completed only after the caps have been placed back on each bottle. (See P-01 QP#3.06 Field Activities documentation for sample label information).
11. Collect QC samples at frequency specified in PQAPP Worksheet #20. QC sample locations to be selected based on consultation with Arcadis RL.

12. Place soil sample bottles in a sealed Ziploc® bag, and then into sample coolers. Store PFAS samples in separate cooler from other samples.
13. Record the label information and time of sampling in the field notes and sampling forms.
14. Fill out laboratory COC and check against the labels on the sample bottles progressively after each sample is collected.
15. Abandon all soil borings to grade as specified in the QAPP Addendum upon completion and before moving to the next boring location. **If single-interval groundwater sample is required, see Section 7.4.1 for procedure. If well is to be installed, see Section 7.4.2 for well construction procedure.**
16. Mark boring location with wooden stake that identifies boring ID for subsequent surveying, as necessary.
17. Manage investigation-derived-waste (IDW) as specified in site-specific work plan.
18. If samples are not shipped the same day as collected, add fresh ice to sample coolers at the end of the day to maintain the temperature between 0 and 6°C. Place ice in sealed polyethylene bags (Ziplock). Do not use blue ice. See QAPP worksheet #19 and 30 for sample containers, preservation and hold times. Sample coolers must remain in the possession of the sampling team at all times or secured under lock and key until shipment to the laboratory.

7.4.1 Air Rotary Single-Interval Groundwater Sampling

Similar to HSA drilling methods, there are limited single-interval groundwater sampling devices for air rotary drilling. Commonly, groundwater samples will be collected with pre-packed PFAS-free temporary PVC well screen and PVC riser. This method is most appropriate for sampling the first encountered groundwater when it is expected to occur in bedrock. Because soil and rock samples are disturbed, it is often not possible to target specific intervals based on transport properties. Unlike single-interval groundwater sampling with DPT, rotosonic, and HSA boreholes, air rotary drill tooling (a.k.a. drill bit stem or drill bit pipe) is a smaller diameter than the drill bit advancing the borehole. Additionally, the central opening of the drill bit is generally too narrow to accommodate a pre-packed PFAS-free temporary PVC well screen. Therefore, single-interval groundwater samples will be collected by inserting the pump through the annular space between the borehole wall and the air rotary drill tooling.

The following steps will be followed to collect the groundwater sample:

1. Ensure 4-gas meter, YSI 6-Series multi-parameter water quality probe, and turbidity meter are calibrated each morning (see QAPP worksheet #22 and P-09 Calibration and Control of Measuring and Test Equipment in PQAPP Appendix A). Document calibration results on equipment calibration log.
2. Advance rotary bit until groundwater is encountered.
3. Arcadis will continuously characterize soil and/or rock cuttings.
4. When groundwater is encountered, the water level in the borehole will be allowed to reach static condition. If a grab groundwater sample is required, a pre-packed PFAS-free temporary PVC well screen will be lowered through the annular space between the borehole wall and the air rotary drill tooling to the desired sample depth.
5. Place LDPE plastic sheeting adjacent to the sample port for use as a clean work area, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other

surface that could compromise sample integrity. Do not allow vehicle exhaust to point towards the sample point.

6. Don a new set of nitrile gloves, connect tubing to sampling pump and flow-through cell and slowly lower tubing and/or pump into well. If possible, use two field personnel to insert tubing/pump into well to avoid contact with surrounding ground surface or other materials that could cause cross-contamination. Insert tubing (peristaltic pump, if depth to water <25 ft bgs), or pump intake (small-diameter bladder pump, if depth to water >25 ft bgs) at the approximate mid-depth of the sampler screen interval.
 - a. Alternately, a Waterra-type inertial pump can be used to retrieve the water sample. If the formation has low-permeability and enough water is not anticipated in the tooling to allow purging of water, a stainless-steel bailer may be considered (after consulting with Arcadis RL).
7. Purge until water is visually clear of sediment, or for a maximum of 20 minutes before collecting GW samples.
 - a. Note: for low-permeability formations, collect a grab sample from the screen point sampling device and/or saturated soil sample from the soil core. Leave temporary well casing and screen in the borehole to allow for overnight recovery of groundwater for follow-up sampling the next day.
8. Don a new set of nitrile gloves prior to collecting the groundwater sample and each QC sample. Do not use gloved hands to handle items (e.g., papers, pens, clothes, equipment) before collecting samples.
9. Fill sample bottles using labelled HDPE bottles that are supplied by laboratory only. Make sure that the cap remains on the bottle until immediately prior to sample collection and gets placed back on the bottle immediately after sample collection. Do not place the cap on any surface; keep in hand opposite of sample collection and do not touch the inside of the cap.
10. If high concentrations of PFASs related to Class B Firefighting foams are expected in a groundwater sample (as specified in the QAPP Addendum), collect and shake a small portion of the sample (approximately 10-25 mL) on site. If foaming is observed, document the foaming on the sample log and on the COC to notify laboratory personnel. The “shaker test” vial can then be disposed of as IDW as specified in Section 8.
11. Collect QC samples at frequency specified in PQAPP Worksheet #20. QC sample locations to be selected based on consultation with Arcadis RL.
12. Place filled sample bottles in a sealed (Ziploc[®]) bag, record any label information that was not pre-filled out (e.g., sample time). Record the label information and time of sampling in the field notes and sampling forms. Place samples into sample coolers. Store PFAS samples in separate cooler from any other types of samples.
13. Fill out laboratory COC and check against the labels on the sample bottles progressively after each sample is collected.
14. Retrieve the temporary well screen dispose of as specified in Section 8.

7.4.2 Air Rotary Monitoring Well Construction

Air rotary well construction methods are the same as when using HSA drilling methods and are outlined below.

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1. If it is necessary to install a monitor well into a permeable zone below a confining layer, particularly if the deeper zone is believed to have water quality that differs significantly from the zone above the confining layer, then a telescopic well construction will be considered. In this case, the borehole is advanced approximately 3 to 5 feet into the top of the confining layer, and a permanent casing (typically PVC, black steel or stainless steel) is installed into the socket drilled into the top of the confining layer. The casing is then grouted in place. Grout should be mixed with PFAS-free water (verified by laboratory analysis of source water). The preferred methods of grouting telescoping casings include: pressure-injection grouting using an inflatable packer installed temporarily into the base of the casing, such that grout is injected out the bottom of the casing until it is observed at ground surface outside the casing; displacement-method grouting (also known as the Halliburton method), which entails filling the casing with grout and displacing the grout out the bottom of the casing by pushing a drillable plug, typically made of wood to the bottom of the casing, following by tremie grouting the remainder of the annulus outside the casing; or tremie grouting the annulus surrounding the casing using a tremie pipe installed to the base of the borehole. In all three cases, the casing is grouted to the ground surface, and the grout is allowed to set prior to drilling deeper through the casing. Site-specific criteria and work plans should be created for the completion of non-standard monitoring wells, including telescopic wells.
2. Before installing a screened well, it is important to confirm that the borehole has been advanced into the targeted saturated zone. This is particularly important for wells installed to monitor the water table and/or the shallow saturated zone, as the capillary fringe may cause soils above the water table to appear saturated. If one or more previously installed monitoring wells exist nearby, use the depth to water at such well(s) to estimate the water-table depth at the new borehole location.

To verify that the borehole has been advanced into the saturated zone, it is necessary to measure the water level in the borehole. For boreholes drilled using water (e.g., Rotosonic), monitor the water level in the borehole as it re-equilibrates to the static level. In low-permeability units like clay, fine-grained glacial tills, shale and other bedrock formations, it may be necessary to wait overnight to allow the water level to equilibrate. Document depth to water in the borehole on the appropriate field forms and field notebook. If there are questions concerning the depth of the well/screen interval, consult with the project technical lead or PM prior to finalizing well depth/screen interval. To the extent practicable, ensure that the depth of the well below the apparent water table is deep enough so that the installed well can monitor groundwater year-round, accounting for seasonal water-table fluctuations. When in doubt, err on the side of slightly deeper well installation.

3. Upon completing the borehole to the desired depth, if a screened well construction is desired, install the monitoring well by lowering the screen and solid PVC risers through the augers or casing. Monitoring wells typically will be constructed of 2-inch-diameter (although sometimes 4-inch), flush-threaded PVC or stainless steel slotted or wire wrapped well screen and blank riser casing. Smaller diameters may be used if multiple wells are to be installed in a single borehole. The screen length will be specified in the Work Plan (or equivalent) based on regulatory

requirements and specific monitoring objectives. Monitoring well screens should be limited to 5 to 10 feet long. The screen length will depend on the purpose for the well and the objectives of the groundwater investigation and will (in most cases) be determined prior to the field mobilization.

The slot size and filter pack gradation should be predetermined in the Work Plan (or equivalent) based on site-specific grain-size analysis (sieve analysis) or other geologic considerations or monitoring objectives. Typically, slot sizes for monitoring wells will range from 0.010 inches to 0.020 inches while the filter pack will be 20-40, Morie No. 0, or equivalent. In very fine-grained formations where sample turbidity needs to be minimized, it may be preferred to use a 0.006-inch slot size and 30-65, Morie No. 00, or equivalent filter pack. Alternatively, where monitoring wells are installed in coarse-grained deposits and higher well yield is required, a 0.020-inch slot size and 10-20, Morie No. 1, or equivalent filter pack may be preferred. If the screen slot size and filter pack have not been based on site-specific grain-size analysis, consider collecting soil samples during well installation so future wells can be properly designed.

Alternately, a monitoring well assembly with a pre-packed screen can be installed. The monitoring well assembly (i.e., regular PVC or pre-packed) should be discussed and decided prior to beginning field work and specified in the QAPP addendum. Pre-packed filters should be verified as PFAS-free prior to use.

A blank riser will extend from the top of the screen to approximately 2.5 feet above grade or, if necessary, just below grade where conditions warrant a flush-mounted monitoring well. For wells greater than 50 feet deep, centralizers may be desired to assist in centering the monitoring well in the borehole during construction.

4. When the monitoring well assembly has been set in place, place a washed silica filter pack in the annular space from the bottom of the boring to a height of 1 to 2 feet above the top of the well screen (following specifications in the Work Plan) using a tremie. The filter pack is placed, and drilling equipment extracted in increments until the top of the sand pack is at the appropriate depth. Verify that the expected volume of filter pack matches with the actual amount installed. There can be differences due to irregularities in the borehole. Washout of the borehole will result in the need for greater than calculated well materials. If a difference of more than 10% is noted, consult with the project technical team. The filter pack will be consistent with the screen slot size and the soil particle size in the screened interval, as specified in the Work Plan (or equivalent). The well should be gently surged to prevent filter pack material bridging and to settle the filter pack prior to well seal installation.
5. A hydrated bentonite seal (a minimum of 2 feet thick) will then be placed in the annular space above the sand pack (alternatively, in some cases a fine sand seal may be installed instead of bentonite—follow the specifications in the Work Plan). Use of a tremie pipe is not required for placement of the bentonite seal (though may be required if a well is very deep and borehole bridging is reasonably anticipated). However, bentonite should be poured into the annular space slowly enough to ensure borehole bridging does not occur. If non-hydrated bentonite is used, the

bentonite should be permitted to hydrate in place for a minimum of 30 minutes before proceeding. *No coated bentonite pellets will be used in monitoring well drilling or construction.* PFAS-free water (verified by laboratory analysis of source water) should be added to hydrate the bentonite if the seal is above the water table. Continuously monitor the placement of the sand pack and bentonite with a weighted tape measure.

6. During the extraction of the augers or casing, a cement/bentonite or neat cement grout will be placed in the annular space from the bentonite seal to a depth approximately 2 ft bgs or as specified in the Work Plan (or equivalent). As with the filter pack, it is recommended that seal material be placed with a tremie pipe. Ensure that seal materials are mixed at the proper ratios with PFAS-free water (verified by laboratory analysis of source water) following manufacturer's recommendations.
7. Install the monitoring well completion as specified Work Plan (or equivalent). Typical completions are a locking, steel protective casing (extended at least 1.5 feet below grade and 2 feet above grade) over the riser casing and secure with a neat cement seal. Alternatively, for flush-mount completions, place a steel curb box with a bolt-down lid over the riser casing and secure with a neat cement seal. In either case, the cement seal will extend approximately 1.5 to 2.0 feet below grade and laterally at least 1 foot in all directions from the protective casing and should slope gently away to promote drainage away from the well.
8. Monitoring wells should be labeled using indelible ink or paint with the appropriate designation on both the inner and outer well casings or inside of the curb box lid. Use caution when labeling the well as paint or indelible ink could potentially contain PFAS materials.
9. When an above-grade completion is used, the riser will be sealed using an expandable locking plug and the top of the well will be vented by drilling a small-diameter (1/8 inch) hole near the top of the well casing or through the locking plug, or by cutting a vertical slot in the top of the well casing. When a flush-mount installation is used, the riser will be sealed using an unvented, expandable locking plug.
10. During well installation, record construction details and actual measurements relayed by the drilling contractor and tabulate materials used (e.g., screen and riser footages; bags of bentonite, cement, and sand) in the field notebook as well as appropriate field forms.
11. After completing the well installation, lock the well, clean the area, and dispose of materials in accordance with the procedures outlined in Section 8 below.

8 WASTE MANAGEMENT

Investigation-derived waste (IDW) including soil cuttings, purge water, and decontamination water generated during cleaning procedures will be collected and placed in Department of Transportation approved containers, segregated by waste streams: see the Investigation-Derived Waste Handling and

Storage TGI for details (Arcadis 2017c). All containers will be labeled as non-hazardous unless otherwise instructed by the project manager. Containerized IDW will be stored on site until it is profiled and subsequently transported to an approved facility for disposal or recycling. Waste manifests for all IDW suspected to have come into contact with PFAS should clearly note the presence of PFAS. Additional IDW sampling and management details will be provided in the site-specific Work Plan (QAPP addendum) and will be consistent with applicable Army policies and Army post requirements. Personal protective equipment (e.g., gloves, disposable clothing, disposable equipment) resulting from personnel cleaning procedures and soil sampling activities will be placed in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

9 DATA RECORDING AND MANAGEMENT

The supervising field lead will be responsible for documenting drilling events to record all relevant information in a clear and concise format. The record of drilling events should include:

- Start and finish drilling dates;
- Project name and location;
- Project number, client, and site location;
- Boring number and depths;
- Soil descriptions;
- Depth to water;
- Well construction specifications, if applicable (screen and riser material and diameter, sump length, screen length and slot size, riser length, sand pack type);
- Quantities of materials used (e.g., bentonite, grout);
- Type of drilling tools used (e.g., rig type);
- Core barrel size;
- Names of contractor's drillers, inspectors, or other people onsite; and,
- Weather conditions.

Field staff should ensure COC Forms are properly completed, and verify which PFAS analytes (e.g., just PFOS and PFOA, some or all of the modified method 537 target analyte list) are required for analysis and note on the COC.

All documents (and photographs) should be scanned and electronically filed in the appropriate project directory for easy access. In addition, the locations of newly-installed wells will be documented photographically or in a site sketch. If appropriate, a measuring wheel or engineer's tape will be used to determine approximate distances between important site features.

The well location, ground surface elevation, and inner and outer casing elevations will be surveyed using the method specified in the site Work Plan. Generally, a local baseline control will be set up. This local baseline control can then be tied into the appropriate vertical and horizontal datum, such as the National

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Geodetic Vertical Datum of 1929 or 1988 and the State Plane Coordinate System. At a minimum, the elevation of the top of the inner casing used for water-level measurements should be measured to the nearest 0.01 foot. Elevations will be established in relation to the National Geodetic Vertical Datum of 1929. A permanent mark will be placed on top of the inner casing to mark the point for water-level measurements.

10 DECONTAMINATION

To avoid cross-contamination during drilling and sampling, all reusable groundwater sampling equipment that has or is suspected to have come into contact with groundwater or soil will be decontaminated between each sample using the following steps. If Class B firefighting foam is a suspected PFAS source at any sampling location, then these steps should be performed twice.

- Don new pair of Nitrile gloves prior to decontamination
- Scrub using a plastic brush and a non-phosphate soap free of VOCs (e.g., Liquinox, Alconox) and plastic brush;
- Double-rinse in potable deionized or distilled water;
- Rinse once with methanol or isopropyl alcohol;
- Rinse once with laboratory-certified PFAS-free water;
- Collect all rinsate in a sealed pail for disposal
- Allow time for equipment to air dry prior to re-use.

While strongly recommended, the use of solvents may be excluded for project-specific H&S concerns. If solvents are prohibited after DQO development, then additional procedures should be evaluated by the project team. Contingencies could include the use of dedicated sampling equipment at each sampling location or amending laboratory procedures to mitigate the increased risk of cross-contamination.

Additionally, the following decontamination procedure could be utilized when organic solvent use is not possible.

- Don new pair of Nitrile gloves prior to decontamination
- Scrub using a plastic brush and a non-phosphate soap free of VOCs (e.g., Liquinox, Alconox) and plastic brush;
- Single-rinse in potable deionized or distilled water;
- Scrub using a plastic brush and a non-phosphate soap free of VOCs (e.g., Liquinox, Alconox) and plastic brush;
- Rinse twice with deionized water and once with PFAS-free water;
- Collect all rinsate in a sealed pail for disposal
- Allow time for equipment to air dry prior to re-use.

Drive casings and other drilling tooling will be steam cleaned or replaced with new equipment between boreholes. Steam cleaning will be performed by the drillers within a temporary decontamination or other containment area designated by the supervising engineer or geologist that is located outside of the work zone. All decontamination water will be collected and containerized for disposal.

See additional specifics in P-04, TGI - Groundwater and Soil Sampling Equipment Decontamination in PQAPP Appendix A.

11 QUALITY ASSURANCE

In general, the following quality assurance and quality control (QA/QC) samples should be collected:

- Equipment blanks
- Field (i.e., reagent) blanks
- Field duplicates
- Matrix spike/matrix spike duplicate

Details on QC sampling requirements (e.g., frequency of collection, types of QA/QC samples) are provided in the PQAPP and will be outlined in various Site-specific sampling scopes of work in the QAPP Addendum. Additionally, detailed procedures related to equipment and field (i.e., reagent) blank sample collection are outlined in the Equipment and Reagent Blank Sample Collection TGI (Arcadis 2018c). In general, equipment blanks should be collected from every piece of downhole equipment that could come in contact with soil or groundwater during sample collection. This includes all downhole tooling (e.g., drill bits, drill rods).

Prior to initiating field activities, water sources to be used during drilling and well construction activities should be sampled to verify those sources are PFAS-free. While not part of the PQAPP, this is considered best practice and should be completed to the extent possible.

Refer to quality control requirements for the project to ensure that appropriate quality assurance and quality control (QA/QC) samples are collected. When collecting QA/QC samples, the same guidelines apply as when collecting regular samples – specifically that:

- Samples should be collected in laboratory-supplied HDPE bottles;
- Bottle caps must remain in the hand of the sampler until replaced on the bottle;
- Labels must be completed after the caps have been placed back on each bottle; and,
- Samples must be stored in appropriate transport containers (coolers) with ice (Ziploc®-type bags for use as ice containers) with appropriate labeling. **Do not use blue ice. Store PFAS samples in a separate cooler from other types of samples.**

12 REFERENCES

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TGI – Monitoring Well Inspection Assessment

Rev #: 1

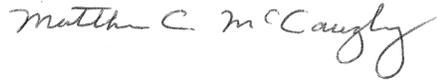
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| | 0 | 4/19/2017 | All | Re-written as TGI | Patrick Nolan M. McCaughey |
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Approval Signatures

Prepared by:

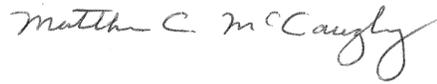


6/30/2022

Matthew C. McCaughey (Preparer)

Date

Reviewed by:



6/30/2022

Matthew C. McCaughey (Subject Matter
Expert)

Date

1. Introduction

This Technical Guidance Instruction (TGI) specifies the procedures for performing inspections and inventories of existing monitoring wells.

2. Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3. Scope and Application

Monitoring well inventories are periodically conducted to assess the condition of existing monitoring wells and to identify the need for repairs, replacement of parts, or replacement of wells that are no longer usable. A well inventory involves an inspection of the overall condition of the well, comparison of measurable quantities (e.g., riser stickup relative to grade and total depth), general verification of survey coordinates and elevation, and measurement of depth to water in the well.

This TGI applies to piezometers constructed analogous to monitoring wells. For simplicity, such piezometers are also referred to as monitoring wells for the remainder of this document. For all other types of wells (e.g., remediation wells such as injection, extraction, sparge, etc.), please refer to the appropriate guidance document regarding procedures for conducting inspections on those specific wells.

4. Personnel Qualifications

Arcadis field personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as identified in the site-specific Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. The HASP will also identify any access control requirements.

Prior to mobilizing to the field, the team will review and be thoroughly familiar with relevant site-specific documents including but not limited to the task-specific work plan or field implementation plan (FIP)/field sampling plan, Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Arcadis personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. Additionally, the Arcadis team will review and be thoroughly familiar with documentation provided by equipment manufacturers and become familiar with the operation of (i.e., hands-on experience) all equipment that will be used in the field prior to mobilization.

The well inspection assessment procedures described below will be carefully adhered to and conducted under the supervision of an experienced geologist, engineer, or other qualified individual. Ideally, Arcadis personnel directing, supervising, or leading well assessment activities will have a minimum of one (1) year of field experience. It is recommended that field employees with less than six (6) months of experience be accompanied by a supervisor (as described above) to ensure that adequate survey techniques are employed.

The Arcadis CPM will be responsible for periodic observation of field activities and review of field generated documentation associated with this TGI and for implementation of corrective action if well conditions necessitate them.

5. Equipment List

The following materials will be available, as required, during performance of a monitoring well inventory:

- Health and safety equipment (as required by the site-specific Health and Safety Plan)
- Ruler or tape measure
- Water level indicator and/or interface probe
- Bailer
- Metal detector
- Indelible ink pen
- Paint pen
- Well keys
- Wrenches or ratchet set for accessing flush-mount well covers
- Cleaning equipment

- Well construction information (e.g., construction log, as-built, summary table, etc.)
- Digital camera (or phone with camera)
- Field notebook and digital data collection device (tablet or smartphone)
- Appropriate field form(s) (see Attachment A)

If feasible, a supply of typical replacement parts (e.g., locks, bolts, well caps) should be available to enable immediate usage, as necessary.

6. Cautions

It is important to confirm the correct identity of wells, particularly to those installed in a cluster. In these cases, however, the wells usually differ significantly in terms of depth below grade. During the well inspection assessment, verify that all wells are properly labeled by comparing their measured depth to the reported depth as installed. If the well identity is incorrectly labeled or not labeled, provide a clear, correct label using an indelible ink pen on the inside of the steel protective cover for the well, or on the outside of the steel protective cover using a paint pen. Take photos to document, as necessary.

One challenge with performing this task is locating existing monitoring wells in the field. Compilation and use of existing well records such as well location photos and aerial map images (e.g., Google Earth) are recommended. Note that the Well inspection Survey Assessment Form (**Attachment A**) includes a sketch area to help locate wells in future surveys.

If present, remove standing water from the curb box using a bailer before opening the monitoring well. Refer to project waste management plan for proper disposal of water.

7. Health and Safety Considerations

Field activities associated with monitoring well installation will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities. Care should be taken using tools to access flush-mount curb boxes. Wells in or near roadways may require a traffic control plan and traffic control measures (such as cones, flagging, and/or signs) prior to accessing. Access to wells containing chemicals of concern may pose a chemical exposure and biological hazard.

8. Procedure

The typical procedure for assessing the integrity of a monitoring well is outlined below.

8.1 Planning

Compile a list of wells to be inventoried and available information concerning their location and physical characteristics including photos and aerial maps.

8.2 Well Inspection Assessment

The well inspection procedure is described below:

- Locate the monitoring well using site maps and, if needed, a metal detector. Record field observations using the FieldNow application on digital device or an appropriate field form.
- Two field forms are provided in **Attachment A**. The first form is designed for a detailed description of an individual well inspection, while the second form is designed for the collection of multiple well inspections and observations on one page and can be used as a report summary table.
- Examine the well for the presence of an identification label. If absent, label the well with the appropriate well number after measuring the total depth of the well to verify that the depth matches the well number. If the well identity is incorrectly labeled or not labeled, provide a clear, correct label using an indelible ink pen on the inside of the steel protective cover for the well, and on the outside of the steel protective cover using a paint pen.
- Examine the surface condition of the well. Record the type of well (i.e., flush mount or above-grade stickup), condition of the well cover and surface seal. Confirm the protective casing is not bent or rusted through, the PVC casing is not broken or chipped, there is no evidence of frost heaving or subsidence.
- Unlock and open the well. Record the type (e.g., PVC or stainless steel), dimensions (i.e., casing diameter and riser stickup relative to grade), condition of the well casing, and type of well cap. Record any observations of recent modifications of the well casing. If the well cap is missing, replace with available parts or record the type of cap required.
- Measure the above-grade portion of the well riser stickup and compare to the known length of the stickup measured during well installation (surveyed top of inner casing elevation minus ground surface elevation). If the difference between the observed stickup length and the known stickup length is greater than 0.1 foot, the monitoring well location and elevation should be re-surveyed.
- Locate the marked measuring point along the top of the well casing. If no mark is visible, add a mark at the highest point of the casing using an indelible ink pen.
- Measure the depth to water, total depth of the well and any non-aqueous phase liquids (NAPL) thicknesses. For total depth measurements, account for any difference in calibration of the measuring tape on the probe (i.e., distance from part of probe that measures depth to water and the physical bottom of the probe that will measure total depth of the well). Record any obstructions encountered and a description of the feel of the well bottom (i.e., soft due to sediment or hard).
- Compare observations concerning the measured dimensions of the well with the listed values. Based on these results as well as other observations concerning the condition of the well, record any appropriate recommendations on the Well Inspection form (**Attachment A**).
- Perform any recommended maintenance activities that can be accomplished with available equipment.
- Remove all equipment from the well. If no additional maintenance activities are to be performed, close the well and collect all personal protection equipment (PPE) and other wastes generated for disposal.

8.3 Post-Assessment Activities

Depending on the results of the well inventory, several additional activities may be warranted prior to future usage of the well. Typical follow-up activities include replacement of missing parts, removal of sediment from the base of the well, re-surveying of the well, or complete replacement if the well is determined to be unusable.

As stated above, a supply of locks, bolts, and well caps should be available for immediate usage during performance of the well inventories. However, it may not be feasible to maintain a supply of all potential replacement parts due to the variety of well types in use. Therefore, a list of required replacement parts should be compiled during the performance of a well inventory event. At the conclusion of the event, the necessary replacement parts for all wells should be obtained and installed.

9. Waste Management

Materials generated during well inventory activities, including disposable equipment, will be disposed in appropriate containers.

10. Data Recording and Management

10.1 Digital Data Collection Process Overview

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

10.2 Digital Data Collection Tools for Well Inspections

Arcadis is transitioning from the use of paper forms to digital field forms using web based FieldNow applications accessible on field tablets and smart phones. Company-wide roll out of a FieldNow application for a Well Inspection app is targeted by the end of 2022. Paper forms are included in Revision 1 (June 2022) of this TGI. Specifically, a blank well inspection form is provided in **Attachment A**. Additional guidance and examples of the digital data collection tools for soil descriptions will be provided in the next revision to this TGI.

11. Quality Assurance

Field measurements will be double-checked periodically (e.g., at least one of these measurements per well should be repeated to verify accuracy).

12. References

No references apply to this TGI.

Attachment A

Monitoring Well Inspection Form

Monitoring Well Integrity Assessment Form

(For each item, check appropriate response or fill in the blank)

| | |
|---|--------------------------|
| | Date _____ |
| Well ID _____ | ID Clearly Marked? _____ |
| Photo filename _____ | Project Name _____ |
| Weather _____ | Project Number _____ |
| General Description of Surroundings _____ | Field Personnel _____ |

| | |
|---|--|
| <p>Well Condition:</p> <p>Damaged? <input type="checkbox"/> (Describe Below)</p> <p>Abandoned? <input type="checkbox"/></p> <p>Stick Up <input type="checkbox"/> Flush Mount <input type="checkbox"/></p> <p>Lockable cover? _____</p> <p>Lock present? _____</p> <p>Key number: _____</p> <p>Stick up height _____</p> <p>Casing material _____</p> <p>Well diameter _____</p> <p>Protective casing material: _____</p> <p>Protective casing diameter: _____</p> <p>Cap present? Type? _____</p> <p>Vented? If so, how? _____</p> <p>Measuring point clearly marked? _____</p> <p>Total depth reported: _____</p> <p>Total depth measured: _____</p> <p>DTW: _____</p> <p>Well obstructed? If so, depth? _____</p> <p>Well bottom soft (sediment) or firm? _____</p> <p><i>Flush Mount Wells Only</i></p> <p>Gasket present? _____</p> <p>Bolts present? _____</p> <p>Teflon washers present? _____</p> | <p>Surface Condition:</p> <p>Damaged? <input type="checkbox"/> (Describe Below)</p> <p>Pad/cement intact? _____</p> <p>Curb box/well cover present? _____</p> <p style="padding-left: 100px;">Intact? _____</p> <p>Seal condition _____</p> <p>All bolts present? _____</p> <p>Ground surface slopes away from well? _____</p> <p>Location Sketch</p> |
|---|--|

Comments/Recommendations:

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TGI – Groundwater and Soil Sampling Equipment Decontamination

Rev: 2

Rev Date: June 14, 2022

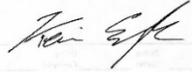
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| | 0 | February 23, 2017 | All | Conversion from SOP to TGI | Cassandra McCloud / Pete Frederick |
| | 1 | May 8, 2020 | 4, 5 | Added note regarding use of Liquinox and 1,4-Dioxane | Marc Killingstad |
| | 2 | June 14, 2022 | All | Conversion to new TGI format and minor edits. | Kevin Engle / Marc Killingstad |

Approval Signatures

Prepared by:

6/14/2022



Name (Preparer)

Date

Reviewed by:

6/14/2022



Marc Killingstad (Subject Matter Expert)

Date

1 Introduction

This document is intended to provide guidance to staff performing decontamination procedures at project sites. The content in this document describes the intended use, scope and application, personnel qualifications, equipment, cautions, health and safety considerations, procedures, waste management, data recording and management, and quality assurance of decontamination procedures.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

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3 Scope and Application

Decontamination is performed on sampling equipment prior to sample collection to ensure that the sampling equipment that contacts a sample, or monitoring equipment that is brought into contact with environmental media to be sampled, is free from analytes of interest and/or constituents that could interfere with laboratory analysis for analytes of interest. Sampling equipment must be appropriately cleaned prior to use for sampling or coming into contact with environmental media to be sampled and following completion of the sampling event prior to shipment or storage. The effectiveness of the decontamination procedure should be verified by collecting and analyzing equipment blank samples.

The sampling equipment cleaning procedures described herein includes pre-field, in the field, and post-field cleaning of sampling equipment which may be conducted at an established equipment decontamination area (EDA) on site, as appropriate and necessary. Sampling equipment that may require decontamination at a given site include soil sampling tools; groundwater, sediment, and surface-water sampling devices; water testing instruments; down-hole instruments; and other activity-specific sampling equipment. Non-disposable equipment will be cleaned before collecting each sample, between each sample collected, and prior to placing sampling equipment in protective cases, or containers for transport. Cleaning procedures for sampling equipment should be monitored by collecting equipment blank samples as required in project work plans, field sampling plans, quality assurance project plans (QAPP), or other pertinent project documents. Dedicated and/or single-use (i.e., not to be re-used) sampling equipment will not require decontamination.

4 Personnel Qualifications

Arcadis field sampling personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour hazardous waste operations and emergency response (HAZWOPER) training and/or Occupational Safety and Health Administration (OSHA) HAZWOPER site supervisor training. Arcadis personnel will also have current training as specified in the Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. In addition, Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and Technical Guidance Instructions (TGIs) and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. The project HASP and other documents will identify other training requirements or access control requirements.

5 Equipment List

The equipment required for equipment decontamination is presented below. Note that certain contaminants may require specific materials be used that are not captured in this list. Always review project and contaminant specific TGIs or work plans to ensure proper equipment is utilized. Note for per- and polyfluoroalkyl substances (PFAS) see *TGI – Per- and Polyfluoroalkyl Substances (PFAS) Field Sampling Guide*.

- Health and safety equipment, including appropriate personal protective equipment (PPE), as required in the site HASP
- Deionized water that meets the analytical criteria for deionized water with no detectable constituents above the reporting limits for the methods to be used and analytes being analyzed for. Deionized water is used for inorganics, and organic-free water for volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), pesticides, etc.
- Non-phosphate detergent such as Alconox® or, if sampling for phosphorus or phosphorus-containing compounds, Liquinox (or equivalent). NOTE: Liquinox has shown to provide false positives for 1,4-Dioxane and should not be used at sites where that may be a constituent of concern (COC).
- Tap water
- Rinsate collection plastic containers

- Department of Transportation (DOT)-approved waste shipping container(s), as specified in the work plan, field sampling plan, or regulatory requirements if decontamination waste is to be shipped for disposal
- Brushes
- Large heavy-duty garbage bags
- Spray bottles
- (Optional) – Isopropyl alcohol (free of ketones) or methanol. These can be wipes or diluted with water (usually 1part isopropyl/methanol to 10 parts water) if a spray is needed.
- Airtight, sealable plastic baggies, such as Ziploc®-type
- Plastic sheeting

6 Cautions

Rinse equipment thoroughly and allow the equipment to dry before re-use or storage to prevent introducing solvent into sample medium. If manual drying of equipment is required, use clean lint-free material to wipe the equipment dry. Ensure all rinse materials do not adversely affect sample collection efficiency or analytical results.

Store decontaminated equipment in a clean, dry environment. Do not store near combustion engine exhausts. Properly containerize equipment to ensure cross-contamination doesn't happen from other uncontaminated surfaces or equipment.

If equipment is damaged to the extent that decontamination is uncertain due to cracks, gouges, crevices, or dents, the equipment should not be used and should be discarded or submitted for repair prior to use for sample collection.

A proper shipping determination regarding hazardous materials will be performed by a DOT-trained individual for cleaning materials shipped by Arcadis.

Caution should be exercised to avoid contact with the pump casing and water in the container while the pump is running (do not use metal drums or garbage cans) to avoid electric shock.

7 Health and Safety Considerations

Review the safety data sheets (SDS) for the cleaning agents and materials used in decontamination. If solvent is used during decontamination, use appropriate PPE and work in a well-ventilated area and stand upwind while applying solvent to equipment. Apply solvent in a manner that minimizes potential for exposure to workers and bystanders. Follow health and safety procedures outlined in the HASP.

8 Procedure

A designated area will be established to clean sampling equipment in the field prior to and following sample collection. Equipment cleaning areas will be set up within or adjacent to the specific work area, but not at a location that expose equipment to contamination (i.e., exposed to combustion engine exhaust). Detergent solutions will be prepared in clean containers for use in equipment decontamination. Decontaminated equipment

will be handled by workers wearing clean gloves, properly changed to prevent cross-contamination. The procedures detailed in this section provide an overview of common decontamination techniques. Additional steps may be required based on the type of contaminant present or client/site requirements.

Cleaning Sampling Equipment

1. Wash the equipment/pump with potable water.
2. Wash with detergent solution (Alconox®, Liquinox® or equivalent) to remove all visible particulate matter and any residual oils or grease. NOTE: Liquinox® has shown to provide false positives for 1,4-Dioxane and will not be used at sites where that may be a COC.
3. If equipment is very dirty, precleaning gross debris with a brush and tap water may be necessary.
4. If non-aqueous phase liquids are present, the use of isopropyl alcohol (free of ketones) or methanol is recommended. Cloth wipes or diluted solution can be used to remove the non-aqueous phase liquids that are hard to remove with detergent solution in step 2. Consult with project manager if non-aqueous phase liquids are present onsite and design an appropriate decontamination procedure that includes step 4.
5. Rinse with deionized water.

Decontaminating Submersible Pumps

Submersible pumps may be used during well development, groundwater sampling, or other investigative activities. The pumps must be cleaned and flushed before and between uses. This cleaning process will consist of an external detergent solution wash and tap water rinse, a flush of detergent solution through the pump, followed by a flush of potable water through the pump. Flushing will be accomplished by using an appropriate container filled with detergent solution and another container filled with potable water. The pump will be flushed with deionized water as the last step prior to use. The pump will run long enough to effectively flush the pump housing and hose (unless new, disposable hose is used). Disconnect the pump from the power source before handling. The pump and hose will be placed on or in clean polyethylene sheeting to avoid contact with the ground surface.

9 Waste Management

Equipment decontamination rinsate will be managed in conjunction with all other waste produced during the field sampling effort. Waste management procedures are outlined in the work plan or Waste Management Plan (WMP).

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff

are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Equipment cleaning and decontamination will be noted during project documentation. Information will include the type of equipment cleaned, the decontamination location, specific procedures utilized, solvents and/or cleaning agents used, source of water, and deviations or omissions from this TGI.

Unusual field conditions should be noted if there is potential to impact the efficacy of the decontamination or subsequent sample collection.

An inventory of the solvents brought on site and used and removed from the site will be maintained in the project documentation. Records will be maintained for solvents used in decontamination, including lot number and expiration date.

Containers with decontamination fluids will be labeled.

11 Quality Assurance

Equipment blanks should be collected to verify that the decontamination procedures are effective in minimizing potential for cross contamination. The equipment blank is prepared by pouring deionized water (or organic-free water, for organic analyses) over the clean and dry tools and collecting the water into appropriate sample containers. Equipment blanks should be analyzed for the same set of parameters that are performed on the field samples collected with the equipment that was cleaned as specified in the sampling and analysis plan. Equipment blanks are collected per equipment set, which represents all the tools needed to collect a specific sample.

12 References

USEPA Region 9 - Field Sampling Guidance #1230, Sampling Equipment Decontamination.

USEPA Region 1 - Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells.

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TGI – Per- and Polyfluoroalkyl Substances (PFAS) Field Sampling Guide

Rev: 10

Rev Date: January 26, 2022

Version Control

| Issue | Revision No. | Date Issued | Page No. | Description | Reviewed By |
|-------|--------------|-------------------|--------------------|--|---|
| | 0 | April 27, 2017 | All | Initial Release | Erica Kalve Erika Houtz Sue Tauro |
| | 1 | June 19, 2018 | 1 through 4 and 17 | Updated Information on Sampling Materials | Erica Kalve Erika Houtz |
| | 2 | October 15, 2018 | 6 to 16 | Minor updates on laboratory elements, updates to decontamination procedures, and clarification on equipment and reagent blank collection | Erika Houtz Erica Kalve |
| | 3 | December 17, 2018 | 4, 6, 17 | Removed Sharpies from acceptable field writing implements; Changed language in Section 3.2 and Section 10.5 to provide stricter guidance for DoD projects. | Erika Houtz, Erica Kalve |
| | 4 | March 26, 2019 | 4,5 | Removed Citranox from acceptable Decon solutions in Table 1a, added all fluoropolymer containing materials to prohibited items in Table 1b. Made a correction that Liquinox contains trace levels of 1,4 Dioxane, not Alconox. | Erika Houtz |
| | 5 | October 16, 2020 | 14 | Added Air Force preference to sample surface water at surface for Air Force investigations. | Erika Houtz |

| | | | | |
|---|------------------|--|--|-------------------------------|
| 6 | March 23, 2021 | 4, 5, 7, 12, 13, 14, 15, 16, 17 | Made clarifications that fine/ultra-fine point Sharpies are allowed. Referenced 2018 MDEQ sampling guidance. Made updates to 'After Sample Collection' in Section 7. | Kevin Engle |
| 7 | April 18, 2021 | All | Changed title from Poly- and Perfluoroalkyl Substances to “Per- and Polyfluoroalkyl Substances” and changed PFASs to PFAS. | Rosario Varrella, Erika Houtz |
| 8 | May 4, 2021 | 12, 13, 15, 16 | Clarified that sample containers should have an HDPE lined screw cap and that LDPE plastic sheeting should be used. | Kevin Engle, Erika Houtz |
| 9 | October 20, 2021 | Note that numbers have shifted one page forward relative to prior versions. 5, 7, 9-12, 15, 16, 18-25. | Specific acceptable sunscreen and insect repellent brands were added to Table 1. Clarified language regarding footwear and H&S trainings. Laboratories section and Section 10.5 was updated to reflect new laboratory names and an updated version of the QSM. Sections 5 and 6 were updated to provide clearer language on health and safety protocols for sunscreen, insect repellent, and rain events. Added language to specify decontamination of reusable equipment prior to initial use in Section 7.1. Section 8 on Waste Management was updated to state that | Kevin Engle, Erika Houtz |

waste storage and disposal should be determined in the site specific workplan. Section 9 was updated to include Rite in the Rain® notebooks as approved for PFAS sampling. Changed the term “sample port” to “sample location” when describing where to place plastic sheeting. Section 10.1 was updated to indicate an equipment blank can be collected for unvetted hazard controls that contact a sample. References were updated to include the newer version of the DoD QSM, MDEQ Sampling Guidance, and California State Water Board PFAS Sampling Guidance.

| | | | |
|----|------------------|--------------------|--|
| 10 | January 26, 2022 | Various, Section 7 | <p>TGI formatted to comply with new QMS TGI template and Arcadis brand compliance.</p> <p>Indicated to avoid use of anti-fog spray on safety glasses due to possible presence of PFAS.</p> |
|----|------------------|--------------------|--|

Approval Signatures

Prepared by:

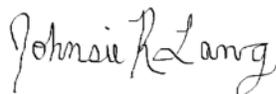
1/26/2022



Kevin Engle, PG Geologist (Preparer)

Date

Reviewed by:



1/26/2022

Johnnie Lang, PhD (Subject Matter Expert)

Date

1 Introduction

This document is intended to provide guidance to field staff sampling for Per- and Polyfluoroalkyl Substances (PFAS). The content in this document describes the intended use, scope and application, personnel qualifications, equipment, cautions, health and safety considerations, procedures, waste management, data recording and management, and quality assurance of PFAS sampling.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

The purpose of this Technical Guidance Instructions (TGI) is to provide guidance on field sampling to be used for **Per- and Polyfluoroalkyl Substances** (PFAS). This protocol was adapted from various sources including Arcadis Australia, Transport Canada, and the U.S Army Corp of Engineers (USACE) Omaha. In general, sampling techniques used for PFAS site characterization are consistent with conventional sampling techniques used in the environmental industry, but special consideration is made regarding PFAS-containing materials and cross-contamination potential. **Table 1a** provides a summary of materials that have been approved for site investigation; this list is expected to grow longer as industry experience increases. **Table 1b** provides a summary of field equipment and materials that have available testing information and/or industry knowledge regarding

PFAS cross-contamination potential, and it is recommended that these materials be prohibited for sample collection; for materials that are suspected of containing PFAS and/or to retain PFAS, these recommendations are considered preliminary and subject to change. Further discussion of approved and prohibited materials is found throughout this document.

Table 1a: Summary of Acceptable Sampling Equipment and Materials for PFAS Site Investigations

| Sampling Materials | Additional Considerations | References |
|--|--|--|
| Water Sampling Materials | | |
| High density polyethylene (HDPE) or silicone tubing materials | -- | DER 2016; USACE 2016; NHDES 2016; MassDEP 2017 |
| HDPE HydraSleeves™ | Low density polyethylene (LDPE) HydraSleeves™ are not recommended | USACE 2016; MassDEP 2017 |
| Drilling and Soil Sampling Materials | | |
| PFAS-free drilling fluids | -- | DER 2016 |
| PFAS-free makeup water | Confirm PFAS-free water source via laboratory analysis prior to investigation | -- |
| Acetate liners | For use in soil sampling | USACE 2016 |
| Sample Containers and Storage | | |
| HDPE sample containers with HDPE lined lids for soil and water samples | Laboratory should provide; whole bottle analysis of aqueous samples combined with a solvent rinse of bottle is recommended | DER 2016, MassDEP 2017 |
| Ice contained in plastic (polyethylene) bags (double bagged) | -- | DER 2016; USACE 2016; NHDES 2016; MassDEP 2017 |
| Field Documentation | | |
| Ball point pens | -- | MassDEP 2017 |
| Standard paper and paper labels | -- | DER 2016; USACE 2016; NHDES 2016; MassDEP 2017 |
| Fine/Ultra-Fine point Sharpies® | Larger point Sharpies® should be avoided. | MDEQ 2018 |
| Decontamination | | |
| Water-only decontamination | Confirm PFAS-free water source via laboratory analysis prior to investigation | DER 2016 |

| Sampling Materials | Additional Considerations | References |
|--|--|--------------------------------------|
| Alconox® or Liquinox® followed by deionized water or PFAS-free water rinse | Liquinox® known to contain trace levels of 1,4-dioxane | NHDES 2016; USACE 2016; MassDEP 2017 |
| Methanol, isopropanol, or acetone | Special health and safety precautions are necessary | UNEP 2015; USACE 2016 |
| <i>Sun and Biological Protection</i> | | |
| OFF Deep Woods, Sawyer Permethrin | Apply >10 m away from sampling area | MDEQ 2018 |
| Banana Boat, Coppertone, Neutrogena, Meijer, and L'Oreal Sunscreens | Apply >10 m away from sampling area | MDEQ 2018 |

Note: This list is considered preliminary and additional materials may be added as additional information becomes available. Project teams are expected to follow a methodical evaluation process of materials to be used and confirm acceptance prior to implementation of field activities.

Table 1b: Summary of Sampling Equipment and Materials Not Recommended for PFAS Site Investigations.

| Sampling Materials | Known PFAS-Containing Materials | Suspected PFAS-Containing Materials | Materials with Potential to Retain PFAS | References |
|--|---------------------------------|-------------------------------------|---|--|
| <i>Water Sampling Materials</i> | | | | |
| Teflon®, PTFE-containing or other fluoropolymer coated or containing field equipment (e.g., tubing, bailers, liners, tape, plumbing paste, pump parts) | x | | | DER 2016; USACE 2016; NHDES 2016; MassDEP 2017 |
| Passive diffusion bags | | | x | MassDEP 2017 |
| LDPE HydraSleeves™ | | | x | USACE 2016; MassDEP 2017 |
| Water particle filters | | | x | MassDEP 2017 |
| <i>Drilling and Soil Sampling Materials</i> | | | | |
| Aluminum foil | | | x | DER 2016; USACE 2016; NHDES 2016; MassDEP 2017 |
| Drilling fluid containing PFAS | x | x | | DER 2016 |
| <i>Sample Containers and Storage</i> | | | | |
| Glass sample containers with lined lids | | | x | DER 2016; USACE 2016; NHDES 2016; MassDEP 2017 |

| Sampling Materials | Known PFAS-Containing Materials | Suspected PFAS-Containing Materials | Materials with Potential to Retain PFAS | References |
|---|---------------------------------|-------------------------------------|---|---|
| LDPE containers and lined lids | | | x | USACE 2016 |
| Teflon® or PTFE- lined lids on containers (e.g., sample containers, rinsate water storage containers) | x | | | DER 2016; USACE 2016; NHDES 2016; MassDEP 2017 |
| Reusable chemical or gel ice packs (e.g., BlueIce®) | | x | | DER 2016; USACE 2016; NHDES 2016; MassDEP 2017 |
| Field Documentation | | | | |
| Self-sticking notes and similar office products (e.g., 3M Post-it-notes) | | x | | DER 2016; USACE 2016; NHDES 2016; MassDEP 2017 |
| Waterproof paper, notebooks, and labels | x | | | DER 2016, MassDEP 2017 |
| Markers | | x | | NHDES 2016 |
| Decontamination | | | | |
| [Some] detergents and decontamination solutions (e.g., Decon 90® Decontamination Solution) | x | x | | DER 2016; NHDES 2016; MassDEP 2017 |

Note: For materials that are suspected of containing PFAS, or have the potential to retain PFAS, project specific considerations may provide adequate justification for use during the field event. For example, further evaluation may be conducted in the form of pre-field equipment blank sample analysis.

Given the extremely low detection limits associated with PFAS analysis and the many potential sources of trace levels of PFAS, field personnel are advised to err on the side of caution by strictly following these protocols, frequently replacing nitrile gloves, and rinsing field equipment to help mitigate the potential for false detections of PFAS. A summary of other specific items related to field sampling for PFAS are discussed in the sections below.

This TGI applies to all Arcadis and subcontractor personnel involved in field sampling for PFAS.

4 Personnel Qualifications

4.1 Sampling Personnel

Field personnel must have current health and safety training, including 40-hour HAZWOPER training, up to date 8-hour refresher, site supervisor training, and site-specific training, as needed. In addition, field personnel will be versed in the other relevant SOPs (e.g., low flow sampling) and will possess the skills and experience necessary

to successfully complete the desired field work. The site Health and Safety Plan (HASP) and other documents will identify any other training requirements such as site-specific safety training or access control requirements.

4.2 Laboratories

These laboratories are example laboratories that could be used to analyze environmental media for PFAS, pending project approval:

- United States: Pace, SGS, Vista, ALS, and Eurofins
- Canada: AXYS-SGS and Bureau Veritas

Other laboratories may be used if they are appropriately accredited for PFAS analysis according to any project requirements. It is recommended that a laboratory is Environmental Laboratory Accreditation Program (ELAP)-accredited for PFAS analysis in accordance with the Department of Defense (DoD) Quality Systems Manual (QSM) 5.3 Table B-15 or any subsequent updates. **For all data collection efforts at DoD sites, PFAS data must be obtained using a method that is DoD ELAP-accredited under QSM 5.3 or later.**

5 Equipment List

The following equipment and materials must be available for sampling:

- Site plan of sampling locations, relevant work plan (or equivalent), and this TGI;
- Appropriate health and safety equipment, as specified in the site HASP;
- Dedicated plastic sheeting (preferably high-density polyethylene [HDPE]) or other clean surface to prevent sample contact with the ground;
- Conductivity/temperature/pH meter;
- Dissolved oxygen meter, oxidation reduction potential meter, and turbidity meter;
- Depth to water meter;
- If using low-flow groundwater sampling techniques, peristaltic pump (groundwater sampling)/bladder pump (with PFAS free bladder/ HDPE bladder), flow through cell, and accompanying HDPE and silicone tubing;
- Hydrasleeves™, if using Hydrasleeves™ for groundwater sampling;
- Metal trowel for soil samples; specialized soil/sediment sampling equipment as required;
- Brushes for scrubbing sampling equipment;
- Pens, pencils, and/or fine/ultra-fine point Sharpies® for writing;
- Clipboards, field binders, and field note pages that are not waterproof;
- Labeled sample bottles:
 - Water: HDPE bottles fitted with polypropylene screw cap only; some types of PFAS samples (primarily drinking water) may require preservative, which will be indicated by the laboratory conducting the analysis. The laboratory will specify the sample bottle volume.

- Soil and sediment: HDPE bottles fitted with polypropylene screw cap only; no preservatives. The laboratory will specify the sample bottle volume.
- If high concentrations of PFAS related to class B firefighting foams are expected, bring additional small vials to conduct field-based shaker tests for foaming;
- Ziploc® bags to hold ice and samples;
- Bottles containing “PFAS-free” water used for reagent blanks;
- Labeled, thoroughly decontaminated coolers for samples with ice; Blue ice is not permitted;
- Deionized or distilled water for initial decontamination rinsing;
- “PFAS-free” water provided by the laboratory for final decontamination rinsing;
- Methanol, isopropanol, or acetone if able to be brought safely to field site; especially important for decontamination during soil sampling;
- Alconox or Liquinox®;
- Packing and shipping materials;
- Groundwater and/or Sampling Log; and
- Chain-of-Custody (COC) Forms.

6 Cautions

6.1 Food Packaging

Some food packaging may be treated with PFAS-containing chemicals to prevent permeation of oil and water in the food outside of the packaging. To avoid potential food packaging-related PFAS contact:

- Do not bring any food outside of the field vehicles onsite and eat snacks and meals offsite.
- Wash hands after eating.
- Remove any field garments or outer layers prior to eating. Do not put them back on until done eating and hands are washed.

6.2 Field Gear

6.2.1 Clothing

Many types of clothing are treated with PFAS for stain and water resistance, in particular outdoor performance wear under brand names such as Gore-Tex®. To avoid potential clothing-related PFAS contact:

- Do not wear any outdoor performance wear that is water or stain resistant, or appears to be. Err on the side of caution.

- Wear pre-laundered (multiple washings, i.e., 6+) clothing that is not stain resistant or waterproof (unless made from the materials listed in Section 5.3.1).
- Natural fabrics such as cotton are preferred. Synthetic fabrics may also be acceptable if there is no indication on the label that the fabric is water and stain resistant.
- Most importantly, avoid contacting your clothing with sampling equipment, bottles, and samples.

6.2.2 Personal Protective Equipment

Safety Footwear

Some safety footwear has been treated to provide a degree of waterproofing and increased durability and may represent a source of trace PFAS. If at all possible, Gore-Tex footwear should not be worn and safety footwear without waterproofing should be worn; footwear that provides adequate safety from physical hazards is required and takes precedence over potential PFAS concerns. To avoid any PFAS cross contamination to samples from footwear:

- Do not contact your footwear with equipment, bottles, or samples in any way.
- Do not allow gloves used for sampling to come in contact with safety footwear.

Nitrile Gloves

Wear disposable nitrile gloves at all times. Don a new pair of nitrile gloves **before** the following activities at each sample location:

- Decontamination of re-usable sampling equipment;
- Contact with sample bottles or “PFAS-free” water bottles;
- Insertion of anything into the sample ports (e.g., HDPE tubing); and
- Handling of any quality assurance/quality control (QA/QC) samples including field blanks and equipment blanks.

Don a new pair of nitrile gloves **after** the following activities:

- Handling of any non-dedicated sampling equipment;
- Contact with contaminated surfaces; or
- When judged necessary by field personnel.

6.3 Personal Hygiene

- Shower at night.
- Do not use personal care products after showering such as lotions, makeup, and perfumes, UNLESS medically necessary.
- Use sunscreen and insect repellent as necessary for health and safety, i.e., if sampling is to occur outdoors in direct sunlight and/or if insect hazards may be present. Specific products that are acceptable for PFAS

sampling are listed in Table 1 and in Section 6.1. Apply sunscreen and insect repellent prior to initiating field sampling. If sunscreen and/or repellent need to be reapplied, ensure a safe distance away from the sampling locations and equipment (i.e., more than 10 meters (m) away). Wash hands after application and don new gloves following hand washing.

6.4 Visitors

Visitors to the site are asked to remain at least 10 m from sampling areas.

7 Health and Safety Considerations

7.1 Biological and Environmental Hazard Controls

7.1.1 Sunscreens and Insect Repellents

When site conditions warrant, insect repellent and sunscreen should be applied. Some insect repellents and sunscreen have been approved for PFAS sampling by individual states. According to Michigan Department of Environmental Quality (MDEQ; now known as Michigan Department of Environment, Great Lakes, and Energy [EGLE]), the products below are allowable (MDEQ 2018). Note that California State Water Quality Control Board's PFAS sampling guidance refers to MDEQ/EGLE's allowable list of sunscreens and insect repellents (California State Water Quality Control Board 2020).

Insect Repellents

- OFF Deep Woods
- Sawyer Permethrin

Sunscreen

- Banana Boat Sport Performance Sunscreen Lotion Broad Spectrum SPF 30
- Meijer Sunscreen Lotion Broad Spectrum SPF 30
- Neutrogena Ultra-Sheer Dry-Touch Sunscreen Broad Spectrum SPF 30
- Banana Boat for Men Triple Defense Continuous Spray Sunscreen SPF 30
- Banana Boat Sport Performance Coolzone Broad Spectrum SPF 30
- Banana Boat Sport Performance Sunscreen Lotion Broad Spectrum SPF 30
- Banana Boat Sport Performance Sunscreen Stick SPF 50
- Coppertone Sunscreen Lotion Ultra Guard Broad Spectrum SPF 50
- Coppertone Sport High-Performance AccuSpray Sunscreen SPF 30
- Coppertone Sunscreen Stick Kids SPF 55
- L'Oréal Silky Sheer Face Lotion 50+
- Meijer Clear Zinc Sunscreen Lotion Broad Spectrum SPF 15, 30 and 50
- Meijer Wet Skin Kids Sunscreen Continuous Spray Broad Spectrum SPF 70
- Neutrogena Beach Defense Water + Sun Barrier Lotion SPF 70
- Neutrogena Beach Defense Water + Sun Barrier Spray Broad Spectrum SPF 30
- Neutrogena Pure & Free Baby Sunscreen Broad Spectrum SPF 60+

Please plan for sampling events and purchase these products ahead of time. For any sunscreens and bug sprays, including those listed above, always follow these instructions for application:

- Insect repellents and sunscreen should be applied away from the work area prior to initiating sampling.
- When re-applying, stay at least 10 m away from the sampling locations and equipment.
- Wash hands after application and don new nitrile gloves.

7.1.2 Rain Event

Special care should be taken when rain is falling at the project site:

- Field sampling during extreme rainfall should be avoided if possible. If sampling needs to take place during a rain event (or other extreme weather condition), ensure the rain gear or other safety clothing is appropriate. For example, rain gear made from the following materials is allowable: polyurethane, PVC, wax coated fabrics, rubber/neoprene, uncoated Tyvek® (MDEQ 2018).
- If project timelines are tight, consider the use of a gazebo tent that can be erected over the top of the monitoring well to provide shelter from the rain. The canopy material is possibly a PFAS-treated surface and should be managed as such; therefore, wear gloves when moving the tent, change them immediately after moving the tent, and avoid further contact with the tent until all sampling activities have been finished and the team is ready to move on to the next site.

7.1.3 Other H&S Considerations

- ***If an unapproved or potentially suspect hazard control is needed for health and safety, apply or keep that control away from the samples, document its use in field notes, and, if it does contact a sample, take an equipment blank with that material.***
- The ability to safely access the surface water sampling locations must be verified before sampling.
- Field activities must be performed in accordance with the site HASP, a copy of which will be present onsite during such activities.
- Safety hazards associated with sampling surface water include fast-moving water, deep water, and steep slopes close to sampling sites. Use extreme caution when approaching sampling sites.
- If thunder or lightning is present, discontinue sampling and take cover until 30 minutes have passed after the last occurrence of thunder or lightning.
- Use caution when removing well caps as well may be under pressure, cap can dislodge forcefully and cause injury.
- Avoid the use of anti-fog sprays on glasses, which may contain PFAS. It's recommended to instead purchase pre-treated anti-fog safety glasses.

8 Procedure

8.1 Field Equipment Cleaning

Reusable field sampling equipment will require cleaning before initial use and between uses. For groundwater sampling, between uses, decontaminate the flow-through cell and any non-dedicated equipment (i.e., interface probe of depth to water meter) that comes into contact with well water. Trowels and other materials used to sample soil samples will also require decontamination, although dedicated, single use equipment such as liners should be used where possible.

After donning a new pair of nitrile gloves:

- Rinse sampling equipment with Alconox or Liquinox® cleaning solution; Scrub equipment with a plastic brush if needed;
- Rinse two times with distilled water or deionized water;
- Rinse one time with “PFAS-free” water or once with methanol/isopropanol/acetone, if it is available, and once with “PFAS-free” water; organic solvents are especially useful for decontaminating soil sampling equipment. If organic cleaning solvents cannot be brought to site, scrub equipment a second time after a single distilled or deionized water rinse, then rinse two times with distilled or deionized water and once with “PFAS-free” water (i.e., two scrubbing and four water rinsings total).
- Collect all rinsate in a sealed pail for disposal. Do not reuse decontamination solutions between sampling locations.

8.2 Borehole/Monitoring Well Development

If a drill rig is being used to drill for soil cores or to install monitoring wells, wear clean nitrile gloves before collecting each continuous soil sample. Additional requirements include the following:

- Verify in writing with the manufacturer that single-use liners used to collect each sample are made of a material that does not contain PFAS;
- Collect soil samples in laboratory-supplied HDPE bottles.
- Store the sample bottles in coolers and keep at a temperature of 0 to 6°C until transported to the laboratory.

8.2.1 Well Condition Survey/ Water Level Monitoring

Using equipment that has been thoroughly decontaminated according to the procedures in Section 7.1, conduct the well condition surveys and water level monitoring:

- Conduct monitoring well inspections and record water levels.
- Use an interface probe to evaluate presence/absence of non-aqueous phase liquid (NAPL).
- Measure the depth to water from the top of the polyvinyl chloride (PVC) riser and the total depth of the well.
- Record information in the field notes.

8.2.2 Monitoring Well Development and Purging

Follow these requirements for monitoring well development and purging:

- Do not use Teflon™ tubing for purging or sample collection. HDPE tubing is acceptable.
- Do not re-use materials between wells. Upon completion of use, remove all disposable materials (such as HDPE and/or silicone tubing) and place in heavy duty garbage bags for disposal.
- During development of the well, create sufficient energy to agitate the water column and create flow reversals in the well screen, filter pack and formation to loosen fine-grained materials and draw them into the well. The pumping or bailing action should then draw all drilling fluids and fine-grained material out of the borehole and adjacent formation and then out of the well. Review the Arcadis Monitoring Well Development guidance (Arcadis 2010) for more detailed information.
- Follow the low-flow purge and sampling techniques per the U.S. Protection Agency's (EPA's) guidance document titled *Low Stress (Low Flow) purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells* (2010) and ASTM's standard titled *Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations* (2002). Also available for review is the Arcadis Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells (Arcadis 2011).
- To purge the well, if using HDPE tubing and a peristaltic pump, insert the end of the tubing to the approximate depth of the midpoint of the screened section of the monitoring wells. Measure the length of HDPE tubing to be inserted into each monitoring well and pre-cut it to approximate lengths (such as the previously measured arm span of a field technician) to avoid contact with any materials other than the monitoring well and peristaltic pump. Flow rates should be as low as can be reasonably achieved. Collect and appropriately dispose of purge water.
- Silicone tubing should direct the purge water through a flow-through cell for field parameter measurements of pH, conductivity, temperature, dissolved oxygen, and turbidity. Calibrate the instrument in the field prior to use. Decontaminate the instrument and flow-through cell at each monitoring well location before purging.
- Record field parameters in intervals (generally of 3-minute duration) to ensure purge water has cycled through the flow-through cell. Sample the wells after field parameter measurements indicate stabilization, which allows collection of representative formation water (generally acceptable standards are three consecutive pH readings to within ± 0.1 units, and three consecutive conductivity, temperature and dissolved oxygen measurements to within 3%). Turbidity must be monitored, but does not need to be used as a stabilization indicator of purge completion. Record field parameter measurements at each well. Drawdown should be monitored throughout the purge.
- If wells are suspected to be dewatering throughout the purge (i.e., reduced flow rate/difficulty pumping water or bubbles begin to come through the flow through cell), turn off the pump and allow the water level to recover for ½ hour, followed by sample collection. Document these activities in the field notes.

8.3 Sample Collection

Different laboratories may supply sample collection bottles of varying sizes depending on the type of media to be sampled.

8.3.1 Sample Containers

- Collect samples in HDPE bottles fitted with a HDPE lined (no Teflon™) screw cap.
- Complete bottle labels after the caps have been placed back on each bottle.
- Do not use glass bottles due to potential loss of analyte through adsorption. This is particularly important for aqueous samples.
- Review with analytical lab the sample size, sample container, etc. depending upon the type of PFAS analysis that is being requested.

8.3.2 Soil Sampling

Before Sample Collection

- Place LDPE plastic sheeting adjacent to the sample location for use as a clean work area, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity.
- Trowels or drilling equipment that will come into contact with a sample should be decontaminated prior to sample collection, preferably with methanol/isopropanol/acetone;
- Don a new set of nitrile gloves. Do not use gloved hands to subsequently handle papers, pens, clothes, etc., before collecting samples.
- Use the HDPE bottles that are supplied by the laboratory. Make sure that the caps remain on the bottle until immediately prior to sample collection.

During Sample Collection

- Collect soil samples using a clean stainless-steel trowel or with single-use PFAS-free liners;
- Place soil samples in labeled HDPE bottles supplied by the laboratory.
- Note the time on the sample label.
- Collect any necessary duplicates/co-located samples and matrix spikes – verify with laboratory whether they need to be collected in separate sample bottles.
- Collect any necessary equipment blanks. The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFAS, after the sampling equipment has been appropriately decontaminated.
- Collect any necessary field reagent blanks. This sample should be collected after field staff return from an offsite break (e.g., lunch) to capture any potential cross-contamination from field personnel.

After Sample Collection

- Place each sample bottle in two sealed Ziploc® bags. Another brand of LDPE bag is acceptable.
- Record the label information and time of sampling in the field notes.
- Place soil sample bottles in coolers that are durable in transportation and keep the temperature between 0 and 6°C until transported to the laboratory. Do not use blue ice.

8.3.3 Groundwater Sampling

Before Sample Collection

- Place LDPE plastic sheeting adjacent to the sample location for use as a clean work area, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity.
- Don a new set of nitrile gloves. Do not use gloved hands to subsequently handle papers, pens, clothes, etc., before collecting samples.
- Use the labeled HDPE bottles that are supplied by the laboratory. Make sure that the caps remain on the bottle until immediately prior to sample collection.
- Measure depth to water and field parameters. Turbidity and the physical appearance of the purged water should be noted on the Groundwater Sampling Log.

During Sample Collection

- Start groundwater sample collection upon stabilization of field parameters.
- If low-flow groundwater sampling techniques are being used, disconnect the silicone tubing from the flow-through cell, enabling collection of groundwater samples without passing through the cell.
- Hydrasleeves are also considered acceptable for sampling of PFAS in groundwater – consult the project manager to determine which technique should be used. In general, low flow sampling is preferable.
- Collect groundwater samples (to the neck of the bottle, some headspace is acceptable) from the dedicated sampling ports at the center of the well screen. While collecting the sample, make sure the bottle cap remains in the other hand of the sampler, until replaced on the bottle.
- To mitigate cross contamination, collect groundwater samples in a pre-determined order from least impacted to greater impacted based on previous analytical data or knowledge about past activities at the site. If no analytical data are available, samples are to be collected in the following order:
 1. First sample the upgradient well(s).
 2. Next, sample the well located furthest downgradient of the interpreted or known source.
 3. The remaining wells should be progressively sampled in order from downgradient to upgradient, such that the wells closest to the interpreted or known source are sampled last.
- NOTE: If high concentrations of PFAS related to class B firefighting foams are expected in a groundwater sample, conduct a Shaker test by collecting and shaking a small portion of the sample (~10 to 25 mL) on site in a small disposable vial. If foaming is noted within the sample, document the foaming when samples are

submitted for analysis; the 'shaker test' vial can then be disposed. This shaker test provides information about how each of the samples should be handled analytically.

- After collecting the sample, tightly screw on the polypropylene cap (snug, but not too tight). This will minimize leaking or cross contamination of the sample. Most PFAS, including all analytes measured by USEPA Method 537, are not volatile at environmental pH.
- Note the time on the sample label.
- Collect any necessary duplicates and matrix spikes. As the laboratory should be analyzing the entire aqueous sample rather than sub-sampling, separate bottles will be required for these samples.
- Collect any necessary equipment blanks. The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFAS, after the sampling equipment has been appropriately decontaminated.
- Collect any necessary field reagent blanks. This sample should be collected after field staff return from an offsite break (e.g., lunch) to capture any potential cross-contamination from field personnel.
- Do not rinse PFAS sample bottles during sampling. Do not filter samples.

After Sample Collection

- Place each sample bottle in two sealed Ziploc® bags. Another brand of LDPE bag is acceptable.
- Record the label information and time of sampling in the field notes and COC. Note 'shake test' results if appropriate.
- Place groundwater samples in coolers that are durable in transportation and keep the temperature between 0 and 6°C until transported to the laboratory. **Do not use blue ice. Store PFAS samples in a separate cooler from other types of samples.**

Treat all disposable sampling materials as single use and dispose of them appropriately after sampling at each monitoring well.

8.3.4 Sediment Sampling

Before Sample Collection

- Place LDPE plastic sheeting adjacent to the sample location for use as a clean work area, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity.
- Don a new set of nitrile gloves. Do not use gloved hands to subsequently handle papers, pens, clothes, etc., before collecting samples.
- Use the HDPE bottles that are supplied by the laboratory. Make sure that the caps remain on the bottle until immediately prior to sample collection.

During Sample Collection

- Where surface water samples and sediment samples are collected at the same location, collect surface water samples first to minimize siltation.

- Collect sediment samples either manually using a stainless-steel trowel or using a petite ponar grab sampler, depending on field conditions at each sampling location during sampling program.
- Collect sediment samples from the upper 10 cm of sediment.
- For a sample to be acceptable overlying, low turbidity water must be present.
- Decant the overlying water and use a stainless-steel trowel to collect only the upper 5 centimeters (cm) of sediment.
- Collect sediment samples directly into laboratory-supplied bottles that are suitable in both material and size.
- Do not overfill the sample bottle.
- Make sure that the sample does not contain vegetation, that the sediment is undisturbed, and that the sampler shows no signs of winnowing or leaking.
- Make sure bottle caps remain in the gloved hand of the sampler until sampling is complete and caps are replaced on the bottle.
- Note the time on the sample label.
- Collect any necessary duplicates and matrix spikes.
- Collect any necessary equipment blanks. The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFAS, after the sampling equipment has been appropriately decontaminated.
- Collect any necessary field reagent blanks. This sample should be collected after field staff return from an offsite break (e.g., lunch) to capture any potential cross-contamination from field personnel.

After Sample Collection

- Place each sample bottle in two sealed Ziploc® bags. Another brand of LDPE bag is acceptable.
- Record the label information and time of sampling in the field notes.
- Place samples in coolers that are durable in transportation and keep the temperature between 0 and 6°C until transported to the laboratory. **Do not use blue ice. Store PFAS samples in a separate cooler from other types of samples.**
- Measure surface water pH, conductivity, temperature, and total dissolved solids (TDS) at each location **after** both surface water and sediment sampling is completed.

8.3.5 Surface Water Sampling

Before Sample Collection

- Place LDPE plastic sheeting adjacent to the sample location for use as a clean work area, if conditions allow. Otherwise, prevent sampling equipment from contacting the ground or other surface that could compromise sample integrity.
- Don a new set of nitrile gloves. Do not use gloved hands to subsequently handle papers, pens, clothes, etc., before collecting samples.

- Use the HDPE bottles that are supplied by the laboratory. Make sure that the caps remain on the bottle until immediately prior to sample collection.

During Sample Collection

- Avoid sampling the surface, in general.
- However, for Air Force investigations, collect samples from the water surface.
- Where surface water samples and sediment samples are collected at the same location, collect surface water samples first to minimize siltation.
- Collect surface water samples directly into laboratory-supplied bottles; wide-mouth bottles may be preferable to narrow mouth bottles for ease of surface water collection.
- Make sure bottle caps remain in the gloved hand of the sampler until sampling is complete and caps are replaced on the bottle.
- Note the time on the sample bottle.
- Collect any necessary duplicates and matrix spikes. As the laboratory should be analyzing the entire aqueous sample rather than sub-sampling, separate bottles will be required for these samples.
- Collect any necessary equipment blanks. The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFAS, after the sampling equipment has been appropriately decontaminated.
- Collect any necessary field reagent blanks. This sample should be collected after field staff return from an offsite break (e.g., lunch) to capture any potential cross-contamination from field personnel.

After Sample Collection

- Place each sample bottle in two sealed Ziploc® bags. Another brand of LDPE bag is acceptable.
- Record the label information and time of sampling in the field notes.
- Place samples in coolers that are durable in transportation and keep the temperature between 0 and 6°C until transported to the laboratory. **Do not use blue ice. Store PFAS samples in a separate cooler from other types of samples.**
- Measure surface water pH, conductivity, temperature, and TDS at each location **after** both surface water and sediment sampling.

8.4 Shipping

- If samples cannot be shipped the same day as collected, arrange an appropriate means of keeping the samples cool overnight and maintain the temperature between 0 and 10°C for the first 48 hours after collection, and then between 0 and 6°C thereafter.
- Store samples in appropriate transport bottles (coolers) with ice (Ziploc® bags for use as ice containers) with appropriate labeling. **Do not use blue ice. Store PFAS samples in a separate cooler from other types of samples.**

- Complete the appropriate procedures for COC, handling, packing, and shipping.
- Fill out and check COC Forms against the labels on the sample bottles progressively after each sample is collected.
- Place all disposable sampling materials (such as plastic sheeting, and health and safety equipment) in appropriate containers.
- Ship samples via courier service with priority overnight delivery. Tracking numbers for all shipments should be provided and recorded after they have been sent out to ensure their timely delivery.
- Do not ship samples via Fed Ex for Saturday delivery.

9 Waste Management

All rinsate should be collected in a sealed pail for disposal. Drill cuttings and purge water will be managed as specified in the Field Sampling Plan (FSP) or Work Plan, and according to state and/or federal requirements. PPE and decontaminated fluids will be contained separately and staged at the sampling location. Containers must be labeled at the time of collection. Labels will include date, location(s), site name, city, state, and description of matrix contained (e.g., soil, groundwater, PPE). General guidelines for investigation derived waste (IDW) handling and storage are set forth in a separate IDW guidance document (Arcadis 2009).

Typical waste characterization procedures include collection of a composite sample of the drill cutting material and a composite sample of the purge water for laboratory analysis. Samples are typically analyzed for disposal toxicity characteristic leaching procedure (TCLP) analysis for metals and VOCs. For PFAS, a simple leach test with neutral pH water may be more indicative of actual risk. Additionally, generators of waste are required to include analysis of other constituents that are reasonably believed to be present including (in this case) PFAS.

Waste storage and final waste disposition should be determined in the site specific workplan.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

If digital data collection isn't possible, waterproof field books should be avoided for field notes. Instead, field notes on loose paper on Masonite, plastic, or aluminum clip boards is preferred. Please note that newer Rite in the Rain® notebooks are approved for PFAS sampling. Other requirements for field notes include:

- Pens, pencils, and fine/ultra-fine point Sharpies® may be used.
- Keep field notes and writing implements away from samples and sampling materials.
- One person should conduct sampling while another records field notes.

- Do not write on sampling bottles unless they are closed.

10.1 Other Project Documentation

- Complete groundwater and/or soil sampling logs.
- Make sure COC Forms are properly completed. Verify which PFAS analytes (e.g., just PFOS and PFOA, some or all of the 537 list, etc.) are required for analysis and note on the COC.

11 Quality Assurance

Refer to quality control requirements for the project to ensure that appropriate quality assurance and quality control (QA/QC) samples are collected. When collecting QA/QC samples, the same guidelines apply as when collecting regular samples – specifically that:

- Samples should be collected in laboratory-supplied HDPE bottles;
- Bottle caps must remain in the hand of the sampler until replaced on the bottle;
- Labels must be completed after the caps have been placed back on each bottle; and
- Samples must be stored in appropriate transport bottles (coolers) with ice (Ziploc® bags for use as ice containers) with appropriate labeling. **Do not use blue ice. Store PFAS samples in a separate cooler from other types of samples.**

11.1 Equipment Blanks (if relevant)

QA/QC sampling typically includes daily collection of equipment blanks using the laboratory-supplied “PFAS-free” water. For peristaltic pump tubing, laboratory supplied “PFAS-free” water should be poured into a clean HDPE sample bottle and then pumped through new HDPE tubing using the peristaltic pump (with new silicone tubing). The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFAS, after the sampling equipment has been appropriately decontaminated. Note that an equipment blank can also be collected if an unapproved or potentially suspect hazard control is needed for health and safety and it contacts a sample, i.e., that material would be exposed to PFAS free water then the water would be collected in a separate sample container.

11.2 Field Duplicates

QA/QC sampling typically includes the collection of one field duplicate for every 10 or 20 samples collected. Each duplicate sample will be collected immediately after the initial sample of which it is a duplicate into a separate laboratory-provided sample bottle. Do not indicate to the laboratory which sample the duplicate replicates, i.e., it should be given a blind reference on the COC and sample name such as “duplicate”.

11.3 Field Reagent Blanks

QA/QC sampling for PFAS typically includes the submission of one laboratory supplied field reagent blank per day. The field reagent blank sample is brought to the site in a laboratory-supplied sample bottle. Field staff

transfer the laboratory-supplied reagent blank to an empty sample bottle. This sample should be collected after field staff return from an offsite break (e.g., lunch) to capture any potential cross-contamination from field personnel and should be placed in the same cooler as the other PFAS samples.

11.4 Matrix Spikes (optional in some cases)

QA/QC sampling includes submitting a sample to be used as a matrix spike if the project requires it. If a separate sample bottle is required, an additional sample will be collected immediately after the initial sample of which it is a duplicate into a separate laboratory-supplied sample bottle.

11.5 Laboratory Analytical QA/QC

- Arcadis recommends that any request for PFAS analysis in groundwater or soil should be conducted by an ELAP-accredited method compliant with QSM 5.3 Table B-15. Requirements laid out in Table B-15 strictly govern acceptable laboratory data quality for PFAS analysis in environmental samples. **For all data collection efforts at DoD sites, PFAS data must be obtained using a method that is DoD ELAP-accredited under QSM 5.3 or later.**
- Laboratory QA/QC should consist of one laboratory blank and one laboratory control sample (or blank spike) per batch of samples, and additional QA/QCs as indicated by the laboratory QA/QC procedures.
- Isotope dilution should be used for quantification with isotope-labeled surrogate standards, as available, according to the guidelines of QSM 5.3 Table B-15. The USEPA has two drinking water methods (USEPA Method 537.1 and USEPA Method 533). Method 537.1 does not allow for isotope dilution but USEPA Method 533 requires isotope dilution.
- For drinking water, groundwater, and surface water samples, laboratories must extract the entire sample and include a solvent rinse of the bottle for analysis. Aqueous samples should generally not be sub-sampled prior to analysis, unless they are high concentration and require serial dilution (US DoD 2017).
- Soil samples should be analyzed in their entirety or thoroughly homogenized before extraction and analysis.
- As part of the internal QA/QC of laboratory results, relative percent difference (RPD) should be calculated between samples and corresponding field or laboratory duplicates. The laboratory quality assurance portion of the laboratory certificates should be reviewed to verify that all calculations/recoveries were within acceptable limits as established by the laboratory method and guidelines in Table B-15 of QSM 5.3 or later (USDoD 2019).

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TGI - Equipment and Reagent Blank Sample Collection for PFAS Analysis

Rev: 2

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Version Control

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| -- | 0 | October 2, 2018 | All | TGI – Equipment and Reagent Blank Sample Collection for PFAS Analysis | Erika Houtz |
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Approval Signatures

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2/21/2022

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Date

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2/21/2022

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Date

1. Introduction

This document is intended to provide guidance to field staff collected equipment blanks for Per- and Polyfluoroalkyl Substances (PFAS). The content in this document describes the intended use, scope and application, personnel qualifications, equipment, cautions, health and safety considerations, procedures, waste management, data recording and management, and quality assurance of PFAS sampling.

2. Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3. Scope and Application

Equipment and reagent blanks will be collected in the field during sampling activities and submitted for laboratory analysis. These samples are primarily intended to verify that sampling and decontamination practices are effectively preventing cross-contamination caused by reusable sample equipment or other per- and polyfluoroalkyl substances (PFAS)-containing materials.

The intent of this Technical Guidance Instruction (TGI) is to provide instructions for collection of equipment and reagent blanks. More detailed instructions related to general PFAS sampling considerations is provided in the Poly- and Perfluorinated Alkyl Substances (PFAS) Field Sampling Guidance (Arcadis 2021).

4. Personnel Qualifications

Equipment and reagent samples will be collected by persons who have been trained in proper sampling procedures under the guidance of an experienced field geologist, engineer, or technician. Blank sampling should be completed with a two-person sampling team.

5. Equipment List

The following equipment and materials must be available for equipment and reagent blank sampling:

- Site plan which specifies frequency/quantity of blank sampling;
- Relevant work plan (e.g., PQAPP);
- Site Safety and Health Plan (SSHP);
- Appropriate health and safety equipment, as specified in the SSHP;
- Laboratory-provided “PFAS-free” water;
- Nitrile gloves;
- Dedicated plastic sheeting (preferably low-density polyethylene) or other clean surface to prevent sample contact with the ground;
- Pail or bucket with closable lid for excess rinse water;
- Garbage bags;
- Appropriate sample containers and labels;
 - Labeled high density polypropylene (HDPE) sample bottles: see the Poly- and Perfluorinated Alkyl Substances (PFAS) Field Sampling Guidance (Arcadis 2021) for PFAS-specific considerations;
 - Ziploc®-style bags to hold ice and samples;
 - Packing and shipping materials;
 - Chain-of-Custody (COC) Forms; see the Sample Chain of Custody Standard Operating Procedure (SOP) for reference (Arcadis 2017a);
 - Appropriate transport containers (coolers) with ice and appropriate labeling; no blue ice is to be used.
- Decontamination:
 - Equipment cleaning materials: see the Poly- and Perfluorinated Alkyl Substances (PFAS) Field Sampling Guidance (Arcadis 2021) or the Groundwater and Soil Sampling Equipment Decontamination TGI (Arcadis 2017b) as applicable;
 - An organic solvent such as isopropanol, methanol, or acetone should be used to decontaminate reusable equipment if it can be brought to the site safely. While strongly recommended, the use of solvents may be excluded for project-specific health and safety concerns. Refer to Section 7.1.1 for more details.
 - Drum labels as required for investigation-derived waste handling: see the Investigation-Derived Waste Handling and Storage TGI for details (Arcadis 2017c);

- Field Notes:
 - Pens, pencils, and/or fine point Sharpies® for writing;
 - Appropriate field forms;
 - Clipboards, field binders, field notebook, and field note pages that are not waterproof.

6. Cautions

In general, sampling techniques used for PFAS sample collection are consistent with conventional sampling techniques used in the environmental industry, but special consideration is made regarding PFAS-containing materials and cross-contamination potential. The most important consideration during PFAS-related sampling is to prevent contact between sample media and suspect PFAS sources. During collection of equipment and reagent blanks, the sampled media (i.e., PFAS-free water) should not contact anything but the sample container. New nitrile gloves should be donned after handling of any non-dedicated sampling equipment; contact with contaminated surfaces; and whenever judged necessary by field personnel. When in doubt change your gloves. More detailed instructions related to general PFAS sampling considerations is provided in the TGI - Poly- and Perfluorinated Alkyl Substances (PFAS) Field Sampling Guidance (Arcadis 2021).

7. Health and Safety Considerations

Field activities associated with equipment and reagent blank sampling will be performed in accordance with the SSHP, a copy of which will be present on site during such activities. Additional health and safety considerations can be found in TGI for PFAS Field Sampling Guidance (Arcadis 2021).

8. Procedure

The specific procedure for equipment and reagent blank sampling was developed after careful review and consideration of project data quality objectives. Procedures for equipment blank sampling and reagent blank sampling are further described in this section. Note: the laboratory has to analyze the entire sample bottle for aqueous solutions of PFASs. When collecting each blank, fill two sample bottles and instruct the lab to hold one of them in reserve. If an unacceptable detection occurs in a blank, the second bottle of sample may be analyzed. For additional sampling considerations, reference the TGI for PFAS Field Sampling Guidance (Arcadis 2021).

8.1 Blank Sampling

8.1.1 Decontamination

Prior to collecting blank samples, the applicable piece of equipment should be properly decontaminated following these steps:

- Hand Tools and Sampling Devices (including hand augers and bladder pumps)
 1. Don new pair of nitrile gloves prior to decontamination
 2. Remove o-rings and bladder (applies only to bladder pump)

3. Scrub using a plastic brush and a non-phosphate soap free of volatile organic compounds (VOCs) (e.g., Liquinox, Alconox);
4. Double-rinse in deionized or distilled water;
5. Rinse once with the site-approved organic rinsing solvent (e.g., isopropanol, methanol, acetone);
6. Rinse once with PFAS-free water;
7. Collect all rinsate in a sealed pail for disposal;
8. Allowed time to air dry prior to re-use.
9. Insert new o-rings and bladder (applies only to bladder pump)

While strongly recommended, the use of solvents may be excluded for project-specific health and safety concerns. If solvents are prohibited, then additional procedures should be evaluated by the project team. Contingencies could include the use of dedicated sampling equipment at each sampling location or amending laboratory procedures to mitigate the increased risk of cross-contamination.

The following decontamination procedure could be utilized when organic solvent use is not possible:

1. Don new pair of nitrile gloves prior to decontamination
2. Remove o-rings and bladder (applies only to bladder pump)
3. Scrub using a plastic brush and a non-phosphate soap free of VOCs (e.g., Liquinox, Alconox);
4. Single-rinse in deionized or distilled water;
5. Scrub using a plastic brush and a non-phosphate soap free of VOCs (e.g., Liquinox, Alconox);
6. Rinse twice with deionized water and once with PFAS-free water;
7. Collect all rinsate in a sealed pail for disposal;
8. Allowed time to air dry prior to re-use.
9. Insert new o-rings and bladder (applies only to bladder pump)

- Drilling Rods
 - Drive casings and other drilling equipment will be steam cleaned or replaced with new equipment between boreholes.
 - The drilling equipment will be cleaned in an area designated by the supervising engineer or geologist that is located outside of the work zone.

After verifying the piece of equipment is properly decontaminated, and after determining an equipment blank is warranted per the sampling quality assurance / quality control (QA/QC) plan, follow the specific procedures for the relevant type of equipment found in the following sections.

8.1.2 Drilling Equipment (Hand Auger or Cutting Shoe/Drill Rod)

Two field personnel should participate in the collection of the equipment blank. One person (“Field Personnel #1”) should hold the sampling bottle and collect the sample, and the second person (“Field Personnel #2”) should pour the rinse water.

The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFASs, after the sampling equipment has been appropriately decontaminated.

1. Label the laboratory-provided HDPE bottles with applicable information (e.g., sample ID, date, time, analysis required). Keep bottle lid on until immediately prior to sample collection.
2. Lay down dedicated plastic sheeting or other clean surface to prevent sample contact with the ground.
3. Place the sealable bucket or pail on top of plastic sheeting.

4. Don a new pair of nitrile gloves prior to blank collection (Field Personnel #1 and #2). Do not use gloved hands to handle other objects (e.g., papers, pens, clothes, equipment) before collecting samples.
5. Open the sample container and position the piece of clean, decontaminated sample equipment (i.e., hand auger or drilling rod/cutting shoe) above the container (Field Personnel #1). Keep the sample cap in the hand of the sampler (Field Personnel #1) until it is replaced on the bottle.
 - a. The bucket of the hand auger can be removed from the rods/handle and held manually.
 - b. The drillers should assist the field staff with removing the cutting shoe from the drill string and positioning it for sampling.
6. Slowly pour laboratory-provided “PFAS-free” water over any surface of the decontaminated sampling device that previously contacted sampled material (Field Personnel #2).
 - a. Pour water through the inside of the hand auger bucket while manually rotating the bucket so that “PFAS-free” water contacts all sides of the sampling device. Collect runoff in the sample container (Field Personnel #1), making sure that any excess “PFAS-free” water is contained in the sealable bucket or pail.
 - b. Pour water through the inside of cutting shoe while drilling contractor holds and manually rotates the shoe so that “PFAS-free” water contacts all sides of the shoe (Field Personnel #2). Collect runoff in the sample container, making sure that any excess “PFAS-free” water is contained in the sealable bucket or pail.
7. After collecting the necessary sample volume, place cap back on the sample bottle (Field Personnel #1). The bottle should be filled approximately full, but some headspace in the bottle is acceptable.
8. Collect the second bottle with the same procedure (Steps 5 to 7), if collecting a backup.
9. Record any label information that was not pre-filled out, if necessary (e.g., sample time), and place filled sample bottles in sealed Ziploc® bags. Record the label information and time of sampling in the field notes.
10. Add sample to the laboratory COC. Double check that the sample labels and COC agree. Note on the COC that one bottle should be held in reserve, if a backup bottle is collected.
11. Place sealed Ziploc® bag into the sample cooler. Store PFAS samples in separate cooler from any other types of samples.
12. Place dedicated plastic sheeting and nitrile gloves in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

8.1.3 Reusable Sediment Sampling Equipment (Stainless-Steel Hand Tools, Petite Ponar Grab Sampler)

Two field personnel should participate in the collection of the equipment blank. One person (“Field Personnel #1”) should hold the sampling bottle and collect the sample, and the second person (“Field Personnel #2”) should pour the rinse water.

The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFASs, after the sampling equipment has been appropriately decontaminated.

1. Label the laboratory-provided HDPE bottles with applicable information (e.g., sample ID, date, time, analysis required). Keep bottle lid on until immediately prior to sample collection.
2. Lay down dedicated plastic sheeting or other clean surface to prevent sample contact with the ground.
3. Place sealable bucket or pail on top of plastic sheeting.
4. Don new pair of nitrile gloves prior to blank collection (both field personnel). Do not use gloved hands to handle other objects (e.g., papers, pens, clothes, equipment) before collecting samples.

5. Open sample container and position the clean, decontaminated piece of sample equipment (i.e., hand tool, grab sampler) above the container (Field Personnel #1). Keep the sample cap in the hand of the sampler (Field Personnel #1) until it is replaced on the bottle.
6. Slowly pour the laboratory-provided “PFAS-free” water over any surface of the sampling device that contacted sampled material (Field Personnel #2).
 - a. Pour water over front and back of all decontaminated hand tools such as spoons, spatulas, and trowels so that “PFAS-free” water touches all sides of the sampling device. Collect runoff in the sample container, making sure that any excess “PFAS-free” water is contained in the sealable bucket or pail (Field Personnel #1).
 - b. Pour water through inside of the decontaminated Petite Ponar Grab Sampler while rotating the sampler (or the “PFAS-free” water container) so that “PFAS-free” water contacts all interior sides of the sampler (Field Personnel #2). Collect runoff in the sample container, making sure that any excess “PFAS-free” water is contained in the sealable bucket or pail.
7. After collecting the necessary sample volume, place cap back on the sample bottle (Field Personnel #1). The bottle should be filled approximately full, but some headspace in the bottle is acceptable.
8. Collect the second bottle with the same procedure (Steps 5 to 7), if collecting a backup.
9. Record any label information that was not pre-filled out, if necessary (e.g., sample time), and place filled sample bottles in sealed Ziploc® bags. Record the label information and time of sampling in the field notes.
10. Add the sample to the laboratory COC. Double check that the sample labels and COC agree. Note on the COC that one bottle should be held in reserve, if a backup bottle is collected.
11. Place sealed Ziploc® bag into the sample cooler. Store PFAS samples in separate cooler from any other types of samples.
12. Place dedicated plastic sheeting and nitrile gloves in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

8.1.4 Disposable Sediment Sampling Equipment (Lexan™ Liner Sleeve)

Two field personnel should participate in the collection of the equipment blank. One person (“Field Personnel #1”) should hold the sampling bottle and collect the sample, and the second person (“Field Personnel #2”) should pour the rinse water.

The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFASs, after the sampling equipment has been appropriately decontaminated.

1. Label the laboratory-provided HDPE bottles with applicable information (e.g., sample ID, date, time, analysis required). Keep the bottle lid on until immediately prior to sample collection.
2. Lay down dedicated plastic sheeting or other clean surface to prevent sample contact with the ground.
3. Place sealable bucket or pail on top of plastic sheeting.
4. Don new pair of nitrile gloves prior to blank collection (both field personnel). Do not use gloved hands to handle other objects (e.g., papers, pens, clothes, equipment) before collecting samples.
5. Open sample container and position a clean, new, and unused section of Lexan™ liner above the container (Field Personnel #1). Keep the sample cap in the hand of the sampler (Field Personnel #1) until it is replaced on the bottle.
6. Slowly pour laboratory-provided “PFAS-free” water over any surface of the sampling device that contacted sampled material (Field Personnel #2).

- a. Pour water through inside of Lexan™ liner while rotating the liner so that “PFAS-free” water contacts all interior sides of the liner. Collect runoff in the sample container, making sure that any excess “PFAS-free” water is contained in the sealable bucket or pail.
7. After collecting the necessary sample volume, place cap back on the sample bottle (Field Personnel #1). The bottle should be filled approximately full, but some headspace in the bottle is acceptable.
8. Collect the second bottle with the same procedure (Steps 5 to 7), if collecting a backup.
9. Record any label information that was not pre-filled out, if necessary (e.g., sample time), and place filled sample bottles in sealed Ziploc® bags. Record the label information and time of sampling in the field notes.
10. Add sample to the laboratory COC. Double check that the sample labels and COC agree. Note on the COC that one bottle should be held in reserve, if a backup bottle is collected.
11. Place sealed Ziploc® bag into the sample cooler. Store PFAS samples in separate cooler from any other types of samples.
12. Place dedicated plastic sheeting and nitrile gloves in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

8.1.5 Reusable Water Sampling Equipment (Peristaltic Pump, Bladder Pump, Stainless-Steel Bailer)

8.1.5.1 Peristaltic Pump

Two field personnel should participate in the collection of the equipment blank. One person (“Field Personnel #1”) should hold the sampling bottle and collect the sample, and the second person (“Field Personnel #2”) should set up the pump and pour/ transfer the blank water.

The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFASs, after the sampling equipment has been appropriately decontaminated.

1. Label the laboratory-provided HDPE bottles with applicable information (e.g., sample ID, date, time, analysis required). Keep bottle lid on until immediately prior to sample collection.
2. Lay down dedicated plastic sheeting over clean surface to prevent sample contact with the ground.
3. Don new pair of nitrile gloves prior to blank collection (both field personnel). Do not use gloved hands to handle other objects (e.g., papers, pens, clothes, equipment) before collecting samples.
4. Pour laboratory-supplied “PFAS-free” water into a clean HDPE sample bottle (Field Personnel #2).
5. Insert new HDPE tubing into the HDPE bottle containing “PFAS-free” water and connect tubing to peristaltic pump (with new silicone tubing) (Field Personnel #2).
6. Open sample container, keeping the sample cap in the hand of the sampler until it is replaced on the bottle (Field Personnel #1).
7. Turn the peristaltic pump on and slowly pump the “PFAS-free” water into the labeled sample container (Field Personnel #2).
8. After collecting the necessary sample volume, place cap back on the sample bottle (Field Personnel #1). The bottle should be filled approximately full, but some headspace in the bottle is acceptable.
9. Collect the second bottle with the same procedure (Steps 6 to 8), if collecting a backup.
10. Record any label information that was not pre-filled out, if necessary (e.g., sample time), and place filled sample bottles in sealed Ziploc® bags. Record the label information and time of sampling in the field notes.

11. Add the sample to the laboratory COC. Double check that the sample labels and COC agree. Note on the COC that one bottle should be held in reserve, if a backup bottle is collected.
12. Place sealed Ziploc® bag into the sample cooler. Store PFAS samples in separate cooler from any other types of samples.
13. Place dedicated plastic sheeting and nitrile gloves in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

8.1.5.1 Bladder Pump

1. Two field personnel should participate in the collection of the equipment blank. One person (“Field Personnel #1”) should hold the sampling bottle and collect the sample, and the second person (“Field Personnel #2”) should set up the pump and pour/ transfer the blank water.
2. The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFASs, after the sampling equipment has been appropriately decontaminated.
3. Label the laboratory-provided HDPE bottles with applicable information (e.g., sample ID, date, time, analysis required). Keep the bottle lid on until immediately prior to sample collection.
4. Lay down dedicated plastic sheeting or other clean surface to prevent sample contact with the ground.
5. Don new pair of nitrile gloves prior to blank collection (both field personnel). Do not use gloved hands to handle papers, pens, clothes, equipment, etc., before collecting samples.
6. Pour laboratory-supplied “PFAS-free” water into an approved container (to avoid PFAS cross-contamination) large enough to submerge the bladder pump.
7. After properly decontaminating the bladder pump and replacing the bladder, attach a new section of HDPE tubing to the bladder pump, long enough to hold and direct flow into the labeled sample container. Submerge the bladder pump into the approved container of “PFAS-free” water (Field Personnel #2).
8. Open sample container, keeping the sample cap in the hand of the sampler until it is replaced on the bottle (Field Personnel #1).
9. Turn the bladder pump on and slowly pump the “PFAS-free” water into the labeled sample container (Field Personnel #2).
10. After collecting the necessary sample volume, place cap back on the sample bottle. The bottle should be filled approximately full, but some headspace in the bottle is acceptable.
11. Collect the second bottle with the same procedure (Steps 6 to 8), if collecting a backup.
12. Record any label information that was not pre-filled out, if necessary (e.g., sample time), and place filled sample bottles in sealed Ziploc® bags. Record the label information and time of sampling in the field notes.
13. Add sample to laboratory COC. Double check that the sample labels and COC agree. Note on the COC that one bottle should be held in reserve, if a backup bottle is collected.
14. Place sealed Ziploc® bag into the sample cooler. Store PFAS samples in separate cooler from any other types of samples.
15. Place dedicated plastic sheeting and nitrile gloves in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

8.1.5.2 Stainless-Steel Bailer

Two field personnel should participate in the collection of the equipment blank. One person (“Field Personnel #1”) should hold the sampling bottle and collect the sample, and the second person (“Field Personnel #2”) should pour the rinse water.

The best timing to collect equipment blanks is immediately after the collection of a sample likely to contain high concentrations of PFASs, after the sampling equipment has been appropriately decontaminated.

1. Label the laboratory-provided HDPE bottles with applicable information (e.g., sample ID, date, time, analysis required). Keep bottle lid on until immediately prior to sample collection.
2. Lay down dedicated plastic sheeting or other clean surface to prevent sample contact with the ground.
3. Place sealable bucket or pail on top of plastic sheeting.
4. Don new pair of nitrile gloves prior to blank collection (both field personnel). Do not use gloved hands to handle other objects (e.g., papers, pens, clothes, equipment) before collecting samples.
5. Open sample container (Field Personnel #1) and position the bailer above the container (Field Personnel #2). Keep the sample cap in the hand of the sampler (Field Personnel #1) until it is replaced on the bottle.
6. Fill the stainless-steel bailer with enough laboratory-provided “PFAS-free” water to collect the necessary sample volume (Field Personnel #2).
7. Slowly pour laboratory-provided “PFAS-free” water from the stainless-steel bailer into the sample container (Field Personnel #2).
8. After collecting the necessary sample volume, place cap back on the sample bottle. The bottle should be filled approximately full, but some headspace in the bottle is acceptable.
9. Collect the second bottle with the same procedure (Steps 5 to 8), if collecting a backup.
10. Place filled sample bottles in sealed Ziploc® bags, record any label information that was not pre-filled out, if necessary (e.g., sample time). Record the label information and time of sampling in the field notes and sampling forms.
11. Fill out the laboratory COC and check against the labels on the Equipment Blank sample bottle(s) progressively after each Equipment Blank is collected. Note on the COC that one bottle should be held in reserve, if a backup bottle is collected.
12. Place sealed Ziploc® bag into the sample cooler. Store PFAS samples in a separate cooler from any other types of samples.
13. Place dedicated plastic sheeting and nitrile gloves in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

8.1.6 Field Reagent Blank Sampling

Two field personnel should participate in the collection of the reagent blank. One person (“Field Personnel #1”) should hold the sampling bottle and collect the sample, and the second person (“Field Personnel #2”) should pour the blank water.

This sample should be collected after field staff return from an offsite break (e.g., lunch) to capture any potential cross-contamination from field personnel.

1. Label the laboratory-provided HDPE bottles with applicable information (e.g., sample ID, date, time, analysis required). Keep the bottle lid on until immediately prior to sample collection.
2. Don new pair of nitrile gloves prior to blank collection (both field personnel). Do not use gloved hands to handle other objects (e.g., papers, pens, clothes, equipment) before collecting samples.
3. Open sample container, keeping the sample cap in the hand of the sampler (Field Personnel #1) until it is replaced on the bottle (Field Personnel #1).
4. Slowly pour laboratory-provided “PFAS-free” water from the laboratory-provided container into the sample container (“Field Personnel #2”).
5. After collecting the necessary sample volume, place cap back on the sample bottle. The bottle should be filled approximately full, but some headspace in the bottle is acceptable.

6. Collect the second bottle with the same procedure (Steps 3 to 5) if collecting a backup.
7. Record any label information that was not pre-filled out, if necessary (e.g., sample time), and place filled sample bottles in sealed Ziploc® bags. Record the label information and time of sampling in the field notes.
8. Add sample to the laboratory COC. Double check that the sample labels and COC agree. Note on the COC that one bottle should be held in reserve, if a backup bottle is collected.
9. Place sealed Ziploc® bag into the sample cooler. Store PFAS samples in separate cooler from any other types of samples.
10. Place dedicated plastic sheeting and nitrile gloves in plastic bags. These bags will be transferred into appropriately labeled waste containers for appropriate disposal.

9. Waste Management

Excess water generated during equipment and reagent blank collection procedures will be collected and contained on site in appropriate containers, (see the Investigation-Derived Waste Handling and Storage TGI for details [Arcadis 2017c]). All investigation-derived waste (IDW) generated will be placed in Department of Transportation approved containers, sealed, and labeled. Containerized IDW will be stored on site until it is profiled and subsequently transported to an approved facility for disposal or recycling. Waste manifests for all IDW suspected to have come into contact with PFAS should clearly note the presence of PFAS. Additional IDW sampling and management details will be provided in the site-specific Work Plan (QAPP addendum) and will be consistent with applicable client policies and requirements. Disposable personal protective equipment (e.g., gloves, disposable clothing, disposable equipment) will be placed in plastic bags. These bags will be transferred into appropriately labeled containers for appropriate disposal.

10. Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

If digital data collection isn't possible, waterproof field books should be avoided for field notes. Instead, field notes on loose paper on Masonite, plastic, or aluminum clip boards is preferred. Please note that newer Rite in the Rain® notebooks are approved for PFAS sampling. Other requirements for field notes include:

- Pens, pencils, and fine point Sharpies® may be used.
- Keep field notes and writing implements away from samples and sampling materials.
- Do not write on sampling bottle labels unless the sample bottle covers are tightly closed.
- Complete sampling logs in their entirety.

- Make sure COC forms are properly completed. Verify that the analysis method requested is US EPA Method 537.1 for potable water and includes the appropriate analytes desired for analysis.

11. Quality Assurance

Refer to quality control requirements for the project to ensure that appropriate QA/QC samples are collected. When collecting QA/QC samples, the same guidelines apply as when collecting regular samples – specifically:

- **Duplicate samples of each equipment blank and reagent blank should be collected and submitted to the laboratory with instructions to hold for analysis. The purpose of this sample is to provide analytical back-up in case there are any issues with the original blank sample.**
- Samples should be collected in laboratory-supplied HDPE bottles;
- Bottle caps must remain in the hand of the sampler until replaced on the bottle;
- Labels must be completed after the caps have been placed back on each bottle; and,

Samples must be stored in appropriate transport containers (coolers) with ice (Ziploc®-type bags for use as ice containers) with appropriate labeling. **Do not use blue ice. Store PFAS samples in a separate cooler from other types of samples.**

12. References

Arcadis. 2021. TGI – Poly- and Perfluorinated Alkyl Substances (PFAS) Field Sampling Guidance. Rev. 9. October 22.

Arcadis. 2017a. SOP – Sample Chain of Custody, Rev. #1. May 23.

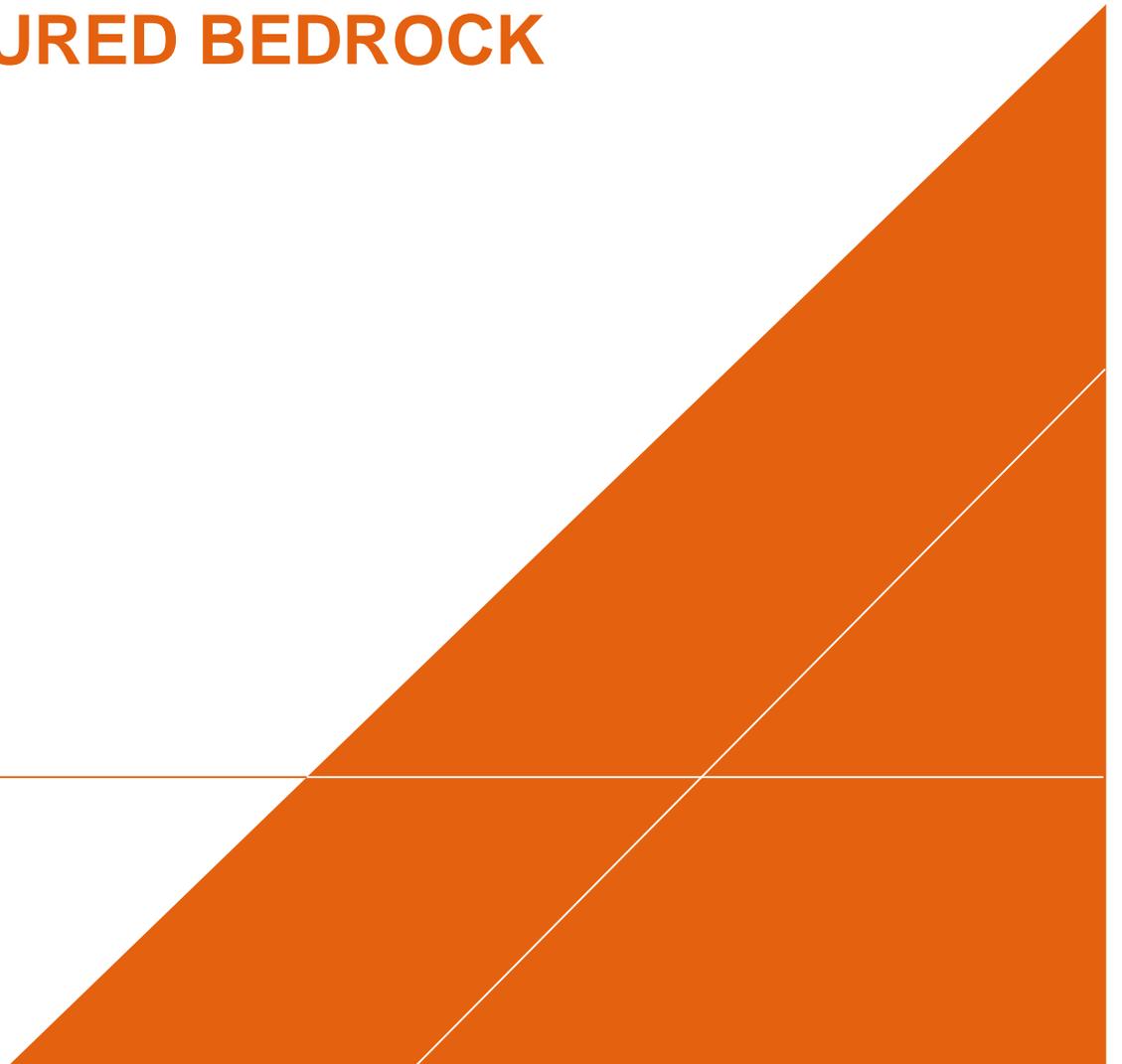
Arcadis. 2017b. TGI – Groundwater and Soil Sampling Equipment Decontamination, Rev. #0. February 23.

Arcadis. 2017c. TGI – Investigation-Derived Waste Handling and Storage, Rev. #0. February 23.

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GEOPHYSICAL INVESTIGATION TECHNICAL GUIDANCE INSTRUCTIONS FOR INVESTIGATING FRACTURED BEDROCK

October 25, 2022





1 INTRODUCTION

This document provides technical guidance instructions (TGI) for geophysical methods that will be used for conducting surface geophysical surveys to investigate bedrock conditions. The broad objective of the geophysical surveys will be to provide subsurface information about the nature of the geologic conditions in the soil and bedrock in support of groundwater investigation and remediation activities. The types of information that are germane to this work include: 1) determination of the depth to the bedrock surface, 2) characterization of the nature and variability of the soil and weathered and competent bedrock, 3) identification of relatively permeable and porous fracture zones in the bedrock to a depth of approximately 300 feet or less in depth.

The geophysical methods selected for addressing project objectives include: 1) electrical resistivity imaging (ERI) and 2) two complementary types of seismic methods, refraction and multichannel analysis of surface waves (MASW)/refraction microtremor (ReMi). The ERI and seismic data are gathered along co-located transects approximately perpendicular to groundwater flow pathways.

2 SCOPE AND APPLICATION

This TGI is intended to be used in conjunction with the field sampling plan (FSP), quality assurance project plan (QAPP), and other quality documents developed for this project. This TGI describes equipment, field procedures, and data reduction necessary to perform surface geophysical surveys.

3 PERSONNEL QUALIFICATIONS

A register senior level geologist/engineer with in depth knowledge and experience in the proposed geophysical surveying methods will oversee the performance of the geophysical surveys. Field data collection will be performed by experienced geophysical staff and supported by properly trained technicians.

4 EQUIPMENT LIST

Several types of equipment and supporting materials will be needed for geophysical surveying. The geophysical survey instruments will include:

- Advanced Geosciences, Inc. (AGI) Super Sting R8 electrical resistivity meter and switch boxes, specialized electrical resistivity cables with up to 112 individual electrodes with maximum spacing of 6 meters, and stainless steel electrode stakes for making ground contact;
- Geometrics Geode Seismograph with 48 channels, specialized seismic cable with 48 take-outs at a spacing of 20 feet (approximately 6 meters), and 48 4.5-Hz geophones;

Other supporting equipment and materials

- Deep cycle lead acid batteries to operate seismic and resistivity systems
- Rock salt for electrode contact
- Sprayers for dispensing of salt water

- Sledge hammer or other percussive source and HDPE strike plate for seismic data collection
- Differentially Corrected Global Positioning System (DGPS)
- Survey flagging (multiple colors)
- Pin flags (multiple colors)
- Wooden Stakes
- Field logbook
- Waterproof and permanent marking pens
- Multiple tape measures (300 feet minimum)

5 CAUTIONS

This TGI is intended to be used in conjunction with the field sampling plan (FSP), quality assurance project plan (QAPP), Health and Safety Plan (HASP) and other quality documents developed for this project. This TGI describes equipment, field procedures, and data reduction necessary to perform surface geophysical surveys. Since the collection of surface geophysical data generally does not expose workers to soils below the ground surface, the risk of chemical exposure is low. However, because there may be vehicles or heavy equipment in use at the site, the field personnel should participate in tail gate meetings and safety briefings.

6 HEALTH AND SAFETY CONSIDERATIONS

Adherence to the site-specific HASP and task-specific JSAs in performance of the geophysical surveys is required. In general, PPE should be assigned by the CPM and TM in adherence with Arcadis and client requirements. However, exceptions related to the specific task will be noted in the applicable JSA. For example, collection of magnetics data requires that the worker be free of metallic objects, particularly those made of iron or other magnetic materials. As a result, the worker must wear safety shoes with composite materials.

7 PROCEDURES

Electrical Resistivity

The electrical resistivity method is highly effective in the delineation of materials or interfaces that have a contrast in electrical resistivity across a vertical or horizontal boundary. Examples of types of materials with contrasting electrical resistivity are: overlying soils vs. bedrock, fresh-water sand vs. clay, and highly contaminated soils vs. background soils. The electrical resistivity method is expected to be useful at the site for the delineation of the overlying soils/bedrock interface, significant zones of fractured bedrock, and potentially chemical impacts where the groundwater has an elevated total dissolved solid signature.

The apparent resistivity for each pair of surface or borehole current/potential electrodes will be recorded using the "SuperSting." The SuperSting is a multi-channel portable memory earth resistivity meter with

memory storage of readings and user defined measure cycles. By varying the spacing between both individual and pairs of electrodes within certain types of geometrical arrays, a vertical 2D grid (X versus Z) of apparent resistivity data is obtained for each deployment. Increasing the distance between current and potential electrode pairs allows the current to travel deeper into the ground and "sample" to greater depths. These grids of data points are then interpreted for the optimal resistivity earth model that would generate the apparent measurements obtained along a given established transect.

The SuperSting has a fully automated measuring system, which allows a full suite of measurements to be taken once the electrode array has been placed in the ground. The ability to collect 2D data (i.e., at several different fixed depths and several different lateral locations) allows an interpretation of vertical changes in resistivity with depth and thus will provided an image of these changes. In addition, by collecting two parallel lines of data separated by approximately 100 feet, it is also possible to estimate the plan-view orientation of various linear features such as vertical fractures, troughs in the top of competent bedrock, and cross-cutting geologic bodies such as igneous intrusives.

Initial Layout

Prior to any data acquisition, available site maps will be reviewed to identify the locations of all utilities and subsurface features that may affect the resistivity readings. The layout of the transect locations will be initially established with engineering tapes. Each transect will be marked at a predetermined interval along its entire length with labeled pin flags. Any utilities within 30 feet of these transects will be marked on the ground so that resistivity anomalies from utilities can be identified in the data collected. Because well casings tend to create an especially strong anomaly, layout of the resistivity transects will avoid well casings by at least 30 to 50 feet if possible.

Stainless steel electrode stakes will then be placed in the ground at each station. There will be a total of up to 112 stations. The stakes will be pushed in by gloved hand if possible, otherwise as small mallet will be used to drive the stake. If there is pavement present, it will be necessary to drill a 3/4-inch diameter hole through the pavement and into the underlying soil. Once stakes are emplaced, the electrode cables will be laid out and attached to the stakes. Once the cables are attached to the stakes and the SuperSting, the manufacturer-recommended tests will be performed, including a contact resistance test. DC resistivity data will then be acquired along the transect using the appropriate array type(s) as determined by the senior professional - dipole-dipole, inverse schlumberger, and strong gradient are some of the more commonly used arrays.

Guidance on Collection and Verification of Resistivity Data

Data Collection Programming

Prior to collection of data, the decision about the which array(s) will be made. Common array types include:

- Dipole-Dipole
- Pole-Dipole
- Pole-Pole

- Schlumberger
- Inverse Schlumberger
- Strong Gradient
- Wenner

Selection of array type is primarily governed by project objectives and geologic conditions. The schlumberger, inverse schlumberger, wenner, and strong gradient arrays are sensitive to horizontal features, but are fair to poor for characterizing vertical features and have relatively sparse data sets. Dipole-dipole, pole-dipole, and pole-pole arrays are more sensitive to vertical features and also have the advantage of higher data density. In addition, these arrays take better advantage of the multichannel nature of the 8-channel SuperSting. Probably the most commonly used array type for geologic work is the dipole-dipole array, possibly supplemented with the strong gradient or inverse schlumberger array. A command file will be created using an application which generates a sequence of commands for the SuperSting. The command file contains instructions regarding which electrodes are to be switched on, and whether they are current or potential electrodes, number of measurement cycles (repeat measurements), and duration of the measurement time in seconds.

The SuperSting will record the results of the contact resistance test(s) which is conducted prior to data collection. During data collection a number of parameters are stored for each command line including: electrode positions, input current, measured voltage, apparent resistivity, measurement error, time, and date.

Contact Resistance Testing

Resistance checks should be run on the electrodes prior to data collection to assure that contact resistances are not too large. It is common practice to add saltwater around electrodes to improve contact resistance. Lowering of contact resistance improves the ability to inject current. Arcadis generally uses a cutoff 20 k Ω for surface data. Higher values may indicate that limited current can be injected for that electrode pair. It is important to witness the contact resistances and record them manually to determine the quality of contact. Note that the SuperSting automatically records the contact resistance for later use, but it is not easily reviewed in the field. Contact resistance values can provide a basis for editing data associated with particular electrodes that are malfunctioning or in poor contact with the formation.

Data Stacking

It is common practice to collect each reading several times and average the results. This procedure is referred to as "stacking." Although collection of repeat measurements increases duration of the survey, this extra time is well worthwhile.

Stacking serves to improve the signal-to-noise ratio because random noise is averaged out. In addition, the standard deviation of the repeat measurements (i.e., the stacking error) provides a means to quantify error and define data weights for inversion. Stacking errors are useful in QA/QC and form another basis for editing datasets prior to inversion.

Pulse Duration

On the SuperSting, the duration of the current injection can be selected. Pulse duration varies from 200 ms to 14.4 seconds. Lower pulse duration results in shorter data acquisition time. Pulses on the order of 200 ms may be acceptable in conductive, low-clay media; in the presence of clays, however, longer durations may be required to achieve equilibrium voltages. The length of the pulse duration can be varied, and surveys repeated, to determine the minimum duration necessary to achieve good data. By default, Arcadis generally uses a pulse duration of 1.2 seconds.

Electrical Resistivity Data Processing and Interpretation

Once the data are collected, the resistance data are inverted to make electrical resistivity images. EarthImager 2D and/or Res2DInv software packages will be used to QC, process and interpret the surface data acquired. Depending on selection of modeling and inversion parameters, these programs can be made to produce similar results; default values differ greatly, however. Selection of many of these parameters can be somewhat subjective, and guided by the geophysicist's intuition or prior knowledge of the site geology or the nature of the targets. For example, in a layered system, one might choose to apply anisotropic smoothing, which will result in a tomogram that has a layered character. For results to be reproducible, it is critical to (1) report all parameter selections and default values; (2) document the algorithm used by the software; and (3) archive a copy of the software code or executable. Justifications of parameter choices should also be documented.

Tomographic inversion results are strongly affected by selected inversion parameters and regularization criteria, especially in the presence of large measurement errors. It is instructive, therefore, to run multiple inversions in order to gain insight into the effects of different software settings. Rarely are default inversion settings appropriate. The process for selecting inversion settings should be guided by available site-specific geologic information or other geophysical data. Such information could include past geophysical results, geologic maps, and drillers logs. If inverted resistivity cross sections are inconsistent with such prior information (e.g., values from electromagnetic logs), this could indicate that settings are sub-optimal or that assumptions (e.g., 2-D heterogeneity) are violated.

An interactive graphic display of the data readings will be used to remove invalid data points or groups of readings, as necessary. One master color scheme will be chosen for all transects acquired that represents the full range of data values interpreted, such that a 2-D section acquired along one transect can be compared to a section acquired along another transect.

References

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5. Zohdy, A.A.R., Eaton, G.P., and Mabey, D.R., 1974, Application of surface geophysics to ground-water investigations: U.S. Geological Survey Techniques of Water-Resources Investigations, book 2, chap. D1, 86 p.

Seismic Methods

The combined seismic methods of refraction, MASW and ReMi will be performed concurrently with the same equipment. It is anticipated that the same transect locations will be used for both seismic and electrical resistivity.

Seismic Refraction

Background

This procedure describes a method for measuring shear and compressional wave velocities in soil and rock. The Seismic Refraction Method is applied by generating compressional waves (P) (and sometimes shear (SH)) on the land surface and measuring the travel time of the corresponding waves from the source to one or more geophones. These measurements are used to interpret subsurface conditions and materials. This travel time, along with distance between source and geophone(s), can also be interpreted to yield depth to refracting layer(s). The calculated seismic velocities can often be used to characterize some of the properties of natural and man-made subsurface materials.

This is a general procedure and does not address all the details and components of a seismic refraction survey. Please refer to the references provided for additional information.

Environmental Conditions

Seismic refraction data are affected by ground vibrations from a variety of sources. These include ambient sources such as wind, water movement (such as waves breaking on a nearby beach), natural seismic activity, and rainfall on the geophones. They also include cultural sources such as vehicular traffic, construction equipment, nearby motors, aircraft, or blasting. Frozen ground can contribute a high-velocity near-surface path that will obscure the contribution of deeper layers.

Such sources should be minimized as much as possible. Where possible, refraction data should not be collected during high winds or rain, or while vehicles are passing.

Measurement Procedure

The specific procedure varies according to the objective for the survey, the design of the survey, and the method used to define the planar refractors. These are described in more detail in other references (1 through 6).

The most important considerations are:

- Location of seismic refraction lines
- Length and orientation of lines
- Geophone spacing
- Location of shots (sources)
- Approach or interpretation method. These can include:
 - Intercept-time or crossover method
 - Delay-time methods and variations thereof
 - Reciprocal methods, including:
 - Common Reciprocal Method
 - Generalized Reciprocal Method
- Ray-tracing methods
- Tomographic methods

Of these approaches, the two methods most commonly used by for detailed refraction surveys are the Generalized Reciprocal Method (GRM) and the Tomographic Method. GRM is acknowledged to be superior to many other methods for modeling irregular dipping refractors and lateral velocity changes. Tomographic Methods are commonly used to image gradual velocity contacts and weathering profiles.

The general field procedures are as follows:

- Check for adequate space to lay out a straight line in accordance with the survey design
- Locate and position first geophone according to design and such that the location can be repeated or identified independently (the line should be referenced to absolute fiducials at several locations).
- Mark geophone locations between endpoints and available intermediate fiducials at the design spacing. Locations must be surveyed to within 5% of the geophone interval (3" for 5ft spacing, and 6" for 10ft spacing). Elevations of geophone locations may be obtained from client-provided survey or from a level survey referenced to available site reference points. A level survey, if performed, shall be closed back to the available site reference points within 0.25ft.
- Lay out geophone cable.
- Place geophones at marked locations. Geophones must be vertical and well-coupled to the ground using the spike provided. Where rock is exposed the spike may be replaced with a tripod base or rock plate.
- Test geophones and cables for shorts or open circuits.
- Set up source(s) at design locations. Shot locations must also be surveyed to within 5% of the geophone interval (3" for 5ft spacing, and 6" for 10ft spacing).
- Place trigger cable.
- Test seismic source and trigger cable.

- Input survey geometry into seismograph.
- Test noise level and set gains and filters.
- Proceed with refraction measurements. Perform forward and reverse and off-end shots as required by the interpretation method selected.

Required Field Records

- Field log for each refraction measurement describing:
 - Location of each geophone.
 - Date and time of test.
 - Tester or data recorder.
 - Description of source (location, amplitude, number of stacks).
 - Any gain or filtering by channel during recording.
 - Any deviations from test plan and action taken as a result.
 - File name as recorded on disk.
 - QA Review

Much of the above information will be automatically recorded in the seismograph header at the time of recording (gains, filtering, and survey geometry) and need not be recorded on the paper log.

Daily backup of the raw field data to a dedicated, labeled flash drive or external hard drive should be performed prior to leaving the work site.

Analysis and Interpretation

Following completion of field work, the recorded digital records are processed by computer and interactively analyzed by an experienced geophysicist to produce plots and tables of P wave velocity versus depth.

Again, the specific procedure varies according to the objective for the survey, the design of the survey, and the method used to define the planar refractors.

Arcadis uses *Geogiga Refractor* to analyze seismic refraction data (Geogiga Technology Corp., Calgary, Alberta, Canada). *Refractor* provides the intercept time method, delay time method, ABC method, and generalized reciprocal method (GRM) for seismic refraction surveys. In general, Arcadis refraction data is processed with the Generalized Reciprocal Method (GRM), one of the most advanced modeling methods currently available for seismic refraction data. Processing steps consist of loading field records into a computer, picking the travel times of first arrivals, entering shot and spread geometry, phantoming data from all shots on a line to obtain one set of forward and reverse travel time curves for each refractor, and applying the GRM to obtain a depth section (model showing different geologic units and their velocities). Preliminary interpretations are carefully verified using available geologic and drilling data and other geophysical results such as MASW and electrical resistivity.

References:

1. ASTM D5777 - 00(2006) "Standard Guide for Using the Seismic Refraction Method for Subsurface Investigation"
2. Redpath, Bruce B. "Seismic Refraction Exploration for Engineering Site Investigations", Explosive Excavation Research Laboratory, Livermore, CA, distributed by NTIS, US Dept. of Commerce, Springfield, VA 1973
3. "Geophysical Exploration for Engineering and Environmental Investigations", Technical Engineering and Design Guides as adapted from the US Army Corps of Engineers, No.23, published by ASCE Press, Reston, VA 1998
4. Dobrin, M.B. 1960 Introduction to Geophysical Prospecting. 2nd Edition. McGraw- Hill Book Co. Inc, New York
5. Telford, W.M., et al, 1976 Applied Geophysics Cambridge University Press
6. Milsom, J. 1989 Field Geophysics Open University Press, Milton Keynes

Multichannel Analysis of Surface Waves (MASW) and Refraction Microtremor (ReMi)

Background

This procedure describes a method for determining shear wave velocity (V_s) profiles, based on surface wave dispersion measurements made on the ground surface. The MASW Method consists of collecting multi-channel seismic data in the field using an active seismic source and ReMi is collected using ambient noise as the energy source. A wavefield transform is applied to the recorded seismic data to obtain the dispersion curve, followed by using iterative forward or inverse modelling to back-calculate the variation of V_s with depth.

This is a general procedure and does not address all the details and components of MASW and ReMi testing. A detailed description of the MASW and ReMi methods is given by Park, 1999a and 1999b.

Environmental Conditions

For MASW, ground vibrations from a variety of sources affect surface wave velocity measurements. These include ambient sources such as water movement (such as waves breaking on a nearby beach) and wind. Cultural noise sources such as vehicular traffic, construction equipment, rotating machinery, or blasting may also degrade data quality. When possible, MASW testing should be conducted when cultural noise levels are at a minimum. ReMi is included since it is common, especially in or near urban areas, to experience some level of ambient noise.

MASW Field Procedure

The specific procedure varies according to the objective for the survey. The most important consideration is the depth investigation. This determines the frequency range of the seismic source and length of array

required. The length of the geophone array should be, at a minimum, 2 to 3 times the desired depth of the investigation.

The MASW field layout is similar to that of the seismic refraction technique. From 24 to 48 geophones are laid out in a linear array with and connected to a multi-channel seismograph (generally the same layout as refraction is used). This technique is ideally suited to 2D V_s imaging, with data collected in a roll-along manner similar to that of the seismic reflection technique. The source is offset at a predetermined distance from the near geophone usually determined by field testing. Following are the basic steps:

- The MASW technique typically uses 24 to 48 4.5Hz geophones arranged in a linear array
- Avoid concrete slabs, utility corridors, and sewer lines, as possible
- Layout survey ropes and mark stationing as necessary. If necessary, a deviation off line up to 5% is tolerable (10m of 200m line). Optionally a total station may be used to survey sensor locations. Geophone spacing should be such that the length of the receiver array is, at a minimum, 2 to 3 times the desired depth of investigation.
- Setup seismograph. Select digitizing rate and record length to match depth/frequency desired. Be sure to turn off or minimize any filtering, except antialiasing filters.
- Acquire sample data and adjust input levels if necessary.
- Activate source for measurements. For impact sources, such as a sledgehammer, several averages are usually required - 5 to 10 is typical. Multiple source locations may be occupied for a 1-D sounding.
- Download and visually confirm data on laptop. Check every channel for bad connections, and excess noise. Store in separate files or directories with unique names. Beware of overwriting files.
- Record required information on field log or in field notebook. This includes file name, location and orientation of array, location of each sensor within the array, and any other comments.
- Stake and mark center or ends of MASW array if necessary for later surveying. Measure and record azimuth of array line if necessary.
- For ReMi data, once the MASW data collection is complete, record a long-duration record(s) using all geophones. Record length is typically at least 5 minutes in duration (at least 1 repeat is desirable).
- Backup data to a dedicated, labeled flash drive or external hard drive on a daily basis.

Data Analysis and Interpretation

The Rayleigh wave dispersion curve is obtained by a wavefield transformation of the seismic record such as the $f-k$ or $\tau-p$ transforms. These transforms are very effective at isolating surface wave energy from that of body waves. The dispersion curve is picked as the peak of the surface wave energy in slowness (or velocity) – frequency space as shown. One advantage of the MASW/ReMi technique is that the wavefield transformation may not only identify the fundamental mode but also higher modes of surface waves. At some sites, particularly those with large velocity inversions, higher surface wave modes may contain more energy than the fundamental mode. 2-D images of V_s versus depth along a profile are constructed by combining 1-D inversions of dispersion data collected at regular intervals along the profile. Arcadis uses

Geogiga Surface Plus to analyze active and passive surface wave data (Geogiga Technology Corp., Calgary, Alberta, Canada).

Required Field Records

Field log/notebook for MASW array/profiles showing:

- Location and orientation of array
- Date of test
- Field personnel
- Instrumentation
- Data acquisition parameters including record length, sample rate, receiver spacing
- For each seismic record document file name, receiver array location, source location, source type, number of source averages
- Any deviations from test plan and action taken as a result

References

1. Park, C.B., Miller, R.D. and Xia, J., 1999a, "Multimodal analysis of high frequency surface waves", Proceedings of the Symposium on the Application of Geophysics to Engineering and Environmental Problems '99, 115-121.
2. Park, C.B., Miller, R.D. and Xia, J., 1999b, "Multichannel analysis of surface waves", Geophysics, Vol 64, No. 3, 800-808.

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TGI - INVESTIGATION-DERIVED WASTE HANDLING AND STORAGE

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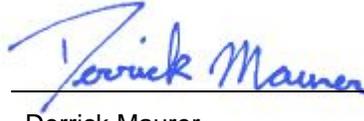


VERSION CONTROL

| Revision No | Revision Date | Page No(s) | Description | Reviewed by |
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APPROVAL SIGNATURES

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Date:

1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to any and all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, state-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

The objective of this Technical Guidance Instruction (TGI) is to describe the procedures to manage investigation-derived wastes (IDW), both hazardous and nonhazardous, generated during site activities, which may include, but are not limited to: drilling, trenching/excavation, construction, demolition, monitoring well sampling, soil sampling, decontamination and remediation. For the purposes of this TGI, IDW is considered to be discarded materials which are defined as solid waste by United States Environmental Protection Agency (EPA) standard 40 CFR § 261.2 (which may include liquids, solids, or sludges). IDW may include soil, groundwater, drilling fluids, decontamination liquids, as well as contaminated personal protective equipment (PPE), sorbent materials, construction and demolition debris, and disposable sampling materials. Hazardous or uncharacterized IDW will be collected and staged at the point of generation. Quantities small enough to be containerized in 55-gallon drums will be taken to a designated temporary onsite storage area (discussed in further detail under Drum Storage) pending characterization and disposal. IDW materials will be characterized using process knowledge and appropriate laboratory analyses to determine the waste classification and evaluate proper safe handling and disposal methods.

This TGI describes the necessary equipment, field procedures, materials, regulatory references, and documentation procedures necessary for proper handling and storage of IDW up to the time it is properly transported from the project site and disposed. The procedures included in this TGI for handling and temporary storage of IDW are based on the EPA's guidance document *Guide to Management of Investigation Derived Wastes* (USEPA, 1992). IDW is assumed to be contaminated with the site constituents of concern (COCs) until analytical evidence indicates otherwise. IDW will be managed to ensure the protection of human health and the environment and will comply with all applicable or relevant and appropriate requirements (ARAR). Although not comprehensive, the following laws and regulations on Hazardous Waste Management should be considered as potential ARAR. It is the Arcadis Certified Project Manager (CPM) and/or designated Technical Expert to determine which laws and regulations, at all levels of government, are applicable to each project site and activity falling under this TGI.

Federal Laws and Regulations

- Resource Conservation and Recovery Act (RCRA) 42 USC § 6901-6987.
- Federal Hazardous Waste Regulations 40 CFR § 260-265

Department of Transportation (DOT) Hazardous Materials Transportation 49 CFR

Occupational Safety and Health Administration (OSHA) Regulations 29 CFR

State Laws and Regulations

- To be determined based on location of site and location of treatment, storage, and/or disposal facility (TSDF) to be utilized.

Regional, County, Municipal, and Local Regulations

- To be determined based on location of site and location of treatment, storage, and/or disposal facility (TSDF) to be utilized.

Initial Storage

Pending characterization, IDW will be temporarily stored appropriately within each area of contamination (AOC). Under RCRA, "storage" is defined as the "holding of hazardous waste for a temporary period, at the end of which the hazardous waste is treated, disposed of, or stored elsewhere" (40 CFR § 260.10). The onsite waste staging area will be in a secure and controlled area. Uncharacterized wastes are considered potentially hazardous wastes and must be stored in DOT approved packaging. Liquid wastes must be stored in DOT approved closed head drums or other approved containers (e.g., portable tank containers) that are compatible with the type of material stored therein. Solid materials must be stored in DOT approved open head drums where practicable. Larger quantities of solid IDW can be containerized in bulk containers (such as in a roll-off box). Soil from large excavation projects may be managed in stockpiles with within the AOC and does not need to be containerized until exiting the AOC.

Characterization

Waste characterization can either be based on generator knowledge, such as using historical process knowledge and safety data sheets (SDS), or can be based upon characterization sampling analytical results. IDW typically is not characterized using SDS as it is a mixture of aged chemicals and environmental media. Historical process knowledge should be used to determine if the IDW is a listed hazardous waste (40 CFR § 261.31-33). If the IDW is not a listed hazardous waste, waste

characterization can be completed by laboratory analysis of representative samples of the IDW. The laboratory used for waste characterization analysis must have the appropriate state and federal accreditations and may be required to be pre-approved by the Client. IDW will be classified as RCRA hazardous or non-regulated under RCRA based on the waste characterization determination.

If IDW is characterized as RCRA hazardous waste, RCRA and DOT requirements must be followed for packaging, labeling, transporting, storing, and record keeping as described in 40 CFR § 262 and 49 CFR § 171-178. Waste material classified as RCRA nonhazardous may be handled and disposed of as nonhazardous waste in accordance with applicable federal, state, and local regulations.

Storage Time Limitations

Containerized hazardous wastes can be temporarily stored for a maximum of 90 calendar days from the accumulation start date for a large quantity generator or a maximum of 180 calendar days from the accumulation start date for a small quantity generator. Wastes classified as nonhazardous may be handled and disposed of as nonhazardous waste and are not subject to storage time limitations.

This TGI may be modified by the CPM and/or Technical Expert for a specific project or client program, as required, dependent upon client requirements, site conditions, equipment limitations, or limitations imposed by the procedure. The resulting procedure employed to execute the work will be documented in the project work plans or reports. If changes to the sampling procedures are required due to unanticipated field conditions, the changes will be discussed with the CPM and/or Technical Expert as soon as practicable, and if approved to be performed, be documented.

3 PERSONNEL QUALIFICATIONS

Arcadis field sampling personnel will have current regulatory- and Arcadis-required health and safety training including 40-hour HAZWOPER training, site supervisor training, site-specific training, first aid, and cardiopulmonary resuscitation (CPR), as needed. Personnel handling and packaging hazardous waste and performing hazardous waste characterizations must have RCRA hazardous waste management training per 40 CFR § 264.16. Additional state-specific hazardous waste management training is required in certain states (i.e., California).

Although not common practice, in certain situations Arcadis personnel may sign waste profiles and/or waste manifests on a case by case basis for clients, provided the appropriate agreement is in place between Arcadis and the client documenting that Arcadis is not the generator, but is acting as an authorized representative of the generator. Arcadis personnel who sign waste profiles and/or waste manifests will have both current RCRA hazardous waste management training per 40 CFR § 264.16 and current DOT hazardous materials transportation training per 49 CFR § 172.704. Arcadis field personnel will also comply with client-specific training. In addition, Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and Technical Guidance Instructions (TGIs) and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. The project health and safety plan (HASP) and other documents will identify other training requirements or access control requirements.

4 EQUIPMENT LIST

The Following Materials, as required, will be available for IDW handling and Storage:

- Appropriate personal protective equipment as specified in the Site Health and Safety Plan (HASP)
- DOT approved containers
- Hammer
- Leather gloves
- Drum dolly
- Appropriate drum labels (outdoor waterproof self-adhesive)
- Portable tank container
- Appropriate labeling, packing, chain-of-custody forms, and shipping materials as determined by the CPM and/or Technical Expert.
- Indelible ink and/or permanent marking pens
- Plastic sheeting
- Appropriate sample containers, labels, and forms
- Stainless-steel bucket auger
- Stainless steel spatula or knife
- Stainless steel hand spade
- Stainless steel scoop
- Digital camera
- Field logbook

5 CAUTIONS

Filled drums can be very heavy, become unbalanced, or spill its contents. Therefore, use appropriate moving techniques and equipment for safe handling. Similar media (e.g. soils with other soils; or liquids with other liquids) will be stored in the same drums to aid in sample analysis and disposal. Drum lids must be secured to prevent rainwater from entering the drums and leakage during movement. Drums containing solid material may not contain any free liquids. Waste containers stored for extended periods of time may be subject to deterioration. Drum Over Packs may be used as secondary containment. All drums must be visually inspected for condition to ensure that they are in good condition without visible evidence of rusting, holes, breakage, etc., to prevent potential leakage and facilitate subsequent disposal. All drum lids must be verified as having a properly functioning secured lid prior to use.

6 HEALTH AND SAFETY CONSIDERATIONS

As determined by the site's known and suspected hazards, appropriate PPE must be worn by all field personnel within the designated work area. Exposure air monitoring may be required during certain field activities as required in the Site Health and Safety Plan. If soil excavation in areas with potentially hazardous contaminants is possible, contingency plans will be developed to address the potential for encountering gross contamination or non-aqueous phase liquids. All excavation activities shall be in compliance with OSHA standard 29 CFR 1926.651 Excavations, and any other applicable regulations.

Arcadis field personnel and subcontractors will be trained in and perform their work in compliance with all applicable federal, state, and local health and safety regulations as well as Arcadis' HASP and applicable Client health and safety requirements.

7 PROCEDURE

Specific waste temporary storage and handling procedures to be used are dependent upon the type of generated waste, including type of media (e.g. soils or free liquids) and constituents of concern. For this reason, IDW can be stored in a secure location onsite in separate 55-gallon storage drums, where solids can be stockpiled onsite (if nonhazardous) and purge water may be stored in portable tank containers. Waste materials such as broken sample bottles or equipment containers and wrappings will be stored in 55-gallon drums unless they were not in contact with sample media.

Management of IDW

Minimization of IDW should be considered by the project team during all phases of the project. Site managers may want to consider techniques such as replacing solvent based cleaners with aqueous-based cleaners for decontamination of equipment, reuse of equipment (where it can be properly decontaminated), limitation of traffic between exclusion and support zones, and drilling methods and sampling techniques that minimize the generation of waste. Alternative drilling and subsurface sampling methods may include the use of small diameter boreholes, as well as borehole testing methods such as a core penetrometer or direct push technique instead of coring.

Drum Storage

Drums containing hazardous waste will be stored in accordance with the requirements of 40 CFR 265 Subpart I (for containers) and 265 Subpart DD (for containment buildings). All 55-gallon drums will be stored at a secure, centralized onsite location that is readily accessible for vehicular pick-up. Drums confirmed as, or assumed to contain hazardous waste will be stored over an impervious surface provided with secondary spill containment. The storage location will, for drums containing liquid, have a containment system that can contain at least the larger of 10% of the aggregate volume of staged materials or 100% of the volume of the largest container. Drums will be closed during storage and be in good condition in accordance with the Guide to Management of Investigation-Derived Wastes (USEPA, 1992).

Hazardous Waste Determination

Waste material must be characterized to determine if it meets any of the federal definitions of hazardous waste as required by 40 CFR § 262.11. If the waste does not meet any of the federal definitions, it must then be established if any state-specific or local-specific hazardous waste criteria exist/apply.

Generator Status

Once hazardous waste determination has been made, the generator status will be determined. Large quantity generators (LQG) are generators who generate more than 1,000 kilograms of hazardous waste in a calendar month. Small quantity generators (SQG) of hazardous waste are generators who generate greater than 100 kilograms but less than 1,000 kilograms of hazardous waste in a calendar month. Very small quantity generators (VSQG) are generators who generate less than 100 kilograms of hazardous

waste per month. Please note that a generator status may change from month to month and that a notice of this change is usually required by the generator's state agency.

Accumulation Time for Hazardous Waste

A LQG may accumulate hazardous waste on site for 90 calendar days or less without a permit and without having interim status, provided that such accumulation is in compliance with requirements in 40 CFR § 262.17. A SQG may accumulate hazardous waste on site for 180 calendar days or less without a permit or without having interim status, subject to the requirements of 40 CFR § 262.16. VSQG requirements are found in 40 CFR § 262.14. NOTE: The federal VSQG and SQG provisions may not be recognized by some states (e.g., California and Rhode Island). State-specific and local-specific regulations must be reviewed and understood prior to the generation of hazardous waste.

Satellite Accumulation of Hazardous Waste Satellite accumulation (SAA) will mean the accumulation of as much as fifty-five (55) gallons of hazardous waste, or the accumulation of as much as one quart of acutely hazardous waste, in containers at or near any point of generation where the waste initially accumulates, which is under the control of the operator of the process generating the waste, without a permit or interim status and without complying with the requirements of 40 CFR § 262.15 and without any storage time limit, provided that the generator complies with 40 CFR § 262.15.

Once more than 55 gallons of hazardous waste accumulates in SAA, the generator has three days to move this waste into storage.

Storage recommendations for hazardous waste include:

- Ignitable or reactive hazardous wastes must be >50 feet from the property line per 40 CFR § 265.176 (LQG generators only).
- Hazardous waste should be stored on a concrete slab (asphalt is acceptable if there are no free liquids in the waste).
- Drainage must be directed away from the accumulation area.
- Area must be properly vented.
- Area must be secure.

Drum/Container Labeling

Drums will be labeled on both the side and lid of the drum using a permanent marking pen. Old drum labels must be removed to the extent possible, descriptions crossed out should any information remain, and new labels affixed on top of the old labels. Other containers used to store various types of waste (e.g., polyethylene tanks, roll-off boxes, end-dump trailers, etc.) will be labeled with an appropriate "Waste Container" or "Testing in Progress" label pending characterization. Drums and containers will be labeled as follows:

- Appropriate waste characterization label (Pending Analysis, Hazardous, or Nonhazardous)
- Waste generator's name (e.g., client name)
- Project Name
- Name and telephone number of Arcadis project manager
- Composition of contents (e.g., used oil, acetone 40%, toluene 60%)
- Media (e.g., solid, liquid)
- Accumulation start date

- Drum number of total drums as reconciled with the Drum Inventory maintained in the field log book.

IDW containers will remain closed except when adding or removing waste. Immediately upon beginning to place waste into the drum/container, a "Waste Container" or "Pending Analysis" label will be filled out to include the information specified above, and affixed to the container. Once the contents of the container are identified as either non-hazardous or hazardous, the following additional labels will be applied.

- Containers with waste determined to be non-hazardous will be labeled with a green and white "Nonhazardous Waste" label over the "Waste Container" label.
- Containers with waste determined to be hazardous will be stored in an onsite storage area and will be labeled with the "Hazardous Waste" label and affixed over the "Waste Container" label.

The ACCUMULATION DATE for the hazardous waste is the date the waste is first placed in the container and is the same date as the date on the "Waste Container" label. DOT hazardous class labels must be applied to all hazardous waste containers for shipment offsite to an approved disposal or recycling facility. In addition, a DOT proper shipping name will be included on the hazardous waste label. The transporter should be equipped with the appropriate DOT placards. However, placarding or offering placards to the initial transporter is the responsibility of the generator per 40 CFR § 262.33.

Inspections and Documentation

All IDW will be documented as generated on a Drum Inventory Log maintained in the field log book. The Drum Inventory will record the generation date, type, quantity, matrix and origin (e.g., Boring-1, Test Pit 3, etc.) of materials in every drum, as well as a unique identification number for each drum. The drum inventory will be used during drum pickup to assist with labeling of drums. The drum storage area and any other areas of temporarily staged waste, such as soil/debris piles, will be inspected weekly. The weekly inspections will be recorded in the field notebook or on a Weekly Inspection Log. Digital photographs will be taken upon the initial generation and drumming/staging of waste, and final labeling after characterization to document compliance with labeling and storage protocols, and condition of the container. Evidence of damage, tampering or other discrepancy should be documented photographically.

Emergency Response and Notifications

Specific procedures for responding to site emergencies will be detailed in the HASP. If the generator is designated as a LQG, a Contingency Plan will need to be prepared to include emergency response and notification procedures per 40 CFR § 265 Subpart D. In the event of a fire, explosion, or other release which could threaten human health outside of the site or when Client or Arcadis has knowledge of a spill that has reached surface water, Client or Arcadis must immediately notify the National Response Center (800-424-8802) in accordance with 40 CFR § 262.265. Other notifications to state and/or other local regulatory agencies may also be necessary.

Drilling Soil Cuttings and Muds

Soil cuttings are solid to semi-solid soils generated during trenching activities, subsurface soil sampling, or installation of monitoring wells. Depending on the drilling method, drilling fluids known as "muds" may be used to remove soil cuttings. Drilling fluids flushed from the borehole must be directed into a settling section of a mud pit. This allows reuse of the decanted fluids after removal of the settled sediments. Soil cuttings will be labeled and stored in 55-gallon drums with bolt-sealed lids.

Excavated Solids

Excavated solids may include, but are not limited to: soil, fill, and construction and demolition debris. Prior to permitted treatment or offsite disposal, potentially hazardous excavated solids may be temporarily stockpiled onsite as long as the stockpile remains in the same AOC from where it was excavated. Potentially hazardous excavated solids removed from the AOC must be immediately containerized in labeled drums or closable top roll-offs lined with 9-mil polyvinyl chloride (PVC) sheeting and are subject to LQG storage time limits. Nonhazardous excavated solids can be stockpiled either inside or outside of the AOC, do not have to be containerized and are not subject to hazardous waste regulations. Potentially hazardous excavated solids must not be mixed with nonhazardous excavated solids. All classes of excavated solid stockpiles should be maintained in a secure area onsite. At a minimum, the floor of the stockpile area will be covered with a 20-mil high density polyethylene liner that is supported by a foundation or at least a 60-mil high density polyethylene liner that is not supported by a foundation. The excavated material will not contain free liquids. The owner/operator will provide controls for windblown dispersion, run-on control, and precipitation runoff. The run-on control system will prevent flow onto the active portion of the pile during peak discharge from at least a 25-year storm and the run-off management system will collect and control at least the water volume resulting from a 24-hour, 25-year storm (USEPA, 1992). Additionally, the stockpile area will be inspected on a weekly basis and after storm events. Individual states may require that the stockpile be inspected/certified by a licensed professional engineer. Stockpiled material will be covered with a 6-mil polyvinyl chloride (PVC) liner or sprayed dust control product. The stockpile cover will be secured in place with appropriate material (concrete blocks, weights, etc.) to prevent the movement of the cover.

Decontamination Solutions

Decontamination solutions are generated during the decontamination of personal protective equipment and sampling equipment. Decontamination solutions may range from detergents, organic solvents and acids used to decontaminate small field sampling equipment to steam cleaning rinsate used to wash heavy field equipment. These solutions are to be labeled and stored in closed head drums compatible with the decontamination solution. Decontamination procedures, including personnel and field sampling equipment, must comply with applicable Arcadis procedural documents.

Disposable Equipment

Disposable equipment includes personal protective equipment (e.g., tyvek coveralls, gloves, booties and APR cartridges) and disposable sampling equipment such as trowels or disposable bailers. If the media sampled exhibits hazardous characteristics per results of waste characterization sampling, contaminated disposable equipment will also be disposed of as a hazardous waste. If compatible with the original IDW waste stream (i.e., the IDW is a solid and the disposal equipment is a solid), the disposable equipment can be combined with the IDW. If these materials are not compatible (i.e., the IDW is a liquid and the disposal equipment is a solid), the disposable equipment will be stored onsite in separate labeled 55-gallon drums. Uncontaminated or decontaminated disposable equipment can be considered nonhazardous waste.

Purge Water

Purge water includes groundwater generated during well development, groundwater sampling, or aquifer testing. The volume of groundwater generated will dictate the appropriate storage procedure. Monitoring

well development and groundwater sampling may generate three well volumes of groundwater or more. This volume will be stored in labeled 55-gallon drums. Aquifer tests may generate significantly greater volumes of groundwater depending on the well yield and the duration of the test. Therefore, large-volume portable polyethylene tanks will be considered for temporary storage pending groundwater-waste characterization.

Purged Water Storage Tank Decontamination and Removal

The following procedures will be used for inspection, cleaning, and offsite removal of storage tanks used for temporary storage of purge water. These procedures are intended to be used for rented portable tanks such as Baker Tanks or Rain for Rent containers. Storage tanks will be made of inert plastic materials. The major steps for preparing a rented tank for return to a vendor include characterizing the purge water, disposing of the purge water, decontaminating the tank, final tank inspection, and mobilization. Decontamination and inspection procedures are described in further detail below.

- **Tank Cleaning:** Most vendors require that tanks be free of any visible sediment and water before returning, a professional cleaning service may be required. Each specific vendor should be consulted concerning specific requirements for returning tanks.
- **Tank Inspection:** After emptying the tank, purged water storage tanks should be inspected for debris, chemical staining, and physical damage. The vendors require that tanks be returned in the original condition (i.e., free of sediment, staining and no physical damage).

8 WASTE MANAGEMENT

Soil/Solids Characterization

Waste characterization will be conducted in accordance with waste hauler, waste handling facility, and local/state/federal requirements. In general, RCRA hazardous wastes are those solid wastes determined by a Toxicity Characteristic Leaching Procedure (TCLP) test or to contain levels of certain toxic metals, pesticides, or other organic chemicals above specific applicable regulatory agency thresholds. If the one or more of 40 toxic compounds listed in Table I of 40 CFR § 261.24 are detected in the sample at levels above the maximum unregulated concentrations, the waste must be characterized as a toxic hazardous waste. Wastes can also be considered “listed” hazardous waste depending on site-specific processes.

Composite soil samples will be collected at a frequency of one sample per 250 cubic yard basis for stockpiled soil or one per 55-gallon drum per different waste stream for containerized. A four-point composite sample will be collected per 250 cubic yards of stockpiled material and for each drum waste stream. Sample and composite frequencies may be adjusted in accordance with the waste handling facility’s requirements and may be reduced for large volumes of waste with consistent properties. Waste characterization samples will be considered valid for consistent waste streams for a period of 1 year. Waste characterization samples may be analyzed for the TCLP volatile organic compounds (VOCs), TCLP semi-volatile organic compounds (SVOCs), TCLP RCRA metals, and polychlorinated biphenyls (PCBs), as well as reactivity and flammability (flashpoint). Additional samples may be collected and analyzed by the laboratory on a contingency basis. Site-specific constituents of concern including pesticides may require additional sampling. Please note that state- or local-specific regulations may require a different or additional sampling approaches.

Wastewater Characterization

Waste characterization will be conducted in accordance with the requirements of the waste hauler, waste handling facility, and local/state/federal governments. In general, purge water should be analyzed by methods appropriate for the known contaminants, if any, that have been historically detected in the monitoring wells. Samples will be collected and analyzed in accordance with the requirements of the waste disposal facility. Wastewater characterization samples may be analyzed for TCLP volatile organic compounds (VOCs), TCLP semi-volatile organic compounds (SVOCs), TCLP RCRA metals, and polychlorinated biphenyls, as well as corrosivity (pH), reactivity and flammability (flashpoint). Additional samples may be collected and analyzed by the laboratory on a contingency basis. Site-specific constituents of concern including pesticides may require additional sampling. Please note that state- and/or local-specific regulations may require different or additional sampling approaches.

Sample Handling and Shipping

All samples will be appropriately labeled, packed, and shipped, and the chain-of-custody will be filled out in accordance with current Arcadis sample chain of custody, handling, packing, and shipping procedures and guidance instructions.

It should be noted that additional training is required for packaging and shipping of hazardous and/or dangerous materials. Please refer to the current Arcadis training requirements related to handling and shipping of samples, shipping determinations, and hazardous materials.

Preparing Waste Shipment Documentation (Hazardous and Nonhazardous)

Waste profiles will be prepared by the Arcadis CPM and forwarded, along with laboratory analytical data to the Client for approval/signature. The Client will then return the profile to Arcadis who will then forward to the waste removal contractor for preparation of a manifest. The manifest will be reviewed by Arcadis prior to forwarding to the Client for approval. Upon approval of the manifest, the Client will return the original signed manifest directly to the waste contractor or to the Arcadis CPM for forwarding to the waste contractor. Arcadis personnel may sign waste profiles and/or waste manifests on a case by case basis for clients, provided the appropriate agreement is in place between Arcadis and the client documenting that Arcadis is not the generator, but is acting as an authorized representative of the generator.

Final drum labeling and pickup will be supervised by an Arcadis representative who is trained and experienced with applicable waste labeling procedures. The Arcadis representative will have a copy of the drum inventory maintained in the field book and will reconcile the drum inventory with the profile numbers on the labels and on the manifest. Different profile numbers will be generated for different matrices or materials in the drums. For example, the profile number for drill cuttings will be different than the profile number for purge water. When there are multiple profiles it is critical that the proper label, with the profile number appropriate to a specific material be affixed to the proper drums. A copy of the Arcadis drum inventory will be provided to the waste transporter during drum pickup and to the facility receiving the waste.

9 DATA RECORDING AND MANAGEMENT

Waste characterization sample handling, packing, and shipping procedures will be documented in accordance with relevant Arcadis procedures and guidance instructions as well as applicable client and/or project requirements, such as a Quality Assurance Project Plan or Sampling and Analysis Plan. Copies of the chain-of-custody forms will be maintained in the project file. Arcadis should photograph or maintain a copy of any hazardous waste manifest signed on behalf of Client in the corresponding office DOT record file.

10 QUALITY ASSURANCE

The CPM or APM will review all field documentation once per week for errors or omissions as compared to applicable project requirements including but not limited to: the proposal/scope of work, QAPP, SAP, HASP, etc. Deficiencies will be noted, tracked, and resolved. Upon correction, they will be noted for project documentation.

11 REFERENCES

United States Environmental Protection Agency (USEPA). 1992. Guide to Management of Investigation-Derived Wastes. Office of Remedial and Emergency Response. Hazardous Site Control Division. January 1992.



TGI – Soil Drilling and Sample Collection

Rev: #3

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Version Control

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| | 1 | May 12, 2020 | None | Review – no changes necessary | Marc Killingstad |
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Approval Signatures

Prepared by:

4/5/2023



Chris Shepherd (Preparer)

Date

Reviewed by:

4/5/2023



Marc Killingstad (Subject Matter Expert)

Date

1 Introduction

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to any and all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, state-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM. All deviations or omissions should be documented.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

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3 Scope and Application

This Technical Guidance Instruction (TGI) describes general drilling procedures and the methods to be used to field screen and collect soil samples for laboratory analysis in unconsolidated or weakly consolidated sediments. For soil description procedures, please refer to the *TGI - Soil Description*. For monitoring well installation in granular aquifers, please refer to the *TGI - Monitoring Well Installation*. For per- and polyfluoroalkyl substances (PFASs) drilling and soil sampling procedures, please refer to: *TGI – PFAS-Specific Drilling and Monitoring Well Installation*, *TGI – Per- and Polyfluoroalkyl Substances (PFAS) Field Sampling Guide*, and *TGI – Equipment and Reagent Blank Sample Collection for PFAS Analysis*.

Overburden (unconsolidated sediments) drilling is commonly performed using the hollow-stem auger drilling method. Other drilling methods suitable for overburden drilling, which are sometimes necessary due to site-specific geologic conditions, include: direct-push, drive-and-wash, spun casing, rotasonic, dual-rotary (Barber Rig), and fluid/mud rotary with core barrel or roller bit. Direct-push techniques (e.g., Geoprobe or cone penetrometer) and hand tools may also be used. Drilling within consolidated materials such as fractured rock is commonly performed using water-rotary (coring or tri-cone roller bit), air rotary or rotasonic methods. For guidance when drilling in consolidated materials (i.e., bedrock), please refer to *the TGI – Bedrock Core Collection and Description*.

The drilling method to be used at a given site will be selected based on site-specific consideration of anticipated drilling depths, targeted chemicals, site or regional geologic knowledge, types of sampling to be conducted, required sample quality and volume, and cost.

Field screening of soil samples is commonly performed using a photoionization detector (PID) and/or a flame ionization detector (FID). These instruments are used to measure relative concentrations of volatile organic compounds (VOCs) for the selection of samples for further laboratory or field analysis. Field screening for dense non-aqueous phase liquids (DNAPL) may be performed using hydrophobic dye (Oil Red O or Sudan IV), which is pertinent at chlorinated solvent sites.

Collection of soil samples for laboratory analysis may be performed using a variety of techniques including grab samples, undisturbed cores, and composite or homogenized samples. Samples may require homogenization across a given depth interval, or several discrete grabs (usually five) may be combined into a composite sample. Samples for VOC analysis will not be homogenized or composited and are collected as discrete grab samples.

No oils or grease will be used on equipment introduced into the boring (e.g., drill rod, casing, or sampling tools). Some lubricants (e.g., vegetable oil-based lubricants) may be acceptable, if the constituents won't interfere with the analyses.

4 Personnel Qualifications

Arcadis field personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or state/federal regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as identified in the site-specific Health and Safety Plan (HASP) which may include first aid,

cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. The HASP will also identify any access control requirements.

Prior to mobilizing to the field, Arcadis field personnel will review and be thoroughly familiar with relevant site-specific documents including but not limited to the task-specific work plan or field implementation plan (FIP), Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Arcadis field personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. Personnel responsible for overseeing drilling operations will have at least 16 hours of prior training overseeing drilling activities with an experienced geologist, environmental scientist, or engineer with at least 2 years of prior experience.

Arcadis personnel directing, supervising, or leading soil sampling activities will have a minimum of 1 year of previous environmental soil sampling experience. Field employees with less than 6 months of experience will be accompanied by a supervisor (as described above) to ensure that proper sample collection techniques are employed.

Additionally, the Arcadis field team will review and be thoroughly familiar with documentation provided by equipment manufacturers and become familiar with the operation of (i.e., hands-on experience) all equipment that will be used in the field prior to mobilization.

5 Equipment List

The following materials will be available, as required, during soil boring drilling, field screening, and sampling activities:

- Site-specific HASP and health and safety documents identified in the HASP
- FIP/work plan that includes site map with proposed boring locations, field sampling plan (with corresponding depths, sample analyses, sample volume required, and sample holding time), and previous boring logs (as available)
- Appropriate personal protective equipment (PPE), as specified in the HASP
- Including but not limited to disposable chemical resistant gloves and Level D PPE
- Traffic cones, delineators, and caution tape as appropriate for securing the work area as specified in the Traffic Safety Plan (TSP)
- Photoionization detector (PID), flame ionization detector (FID) or other air/soil screening equipment, as needed, in accordance with the HASP or workplan
- Sampling equipment:
- Drilling equipment required by *ASTM D1586*, when performing split-spoon sampling including clean sample sleeves
- Disposable plastic liners, when drilling with direct-push equipment
- Stainless steel hand auger and stainless-steel spade if using manual methods
- Appropriate soil sampling equipment (e.g., stainless steel spatulas/spoons/bowls, knife)
- Sealable plastic bags (e.g., Ziploc®)

- Air-tight sample containers and 8-oz. glass Mason jars or driller's jars
- Aluminum foil
- Appropriate sample blanks (trip blank supplied by the laboratory), as specified in the FSP
- Soil sample containers and labels (supplied by the laboratory) appropriate for the analytical method(s) with preservative, as needed (parameter-specific)
- Sample labels
- Indelible ink pens
- Engineer's ruler or survey rod
- Plastic sheeting (e.g., Weatherall Visqueen)
- Appropriate transport containers (coolers) with ice and appropriate labeling, packing, and shipping materials
- Decontamination equipment (buckets, distilled or deionized water, cleansers appropriate for removing expected chemicals of concern, paper towels) in accordance with the *TGI for Groundwater and Soil Sampling Equipment Decontamination*
- Forms/notes:
 - Tablet with digital forms, etc., if appropriate
 - Appropriate soil boring log (**Attachment 1**)
 - Chain-of-custody forms
 - Field notebook
 - Digital camera (or smart phone with camera)
- Drums or other containers appropriate for soil and decontamination water, as specified by the site investigation-derived waste (IDW) management plan, and appropriate drum labels

6 Cautions

Prior to beginning field work, underground utilities in the vicinity of the drilling areas will be delineated by the drilling contractor or an independent underground utility locator service in accordance with the work plan, client requirements, and Arcadis guidance. See appropriate guidance for proper utility clearance protocol. Work will be performed in accordance with the Arcadis *Utility Location and Clearance Health and Safety Standard* and the *Utilities and Structures Checklist* will be completed before beginning any intrusive work.

Prior to beginning field work, the project technical team will ensure that all field logistics (e.g., access issues, health and safety issues, communication network, schedules, etc.) and task objectives are clearly understood by all team members. An internal call with the project technical team to review the FIP/work plan scope and objectives is strongly recommended prior to mobilization to ensure that the field work will be effectively and efficiently executed.

Some regulatory agencies have specific requirements regarding borehole abandonment and grout mixtures. Determine whether the oversight agency has any such requirements prior to finalizing the

drilling plan.

If DNAPL is known or expected to exist at the site, refer to the project specific documents (e.g., DNAPL Contingency Plan) for additional details regarding drilling to reduce the potential for inadvertent DNAPL remobilization.

Similarly, if light non-aqueous phase liquid (LNAPL) is known or expected to be present as “perched” layers above the water table, refer to the DNAPL Contingency Plan. Follow the general provisions and concepts in the DNAPL contingency plan during drilling above the water table at known or expected LNAPL sites.

Avoid using drilling fluids or materials that could impact groundwater or soil quality, or could be incompatible with the subsurface conditions. Water used for drilling, decontamination of drilling/sampling equipment, or grouting boreholes upon completion will be of a quality acceptable for project objectives. Testing of water supply will be considered.

Specifications of materials used for backfilling the borehole will be obtained, reviewed and approved to meet project quality objectives. Bentonite is not recommended where DNAPL is likely to be present or in groundwater with high salinity. In these situations, neat cement grout is preferred.

Store and/or stage empty and full sample containers and coolers out of direct sunlight. Sample container threads should be wiped down with a clean, nonabrasive material (e.g., paper towels) to better ensure the sample container is properly sealed. Be careful not to over-tighten lids with Teflon® liners or septa. Over-tightening can impair the integrity of the seal and can cause the glass to shatter and create a risk for hand injuries.

NOTE: Field logs and some forms are considered to be legal documents. All field logs and forms will therefore be filled out in indelible ink. Do not use permanent marker or felt-tipped pens for labels on sample container or sample coolers. Permanent markers could introduce volatile constituents into the samples.

NOTE: An Arcadis employee that is appropriately trained at the correct level of internal hazardous materials/DOT (Department of Transportation) shipping must complete an Arcadis shipping determination to address applicable DOT and IATA (International Air Transport Association) shipping requirements. Review the applicable Arcadis procedures and guidance instructions for sample packaging and labeling. Prior to using air transportation, confirm air shipment is acceptable under DOT and IATA regulations.

7 Health and Safety Considerations

The HASP will be followed, as appropriate, to ensure the safety of field personnel. Review all site-specific and procedural hazards as they are provided in the HASP, and review Job Safety Analysis (JSA) documents in the field each day prior to beginning work.

Prior to drilling, utility clearance must be performed (see Section 5). Appropriate personal protective equipment (PPE) will be worn at all times in line with the task and the site-specific HASP.

Working outside at sites with suspected contamination may expose field personnel to hazardous materials such as contaminated groundwater or NAPL (e.g., oil). Other potential hazards include biological hazards (e.g., stinging insects, ticks in long grass/weeds, etc.), and potentially the use of sharp cutting tools (scissors, knife). Only use non-toxic peppermint oil spray for stinging insect nests. Review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives

and use appropriate hand protection.

If thunder or lightning is present, discontinue drilling and sampling until 30 minutes have passed after the last occurrence of thunder or lightning.

8 Procedure

The procedures for drilling and the methods to be used to field screen and collect soil samples for laboratory analysis are presented below:

8.1 Drilling Procedures

8.1.1 Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods

1. Find/identify boring location, establish work zone, and set up sampling equipment decontamination area.
 - a. Verify utilities were cleared (see Section 5) and use soft dig technique to clear borehole, if applicable
 - b. Clean sampling equipment in accordance with the FIP/work plan prior to drilling
2. Advance boring to target depth:
 - a. Collect soil samples at appropriate interval as specified in the FIP/work plan (or equivalent) using the appropriate tooling (e.g., split-barrel sampler) and sample containers
 - i. Split-barrel or drive-ahead samples are obtained during drilling
 - ii. A common sampling method that produces high-quality soil samples with relatively little soil disturbance is described in *ASTM D1586 – Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils* (ASTM D1586).
 - b. Always change disposable gloves before handling the sampling equipment
 - c. Collect, document, and store samples for laboratory analysis as specified in the FIP/work plan (or equivalent; see below for additional details on sample collection procedures)
 - d. Field screen samples as specified in the FIP/work plan (or equivalent; see below for additional details on field screening procedures)
 - e. Rotasonic drilling produces soil cores that, for the most part, are relatively undisturbed, but note that when drilling in consolidated or finer-grained sediment the vibratory action during core barrel advancement may create secondary fractures or breaks. The core is retrieved by vibrating the soil/rock into a separate core bag, typically in 5-foot or 10-foot increments. The soil cores may consolidate or expand during retrieval, depending on soils, etc.
 - f. Dual-rotary removes cuttings by compressed air or water/mud and allow only a

- general assessment of geology unless separate coring tools and techniques are used
- g. Decontaminate equipment between samples in accordance with the FIP/work plan (or equivalent)
3. Describe each soil sample as outlined in the appropriate project records (refer to the description procedures outlined in the *TGI - Soil Description*)
- a. Record descriptions on the soil boring log (**Attachment 1**) and/or field notebook
 - b. When possible, photo document the samples (e.g., soil cores, split-barrels)
 - c. During soil boring advancement, document all drilling events in field notebook, including blow counts (i.e., the number of blows from a soil sampling drive weight [140 pounds] required to drive the split-barrel sampler in 6-inch increments) and work stoppages
 - d. Blow counts will not be available if rotasonic, dual-rotary, or direct-push methods are used; however, if standard penetration testing is required during rotasonic drilling, an automatic drop hammer may be used in conjunction with the method to switch from core barrel advancement to standard penetration testing
 - e. If soils are screened with a PID/FID or another instrument, document the measurement in accordance with the work plan
4. The drilling contractor will be responsible for obtaining accurate and representative samples, informing the supervising Arcadis geologist of changes in drilling pressure, drilling penetration rates, and keeping a separate general log of soils encountered, including blow counts
- a. The term “samples” means soil materials from particular depth intervals, whether or not portions of these materials are submitted for laboratory analyses
 - b. Records will also be kept of occurrences of premature refusal due to boulders, construction materials that may have been used as fill, etc.
 - c. Where a boring cannot be advanced to the desired depth, the boring will be abandoned, and an additional boring will be advanced at an adjacent location to obtain the required sample in accordance with the work plan
 - d. Where it is desirable to avoid leaving vertical connections between depth intervals (e.g., if DNAPL or perched LNAPL are known or expected to exist at the site), the borehole will be sealed using cement and/or bentonite (see **Section 5** above)
 - e. Multiple refusals may lead to a decision by the supervising geologist to abandon that sampling location

8.1.2 Direct-Push Method

The direct-push drilling method may also be used to complete soil borings. Examples of this technique include Geoprobe®, Diedrich Environmental Soil Probe (ESP) System, or AMS PowerProbe.

Environmental probe systems typically use a hydraulically operated percussion hammer.

Depending on the equipment used, the hammer delivers 140- to 350-foot pounds of energy with each blow. The hammer provides the force needed to penetrate very stiff to medium dense soil formations. The hammer simultaneously advances an outer steel casing that contains a dual tube liner for sampling soil

(dual tube sampling system).

The outside diameter (OD) of the outer casing ranges from 2.25 to 6 inches and the OD of the inner sampling tube diameter ranges from 1.4 to 4.5 inches. The outer casing isolates overlying soil and permits the unit to continue to probe at depth. The dual tube sampling system provides a borehole that may be tremie-grouted from the bottom up. Alternatively, a single rod system may be used that does not provide a cased boring and which limits tremie-grouting from the bottom up.

Direct-push drilling can generally achieve target depths 100 feet or less depending on the site geology. The known or expected site conditions (e.g., presence of NAPL) will be evaluated when selecting the type of direct-push sampling system to be employed.

1. Find/identify boring location, establish work zone, and set up sampling equipment decontamination area
 - a. Verify utilities were cleared (see Section 5) and use soft dig technique to clear borehole, if applicable
 - b. Clean sampling equipment in accordance with the FIP/work plan prior to drilling
2. Advance soil boring to target depth.
 - a. Collect soil samples at appropriate interval as specified in the FIP/work plan (or equivalent) using clean/disposable sampling equipment (plastic liners)
 - b. Always change disposable gloves before handling the sampling equipment
 - c. Collect, document, and store samples for laboratory analysis as specified in the FIP/work plan (or equivalent; see below for additional details on sample collection procedures)
 - d. Field screen samples as specified in the FIP/work plan (or equivalent; see below for additional details on field screening procedures)
3. Decontaminate equipment between samples in accordance with the FIP/work plan (or equivalent)
4. Describe samples in accordance with the procedures outlined in **Step 3** under **Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods** above (refer to the description procedures outlined in the *TGI - Soil Description*)

8.1.3 Manual Methods

Manual methods may also be used to complete shallow soil borings. Examples of this technique include using a spade, spoon, scoop, hand auger, or slide hammer. Manual methods are typically used to collect surface soil samples (0 to 6 inches) or to complete soil borings/collect soil samples from a depth of 5 feet or less.

1. Find/identify boring location, establish work zone, and set up sampling equipment decontamination area
2. Clear the ground surface of brush, root mat, grass, leaves, or other debris
3. Use a spade, spoon, scoop, hand auger, or slide hammer to collect a sample of the required depth interval
4. Use an engineer's ruler or survey rod to verify that the sample is collected to the correct depth and

record the top and bottom depths from the ground surface

5. To collect samples below the surface interval, remove the surface interval first; then collect the deeper interval
 - a. To prevent the hole from collapsing, it may be necessary to remove a wider section from the surface or use cut polyvinyl chloride (PVC) pipe to maintain the opening
 - b. Collect soil samples at appropriate interval as specified in the FIP/work plan (or equivalent) and transfer to the appropriate, laboratory-supplied container
 - c. Collect, document, and store samples for laboratory analysis as specified in the FIP/work plan (or equivalent; see below for additional details on sample collection procedures)
 - d. Field screen samples as specified in the FIP/work plan (or equivalent; see below for additional details on field screening procedures)
6. Decontaminate equipment between samples in accordance with the FIP/work plan (or equivalent)
7. Describe samples in accordance with the procedures outlined in **Step 3** under ***Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods*** above (refer to the description procedures outlined in the *TGI - Soil Description*)

8.2 Field Screening Procedures

8.2.1 PID and FID Screening

Soils are typically field screened with a PID or FID for a relative measure of the total VOCs at sites where VOCs are known or suspected to exist. PIDs and FIDs require calibration in accordance with the work plan(s) and manufacturer's specifications and PIDs should be calibrated based on the target chemicals. The PID employs an ultraviolet lamp to measure VOCs and the ionization energy (IE) of the site constituents need to be considered when selecting the type of lamp (e.g., 10.6 eV, 11.7 eV) that will be used. In general, any compound with an IE lower than that of the lamp photons can be measured. The FID has a wide linear range and responds to almost all VOCs.

Field screening is performed using one (or both) of the following two methods:

1. Upon opening the sampler, the soil is split open and the PID or FID probe is placed in the opening and covered with a clean, gloved hand. Such readings will be obtained at several locations along the length of the sample.
2. A portion of the collected soil is placed in a jar, which is covered with aluminum foil, sealed, and allowed to warm to room temperature (see below). After warming, the cover is removed, the foil is pierced with the PID or FID probe, and a reading is obtained.

Prior to usage, the PID or FID must be calibrated according to the manufacturer's specifications at a minimum frequency of once per day prior to collecting PID or FID readings. The PID will be calibrated to a benzene-related compound (isobutylene) or other appropriate gas, while the FID will be calibrated to methane. The time, date, and calibration procedure must be clearly documented in the field notebook and/or the calibration form.

If at any time the PID or FID results appear erratic or inconsistent with field observations, then the instrument will be recalibrated.

If calibration is difficult to achieve, then the PID's lamp will be checked for dirt or moisture and cleaned, or technical assistance will be required. Maintenance and calibration records will be kept as part of the field quality assurance program.

Initial PID readings will be recorded on the soil boring log (**Attachment 1**) and/or in the field notes. The soil sample will be separated from the slough material (if any) by using disposable gloves and a pre-cleaned stainless-steel spoon or tool.

For the second method, a representative portion of the sample will be placed in a pre-cleaned air-tight container (as quickly as possible to avoid loss of VOCs), filling the container half full to allow for the accumulation of vapors above the soil. An aluminum foil seal will be placed between the glass and cap and the cap will be screwed on tightly. Unless the screening will be performed immediately after the sample is placed in the container, the sample containers will be stored in a cooler chilled to approximately 4°C until screening can be performed.

The headspace of the container will be measured using a PID or FID as follows:

1. Samples will be taken to a warm workspace and allowed to equilibrate to room temperature for at least one hour.
2. Prior to measuring the soil vapor headspace concentration, the container will be shaken.
3. The headspace of the sample will then be measured directly from the container by piercing the aluminum foil seal with the probe of the PID or FID and measuring the relative concentration of VOCs in the headspace of the soil sample. The initial (peak) reading must be recorded.

8.2.2 NAPL Screening

To screen for the potential presence of non-aqueous phase liquid (NAPL) in soil, drilling procedures must allow for high-quality porous media samples to be taken. Split-barrel samplers or direct-push samplers will be collected continuously ahead of the auger, drill casing/rods, or probe rods. Upon opening each split-barrel sampler or direct-push plastic liner sleeve, the soil will immediately be evaluated for the presence of visible NAPL and odors. If suspected NAPL is immediately visible in the sample, its depth will be noted.

Additionally, the soil will be screened for the presence of organic vapors using a PID or FID, in accordance with the work plan, if applicable. During screening, the soil will be split open using a clean spatula or knife and the PID or FID probe will be placed in the opening and covered with a clean, gloved hand (**Method 1** above). Such readings will be obtained along the entire length of the sample. Alternatively, **Method 2** for PID/FID screening (outlined above) may also be performed. If the PID or FID examination reveals the presence of organic vapors above 100 parts per million (ppm), the sample will undergo further detailed evaluation for visible NAPL.

The assessment for NAPL will include the following tests/observations:

- Evaluation for Visible NAPL Sheen or Free-Phase NAPL in Soil Sampler
 - NAPL sheen will be a colorful iridescent appearance on the soil sample
 - NAPL may also appear as droplets or continuous accumulations of liquid with a color typically ranging from yellow to brown to black, depending on the type of NAPL
 - Creosote DNAPL (associated with wood-treating sites) and coal tar DNAPL (associated with manufactured gas plant [MGP] sites) are typically black and have a characteristic, pungent odor
 - Pure chlorinated solvents may be colorless in the absence of hydrophobic dye. Solvents mixed

with oils may appear brown

- Particular care will be taken to fully describe any sheens observed, staining, discoloration, droplets (blebs), or NAPL saturation
- Soil-Water Pan Test
 - A portion of the selected soil interval with the highest PID or FID reading above 100 ppm will be placed in a disposable polyethylene dish along with a small volume of potable or distilled water
 - The dish will be gently tilted back and forth to mix the soil and water, and the surface of the water will be viewed in natural light to observe the development of a sheen, if any
 - A small quantity of Oil Red O or Sudan IV hydrophobic dye powder should be added in accordance with the work plan, and the soil and dye will be manually mixed for approximately 30 to 60 seconds and smeared in the dish to create a paste-like consistency
 - A positive test result will be indicated by a sheen on the surface of the water and/or a bright red color imparted to the soil following mixing with dye
- Soil-Water Shake Test
 - A small quantity of soil (up to 15 cc) will be placed in a clear, colorless, jar containing an equal volume of potable or distilled water (40-mL vials are well suited to this purpose, but not required)
 - After the soil settles into the water, the surface of the water will be evaluated for a visible sheen under natural light
 - The jar will be closed and gently shaken for approximately 10 to 20 seconds
 - Again, the surface of the water will be evaluated for a visible sheen or a temporary layer of foam
 - A small quantity (approximately 0.5 to 1 cc) of Oil Red O or Sudan IV powder will be placed in the jar in accordance with the work plan
 - The sheen layer, if present, will be evaluated for a reaction to the dye (change to bright red color)
 - The jar will be closed and gently shaken for approximately 10 to 20 seconds
 - The contents in the closed jar will be examined under natural light for visible bright red dyed liquid inside the jar

- A positive test result will be indicated by the presence of a visible sheen or foam on the surface of water, a reaction between the dye and the sheen layer upon first addition of the dye powder, a bright red coating on the inside of the vial (particularly above the water line), or red-dyed droplets within the soil

NOTE: *If NAPL is obviously present upon opening the soil sampler or evaluating the soil sample within the split-spoon sampler or direct-push liner sleeve, it is not necessary to perform a soil-water pan test or soil-water shake test. In addition, it is not necessary to perform both a soil-water pan test and a soil-water shake test; either test method is acceptable. The pan test may be preferred in some circumstances because the presence of a sheen may be easier to see on a wider surface. Further, these tests will only be performed if specified in the work plan(s).*

NOTE: *When using hydrophobic dye in the tests above, color will be assessed outdoors under natural light during the period between sunrise and sunset, regardless of the degree of cloud cover. The hydrophobic dye Safety Data Sheets (SDS) will be incorporated into the HASP and reviewed prior to use and the dyes will be carefully handled and disposed in accordance with regulations, if applicable.*

8.3 Soil Sample Collection for Laboratory Procedures

If not specifically identified in the FIP/work plan, soil samples will be selected for laboratory analysis based on:

1. Their position in relation to identified source areas
2. The visual presence of source residues (e.g., NAPL or staining)
3. The relative levels of total VOCs based on field screening measurements
4. The judgment of the field coordinator
5. Moisture content or relative position with regard to apparent groundwater table/saturation

Samples designated for laboratory analysis will be placed in the appropriate containers.

Sample containers for VOC analysis will be filled first immediately following soil core retrieval to reduce loss of VOCs.

If samples will be collected for other analyses, a sufficient amount of the remaining soil will then be homogenized as described below and sample containers will be filled for other parameters.

VOC samples will be collected as discrete samples using a small diameter core sampler (e.g., En Core® Sampler, Terra Core™ Sampler).

The En Core® Sampler is a disposable volumetric sampling device that collects, stores and delivers soil samples without in-field chemical preservation. The En Core® Sampler requires the use of a reusable T-handle.

The Terra Core™ Sampler is a one-time use transfer tool, designed to collect soil samples and transfer them to the appropriate containers for in-field chemical preservation (e.g., methanol).

The small diameter core samplers will be used according to the manufacturer's instructions (e.g., En Novative Technologies). Some regulatory agencies have specific requirements regarding VOC sample

collection. Determine whether the oversight agency has specific requirements prior to commencing sampling and collect samples at appropriate interval as specified in the FIP/work plan (or equivalent). Samples may require homogenization across a given depth interval, or several discrete grabs (usually five) may be combined into a composite sample.

NOTE: Samples for VOC and PFAS analysis will NOT be homogenized or composited and will be collected as discrete samples as described above.

The procedure for mixing samples is provided below.

1. Mix the materials in a stainless steel (or appropriate non-reactive material) bowl using a stainless-steel spoon (or disposable equivalents)
 - a. When dealing with large sample quantities, use disposable plastic sheeting and a shovel or trowel
 - b. *NOTE: When preparing samples for metals analyses, do not use disposable aluminum (or metal tools or trays other than stainless steel), as it may influence the analytical results*
2. Flatten the pile by pressing the top without further mixing
3. Divide the circular pile by into four equal quarters by dividing out two diameters at right angles
4. Mix each quarter individually using appropriate non-reactive bowls, spoons and/or sheeting
5. Mix two quarters (as described above) to form halves, then mix the two halves to form a composite or homogenized sample
6. Place composite or homogenized sample into specified containers
7. Remaining material will be disposed of in accordance with project requirements and applicable regulations
8. Sample containers will be labeled with sample identification number, date, and time of collection and placed on ice in a cooler (target 4° Celsius)
9. Samples selected for laboratory analysis will be documented (chain-of-custody forms), handled, packed, and shipped in accordance with the procedures outlined in the FIP/work plan (or equivalent).

8.4 Soil Boring Abandonment

All soil borings need to be abandoned in accordance with ***TGI for Monitoring Well and Soil Boring Decommissioning***. See Attachment E of the TGI for specifics.

9 Waste Management

Investigative-Derived Waste (IDW) generated during drilling activities, including soil and excess drilling fluids (if used), and decontamination liquids, will be stored on site in appropriately labeled containers and disposed of properly. Disposable materials will be stored and disposed of separately. Containers must be labeled at the time of collection and will include date, location(s), site name, city, state, and description of matrix contained (e.g., soil, PPE). Waste will be managed in accordance with the ***TGI – Investigation-Derived Waste Handling and Storage***, the procedures identified in the FIP/work plan or QAPP as well as

state-, federal- or client-specific requirements. Be certain that waste containers are properly labeled and documented in the field log.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Management of the original documents from the field will be completed in accordance with the site- specific QAPP.

In general, drilling activities will be documented on appropriate field/log forms as well as in a proper field notebook. All field data will be recorded digitally or with indelible ink. Field forms, logs/notes (including daily field and calibration logs), digital records, and chain-of-custody records will be maintained by the field team lead. Any deviations or omissions from this TGI should be documented.

Initial field logs and chain-of-custody records will be transmitted to the Arcadis CPM and Technical Lead at the end of each day unless otherwise directed by the CPM. The field teamleader retains copies of the field documentation.

Additionally, all documents (and photographs) will be scanned and electronically filed in the appropriate project directory for easy access. Pertinent information will include personnel present on site, times of arrival and departure, significant weather conditions, timing of drilling activities, soil descriptions, soil boring information, and quantities of materials used.

In addition, the locations of soil borings will be documented photographically and in a site sketch. If appropriate, a measuring wheel or engineer's tape will be used to determine approximate distances between important site features.

Records generated as a result of this TGI will be controlled and maintained in the project record files in accordance with project requirements.

11 Quality Assurance

Quality assurance procedures shall be conducted in accordance with the Arcadis Quality Management System or the site-specific QAPP.

All drilling equipment and associated tools (including augers, drill rods, sampling equipment, wrenches, and any other equipment or tools) that may have come in contact with soil will be cleaned in accordance with the procedures outlined in the appropriate TGI.

Field-derived quality assurance blanks will be collected as specified in the FIP/work plan and/or site- specific QAPP, depending on the project quality objectives. Typically, field rinse blanks (equipment blanks) will be collected when non-dedicated equipment (e.g., split-spoon sampler, stainless steel spoon) is used during soil sampling. Field rinse blanks will be used to confirm that decontamination procedures are sufficient and samples are representative of site conditions. Trip blanks for VOCs, which aid in the detection of contaminants from other media, sources, or the container itself, will be kept with the coolers and the sample containers throughout the sampling activities and during transport to the laboratory.

Operate all monitoring instrumentation in accordance with manufacturer's instructions and calibration procedures. Calibrate instruments at the beginning of each day and verify the calibration at the end of each day. Record all calibration activities in the field notebook.

12 References

ASTM D1586 - *Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils*. ASTM International. West Conshohocken, Pennsylvania.

13 Attachments

Attachment 1. Soil Boring Log Form

Attachment 1

Soil Boring Log Form

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TGI – Residential Drinking Water Well Sample Collection

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Date

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Marc Killingstad (Subject Matter Expert)

Date

1 Introduction

This Technical Guidance Instruction is intended to provide general guidance for collecting water samples from residential drinking water wells at various sampling points. The TGI references guidance for the collection of Residential and Commercial Water Supply Wells in Areas of Exploration and Production (E&P) Operations.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

This Technical Guidance Instruction (TGI) provides general guidance, material needs, and methodology for collecting residential drinking water samples from private drinking water systems for biological and chemical analysis.

For specific information regarding sampling of residential water supply systems in areas of exploration and production (E&P) refer to ASTM D8006-16.

This document does not address water quality parameter measurements (e.g., specific conductivity, temperature, pH, ORP, etc.), sample preservation/packaging, chain-of-custody forms, or laboratory analysis. Refer to the [Resilience Environment Quality Management System Library](#) for additional guidance documents that cover those

specific areas as well as the project field implementation plan (FIP)/sampling plan, Quality Assurance Project Plan (QAPP), and the site-specific Health and Safety Plan (HASP), as appropriate.

4 Personnel Qualifications

Arcadis field sampling personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as identified in the site-specific Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. The HASP will also identify any access control requirements.

Prior to mobilizing to the field, the sampling team will review and be thoroughly familiar with relevant site-specific documents including but not limited to the task-specific work plan or field implementation plan (FIP)/sampling plan, QAPP, HASP, historical information, and other relevant site documents.

Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. Additionally, the groundwater sampling team will review and be thoroughly familiar with documentation provided by equipment manufacturers and become familiar with the operation of (i.e., hands-on experience) all equipment that will be used in the field prior to mobilization.

Ideally, Arcadis personnel directing, supervising, or leading drinking water sample collection activities will have a minimum of one year of previous groundwater sampling experience. Field employees with less than six months of experience will be accompanied by a supervisor (as described above) to ensure that proper sample collection techniques are employed.

Arcadis personnel shipping samples need to be HazMat #1 trained.

5 Equipment List

The following materials and supplies will be available, as required, during drinking water sample collection:

- Completed Arcadis Access Agreement (see **Attachment A** for example agreement) and/or Consent to Sample Form (see **Attachment B** for example form).
- Site-specific HASP and health and safety documents identified in the HASP (e.g., Job Safety Analysis [JSA] for residential water sampling).
- Field Implementation Plan (FIP) that includes Site map, well construction records (table or logs), sampling plan (sample analyses, sample volume required, and sample holding time), and prior sampling records (if available).
- Field notebook and/or smart device (smart phone or tablet).
- Indelible ink pen.

- Residential Drinking Water Well Sampling Log (**Attachment C**).
- Well Survey Questionnaire (see **Attachment D** for example questionnaires).
- Arcadis sampling team business cards.
- Chain-of-Custody (COC) and security seals.
- Appropriate personal protective equipment (PPE) (e.g., latex or nitrile gloves, safety glasses, etc.) as specified in the HASP.
- Multiparameter water-quality meter (temperature/pH/specific conductivity/oxidation reduction [ORP]/turbidity/dissolved oxygen) and flow-through measurement cell (as required).
- Gas or multiple parameter meter for atmospheric screening to provide information on lower explosive limits for combustible gases and oxygen levels at a minimum (as required per HASP and Work Plan).
- Two (2) clean graduated buckets with lids.
- Graduated beaker for flow estimation.
- Groundwater sample containers and labels (supplied by the laboratory—include an extra set) appropriate for the analytical method(s) with preservative, as needed (parameter-specific).
- Appropriate blanks (trip blank supplied by the laboratory), as specified in the FIP/sampling plan.
- Ziploc-type freezer bags for use as ice containers.
- Appropriate transport containers (coolers) with ice and appropriate labeling, packing, and shipping materials.
- Chain-of-custody forms.
- Filter, as needed, in accordance with the analytical method and parameter, and as specified in the FIP/sampling plan.
- Decontamination equipment.
 - Buckets.
 - Distilled or deionized water.
 - Cleansers appropriate for removing expected chemicals of concern.
 - Isopropyl alcohol or 5% chlorine bleach solution and swabs.
 - Soap (i.e., Micro 90 or Alconox) and swabs with a small brush for cleaning exterior tap or kitchen faucet.
 - Do not use chlorine bleach if perchlorate is included in laboratory analysis constituent list.
 - Paper towels.
- Plastic sheeting.
- New garden hose (50 feet).

- Flashlight/headlamp if the sampling must be conducted from a tap in a basement or crawl space.

6 Cautions

Prior to mobilizing to the field, the sampling team will verify that:

- The laboratory has the proper certification to conduct drinking water analysis. Also verify that the correct laboratory method detection limits and reporting limits are in place prior to conducting the sampling event.
- The laboratory analytical methods referenced on the chain of custody are in accordance with applicable state and federal drinking water analytical methods.
- The sample collection locations and procedures are conducted in accordance with local and state health department and regulatory organization regulations and guidelines.
- Arcadis legal department has reviewed and approved all access agreement and/or consent forms.
- Arcadis has appropriate access permission to sample the resident's well and that an appointment has been made prior to arriving at the residence.
- The resident has not conducted a chlorine disinfection procedure in the well in the past 48 to 72 hours.

If applicable, be careful not to over-tighten lids with Teflon® liners or septa (e.g., 40-mL vials). Over-tightening can cause the glass to shatter and/or impair the integrity of the seal.

NOTE: Do not enter the residence unless the resident or authorized party is present.

NOTE: If the residential sampling is being conducted as part of an emergency response event, make sure that a client public relations release or briefing has been reviewed prior to discussing details of the event with the resident.

NOTE: Make sure that clean shoes or disposable boot covers are worn into the residence to maintain good community relations and prevent tracking site constituents inside the residence.

NOTE: Field logs and some forms are legal documents. All field logs and forms will be filled out in indelible ink. Do not use permanent marker or felt-tipped pens for labels on sample container or sample coolers. Permanent markers could introduce volatile constituents into the samples.

NOTE: An Arcadis employee that is appropriately trained at the correct level of internal hazardous materials/DOT (Department of Transportation) shipping must complete an Arcadis shipping determination to address applicable DOT and IATA (International Air Transport Association) shipping requirements.

Review the applicable Arcadis procedures and guidance instructions for sample packaging and labeling. Prior to using air transportation, confirm air shipment is acceptable under DOT and IATA regulations.

7 Health and Safety Considerations

The HASP will be followed, as appropriate, to ensure the safety of field personnel.

Appropriate personal protective equipment (PPE) will be always worn in line with the task and the site-specific HASP.

Review all site-specific and procedural hazards as they are provided in the HASP, and review Job Safety Analysis (JSA) documents in the field each day prior to beginning work.

Arcadis advocates the 'buddy system' when conducting door-to-door visits with the public. Stick together while inside the house, stay focused on the sampling task and do not enter other parts of the house unless necessary to complete the task or evaluate the drinking water system.

If the sample point is in a crawl space (i.e., the spigot off the water tank) or behind a treatment system, visually survey the area for safe ingress and egress as well as hazards such as spiders, rodents, or damaged electrical wiring prior to entering the crawl space.

If conditions are observed that may present a hazard to sampling personnel such as illegal activity, aggressive or agitated behavior by the resident, or poor housekeeping, use Stop-Work authority, leave the area, and contact the field sampling coordinator, or project team leader. Do not communicate or discuss your concerns with the resident.

8 Procedure

The general steps to sample residential drinking water wells are outlined below:

1. Verify that Arcadis has appropriate access to sample the well and has scheduled a day and time to meet the resident for collecting the sample. Make sure to have copies of the following (as applicable)
 - a. Signed Access Agreement
 - b. Signed Consent Form
 - c. Completed Questionnaire
2. Review equipment list (**Section 4** above) to confirm that the appropriate equipment has been acquired.
3. Calibrate field instruments according to manufacturer procedures for calibration and document accordingly on the calibration logs, field form, and/or field logbook.
4. All equipment will either be new or decontaminated in accordance with appropriate guidance document (*TGI – Groundwater and Soil Sampling Equipment Decontamination*) prior to use.
5. Examine the Well Survey Questionnaire completed by the owner of the well (as applicable).
6. Go to the residence, and politely knock or ring the bell. When the resident answers the door, introduce yourselves and provide an Arcadis business card and identification, if requested.
7. Conduct a brief interview with the resident, verifying the information that was provided in the questionnaire returned by the resident (if part of a sampling program), or complete the questionnaire with the resident if the resident did not return a completed copy of the questionnaire (see **Attachment D** for example).
 - a. Note the location of the pit-less adapter or other well head equipment.
 - b. Note the presence of a septic tank, a separate well for landscaping or agricultural purposes, a basement, workshop, or signs of a spill or disposal.
 - c. Complete all entries on project sampling forms or record notes in field logbook.

8. Based on the findings of the interview and drinking water system evaluation, determine a suitable sampling location with the assistance of the resident and prepare sampling area as necessary (i.e., place plastic sheeting adjacent to the well for use as a clean work area, if conditions allow).
 - a. The sampling location must be located upstream of any kind of water treatment system, including water softeners, carbon filters, ion-exchange systems, or chlorination systems.
 - b. First look for a spigot installed in the water line where the water line enters the interior of the house. If a spigot cannot be identified or safely accessed, then use a leak-free, cold-water faucet with sufficient space beneath it for the sample containers.
 - c. Do not collect the sample from a “shifter-arm” style faucet or a faucet without separate controls for hot and cold water.
 - d. Do not collect the sample if the well has been treated with chlorine for disinfection within the last 48 hours.

NOTE: If sampling is conducted in the state of Ohio, OAC Rule 3701-28-04 requires that all water samples collected to determine compliance will be collected at the point of discharge at the pressure tank unless the pressure tank is inaccessible. Only when no other sample taps are available the sample may be collected from the closest spigot to the pressure tank.

9. If sampling from a faucet, remove/unscrew the aerator screen, if present, prior to purging the faucet.
10. Sterilization: Don PPE as required in the HASP and using the alcohol/chlorine bleach solution/soap swab the inside and the outside of the spigot or faucet prior to purging
11. If sampling from a crawl space or an outdoor spigot, connect a hose to convey the purge water to an interior drain or discharge to the outdoors.
 - a. If discharging to the outdoors, direct the water towards an open, grassy area away from the wellhead to mitigate the potential for ponding or recharge to the well.
 - b. Remember to sanitize the tap after removing the hose and before collecting the sample.
 - c. If sampling from a faucet, inspect the sink drain and make sure the drain is clear of any obstructions to prevent overflowing the sink or basin.
12. Turn on the sampling location tap or spigot and run the water for *15 minutes if possible*.
 - a. If there is a water tank or pressure tank, and the volume is known, purge at least one tank volume prior to sampling.
 - b. Note if there is a temperature change in the water during purging.
 - c. If a water quality instrument is being used to evaluate specific parameters such as temperature, dissolved oxygen, etc., parameter stabilization will be noted.
 - d. The objective of the 15-minute purge is to clear the static water from the well network and pressure tank; and have the pump turn on to draw fresh water directly from the surrounding aquifer.
13. While purging, record site and residential well identification on the residential water sampling log (**Attachment A**), along with date, arrival time, weather conditions, personnel present, equipment utilized, and other relevant data requested on the log. Prepare sample containers and fill out sample labels with indelible ink. An example for sample nomenclature is as follows:
RW-1234CR1 (032218) is a sample collected from a Residential Well at 1234 County Road 1, collected on March 22, 2018. If more than one sample location is identified at the same residence such as an agricultural well in a barn, then the sample name will be modified to represent this **RW-1234CR1AgWell (032218)**.

14. After 15 minutes or one water tank volume, reduce the flow (to approximately 200 ml per minute) and prepare for sampling.
15. When collecting multiple parameter samples fill the containers in the following order:
 - a. Volatile organic compounds (VOCs)
 - b. Semi volatile organic compounds (SVOCs)
 - c. Coliforms
 - d. Pesticides/PCBs
 - e. Total petroleum hydrocarbons (TPH)
 - f. Total metals
 - g. Alkalinity, BOD, total suspended solids, total dissolved solids, sulfate, and chloride.
 - h. Sulfide
 - i. Cyanide
 - j. Phenols
 - k. Nitrate + nitrite, ammonia, TOC, COD, phosphorous
16. For samples that require acid or base preservation:
 - a. Verify that the required pH is achieved by placing a small amount of sample from the sample container in a clean dish (or equivalent) and test the sample with pH paper.
 - b. Do not place the pH paper in the sample container.
 - c. For VOC vials, it may be necessary to modify the procedure by pouring a few drops onto the pH paper to determine the pH.
 - d. Conduct the pH measurement on the VOC vials on approximately 10% of the samples.
 - e. The VOC vial must be refilled to no headspace.
 - f. Alternatively, fill once extra VOC vial for pH testing, test the contents of the VOC vial, then discard the VOC vial appropriately.
17. Fill the VOC vials completely (i.e., no headspace) directly from the tap until a meniscus form at the bottle opening. Place the cap on the vial and screw it on. Invert the bottle and check for air bubbles. If bubbles are present remove the cap and add more sample. Replace the cap and re-inspect the vial for bubbles.
18. Fill the remaining sample containers to the shoulder. Close and label each container with the appropriate information.
19. Immediately after collection, secure sample containers with packing material and maintain at approximately 4°C on wet ice contained in double Ziploc-type freezer bags in an insulated, durable transport cooler.
20. Complete the procedures for chain-of-custody, handling, packing, and shipping (refer to *Sample Chain-of-Custody SOP*).
 - a. Chain-of-custody forms will be filled out and checked against the labels on the sample containers progressively after each sample is collected
 - b. Ship the samples to the laboratory per project requirements.

21. Record the time sampling procedures were completed on the sampling field forms/field logbook using indelible ink.
22. Properly dispose of personal protective equipment (PPE) and disposable equipment and practice good 'housekeeping'—place all disposable sampling materials (e.g., plastic sheeting, disposable tubing or bailers, and PPE) in appropriate containers and diligently perform housekeeping tasks to maintain good community relations by leaving the residence in the same condition found upon arrival.
23. As necessary, complete decontamination of sampling equipment (e.g., submersible or bladder pump) as appropriate (*TGI – Groundwater and Soil Sampling Equipment Decontamination*).
24. As necessary, at the end of each day of the sampling event, perform calibration check of field instruments and record procedure and results in field log.

Note: When sampling for PFAS as part of the drinking water parameter sampling list, collect the PFAS sample first. The requirements of TGI – Per- and Polyfluoroalkyl Substances (PFAS) Field Sampling Guide apply.

Note: When collecting Lead and Copper Rule Compliance Samples, follow the following guidelines:

- *Select cold-water facet not connected to water softeners, point-of-use filters or other devices designed to change composition of the water.*
- *Do not remove screens or aeration devices.*
- *For first-flush samples, allow water to sit undisturbed for at least six hours prior to sampling and do not intentionally flush the line before the start of the six-hour period.*
- *Collect the first water out of the tap in a 1 Liter container placed under the faucet.*

9 Waste Management

Waste will be managed in accordance with the TGI – Investigation-Derived Waste Handling and Storage, the procedures identified in the FIP or QAPP as well as state-, federal- or client-specific requirements. Investigation-Derived Waste (IDW), including purge water, decontamination liquids, and disposable materials (plastic sheeting, PPE, etc.) will be disposed of properly. As appropriate, disposable materials will be placed in appropriate containers and labeled at the time of collection (including date, location, site name, city, state, and description of matrix contained [e.g., PPE]). Purged tap water resulting from running the tap prior to collecting the sample will be managed as outlined in Step 11 of Section 7 above. Be certain that waste containers are properly labeled and documented in the field notes.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff

are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

[Click to enter text]

11 Quality Assurance

Quality assurance procedures will be conducted in accordance with the Arcadis Quality Management System or the site-specific QAPP.

Field-derived quality assurance blanks will be collected as specified in the FIP/sampling plan, depending on the project quality objectives.

Quality assurance and quality control (QA/QC) samples such as Duplicates, Blind Duplicates, Matrix Spike/Matrix Spike Duplicates, Field Blanks, or Equipment Blanks may be included as part of this sampling process. QA/QC samples may be required by local, state, or federal regulatory agencies depending on the scope of the project. Specific details related to the QA/QC sample collection procedures will be described in detail in the project FIP, QAPP, or project kickoff meeting notes. A brief overview of the collection of QA/QC samples is included here for reference.

Equipment Blanks

Equipment blanks are prepared by filling the sample bottles with distilled or de-ionized water. To avoid confusion, do not use the term “Field Blank” when describing these samples. Equipment blanks needed to be included on the chain-of-custody form. Check the work plan or ask the project manager what the equipment blank sample requirements are for each project.

Duplicate Samples

Duplicate samples are prepared by alternately filling the container for the "investigative sample" for a particular parameter and then filling the container for the "duplicate sample" for that same parameter. Duplicate samples need to be included on the chain of custody. Check the work plan or ask the project manager what the duplicate sample requirements are for each project.

Trip Blanks

Trip blanks (VOCs only) are prepared in the laboratory and are shipped from the laboratory with the VOC sample collection vials for each project. The trip blank vials will be inspected for air bubbles upon receipt from the laboratory. The trip blanks are not opened in the field. A trip blank will be present in each shipping cooler containing VOC samples going to the lab. Trip blanks need to be included on the chain- of-custody form.

Matrix Spike/Matrix Spike Duplicates (MS/MSDs)

Matrix spike/matrix spike duplicates (MS/MSDs), when required for the project, are collected in the same manner as a duplicate sample. Check with the laboratory for the volume requirements for each parameter. MS/MSDs need to be included on the chain-of-custody form. Check the work plan or ask the project manager what the duplicate sample requirements are for each project. Typically, field rinse blanks (equipment blanks) will be collected when non-dedicated equipment (e.g., submersible pump) is used during groundwater sampling. Field rinse blanks will be used to confirm that decontamination procedures are sufficient, and samples are representative of site conditions. Trip blanks for VOCs, which aid in the detection of contaminants from other

media, sources, or the container itself, will be kept with the coolers and the sample containers throughout the sampling activities and during transport to the laboratory.

12 References

ASTM Standard D8006-16. 2016. Sampling and Analysis of Residential and Commercial Water Supply Wells in Areas of Exploration and Production (E&P) Operations.

United States Environmental Protection Agency (USEPA). 2019. Operating Procedure: Potable Water Supply Sampling. USEPA Region 4 Science and Ecosystem Support Division, Athens, Georgia (June 11, 2019) SESDPROC-3050R4.

13 Attachments

Attachment A – Example of Access Agreement

Attachment B – Example Consent to Sample Form

Attachment C – Residential Drinking Water Well Sampling Log

Attachment D – Example Water Well Questionnaires and Well Survey Questionnaire and Sampling Permission Agreement

ATTACHMENT A. Example Site Access Agreement



This SITE ACCESS AGREEMENT ("Agreement") made and entered into on this ____ day of _____, 20__, by and among Arcadis U.S., Inc., ("Consultant"), and _____ ("Client"), and together with Consultant, and _____, ("Owner").

I Recitals

The Client and Consultant desire access to the site described in the attached **Exhibit B** ("Site") to engage in the activities specified in the attached **Exhibit A**.

In consideration of the mutual promises and for any other valuable consideration, the receipt and adequacy of which are hereby acknowledged, Client and Consultant (the "Undersigned") agree as follows:

II Terms and Conditions

A Site Access. Owner hereby grants permission to Undersigned to enter the Site and engage in the activities specified in **Exhibit A**. Upon completion of the activities, the Undersigned will restore the Site to a condition substantially similar to its condition and repair at the time of the activities.

B Release. As consideration for being afforded access to the Site, and unless specifically excluded herein, the Undersigned hereby waives, releases and discharges Owner, its parent and subsidiaries, affiliates and their respective stakeholders, directors, officers and agents from all present or future claims, causes of action, or demands that Undersigned now has or may hereafter accrue on account of any and all known and unknown, or seen and unforeseen bodily and personal injuries or property damage and the consequences thereof resulting, or which may result, from Undersigned's negligent activities upon the Site or the use of any equipment or procedures while on, entering or leaving the Site.

Claims arising out of existing site conditions, the negligence, acts, omissions or willful misconduct of Owner, its parent and subsidiaries, affiliates, and their respective shareholders, directors, officers, and agents are excluded from this Release. Any damages directly caused by Owner related to the activities Consultant performs on the Site, will be the Owner's responsibility and the Owner shall be liable for such damages and the direct and actual costs to repair such damages.

C Data and Reports. Owner understands and agrees that Consultant does not have any obligation or duty to disclose or report to Owner any information, data, reports, or findings resulting from any activities or investigations on the Site.

D Insurance. The Consultant and its Client shall provide and maintain commercial general liability insurance against all claims for damages to person or property or loss of life or of property occurring upon the Site.

E Successors. This Site Access Agreement shall be binding on the successors and assigns of the Owner and the Undersigned. This agreement may not be assigned in whole or in part without the written consent of the Owner and the Undersigned.

Owner

Client

By: _____

By: _____

Date: _____

Date _____

Consultant

By: _____

Date _____

Exhibit A
Description of Work

Activities include:

The depth to water in the existing well(s) will be measured using an electronic measuring device. Water samples will be collected from the existing monitoring wells and water wells. All equipment ARCADIS utilizes for sampling activities will be decontaminated using a laboratory-grade detergent solution and potable water rinse prior to use at each location.

Samples will be collected into laboratory containers and submitted to **LAB** in **CITY, STATE** under appropriate chain-of-custody procedures. The samples will be analyzed for a short list of **CONSTITUENTS OF CONCERN**. Analytical data will be provided upon request.

The Property will be restored to its original condition after the sampling is completed. All water purged from the wells will be disposed of properly and/or placed into containers and transported to the **CLIENT** property for treatment and disposal.

Exhibit B
Site Map

ATTACHMENT B. Example Consent to Sample Form





Consent to Sample Form

Are you willing to allow Arcadis to collect samples from your well? Yes No

Can we re-sample your well on a periodic basis, if needed? Yes No

Can we collect samples outside your home when you are not present? Yes No
(We will leave a note if we sample your well when you are not home)

If you authorize Arcadis to collect samples from your well as described above, please sign here:

Signature

Date

FOR OFFICE USE:

Access Authorization Communicated: Phone Call Date & Time _____

In Person Date & Time _____ Written Date & Time _____

Project No. _____ Project Mgr. _____

ATTACHMENT C. Residential Well Sampling Log



Residential Well Water Sampling Log

Sampling Personnel: _____ Date: _____

Purge Time - Begin: _____ End: _____ Weather: _____

Sampling Location and/or Residential Well(s): _____

Layout of Sampling Location(s) and Specific Site Features:

Is there a preferred sampling location? _____

Has this location previously been sampled? _____

Is there a water softener upstream of the sampling location? _____

How many wells on property? _____

Approximate Flow Rate: _____ How Measured: _____

Gallons Purged (Estimate): _____

Purge Water Observations (Color, Odor, etc.): _____

| Constituents Sampled | Container Description | No. of Containers | Preservative |
|----------------------|-----------------------|-------------------|--------------|
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |

Sample ID: _____

QA/QC or Duplicate Sample Collected at this Location? _____

Sampling Team Leader (signature): _____

Other Observations: (Basement, septic tank, workshop, signs of spills or disposals, etc.).

**ATTACHMENT D. Example Water Well Questionnaires and Well
Survey Questionnaire and Sampling Permission
Agreement**



Well Owner Questionnaire

Interviewer: _____

Date: _____

Name of Property Owner, Lessee or Employee (circle one):

Address: _____

Phone Number: _____

Is this person aware of any water wells located on the property or on adjacent properties?

Y / N

If yes, answer the following questions.

Well No. 1

Location on property (include map): _____

Name or company of water well driller: _____

Date well was installed: _____

Other construction information:

Approximate depth: _____

Casing size: _____

Is the well currently in operation? _____

If not in operation, has the well been plugged? _____

When was it plugged? _____

When did you stop using the well? _____

Is it accessible? _____

Does it contain a pump? _____

If the well is in operation, what is the water used for (list all uses)?

Approximately how much water is used monthly? _____

Has the well ever been sampled? _____

If yes, what was is sampled for and what were the results? _____

Is there anything else you can tell us about the water well? _____

Well No. 2

Location on property (include map): _____

Name or company of water well driller: _____

Date well was installed: _____

Other construction information:

Approximate depth: _____

Casing size: _____

Is the well currently in operation? _____

If not in operation, has the well been plugged? _____

When was it plugged? _____

When did you stop using the well? _____

Is it accessible? _____

Does it contain a pump? _____

If the well is in operation, what is the water used for (list all uses)?

Approximately how much water is used monthly? _____

Has the well ever been sampled? _____

If yes, what was is sampled for and what were the results? _____

Is there anything else you can tell us about the water well? _____



Well Survey Questionnaire and Sampling Permission Agreement

Name (Please print clearly): _____

Address: _____

Daytime Phone No.: _____ Evening Phone No. _____

Are you the property owner? Yes No If not, please provide the property owner contact information.

Property Owner Name: _____ Phone No. _____

Property Owner Address: _____

Do you have a well on this property? Yes No

If yes, what is the well used for? (Check all that apply) Drinking Water Watering, Car Washing, Etc.

What is the diameter of the well in inches? _____ How deep is the well? _____

Do you have a construction log for your well? Yes No

If yes, please provide a copy with the completed questionnaire.

Has the well been treated with disinfectant such as chlorine? Yes No

If so, when was the last time it was treated? _____

Do you have a water softener? Yes No Do you have a septic tank? Yes No

If you have a septic tank, where is it located? _____

Are you willing to allow ARCADIS to collect a sample from your well? Yes No

If you authorize ARCADIS to collect a sample from your well water at above address, please sign here:

Signature

Date

Best time for Arcadis to sample your well: (Check all that apply)

Early morning before work During the day Early evening after work

Please return this form in the enclosed pre-addressed, postage paid envelope to: Arcadis, Attn:

FOR OFFICE USE:

Access Authorization Communicated: Phone call Date & Time: _____

In Person Date & Time: _____ Written Date Received: _____

Project No.: _____ Project Mgr.: _____

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TGI - POLY- AND PERFLUORINATED ALKYL SUBSTANCES (PFAS) POTABLE WATER SAMPLING GUIDANCE

Rev: 3

Rev Date: February 21, 2022

Version Control

| Issue | Revision No. | Date Issued | Page No. | Description | Reviewed By |
|---------------------------|--------------|-------------------|----------|---|---------------------------|
| -- | 0 | November 16, 2017 | All | Initial Release | Erica Kalve, Erika Houtz |
| Analytical Updates Needed | 1 | November 5, 2019 | All | General Updates to Sampling TGI, including references to USEPA Method 537.1 Version 1 | Lisa Rutkowski |
| Outdated Template | 2 | December 10, 2021 | All | Updated to current TGI format. Also updated sections to match the most recent TGI for PFAS Field Sampling Guidance (Arcadis 2021) | Kevin Engle |
| Template Updates | 3 | February 21, 2022 | All | Additional template updates | Kevin Engle, Johnsie Lang |

Approval Signatures

Prepared by:



2/21/2022

Kevin Engle, PG (WY)

Date

Reviewed by:



2/21/2022

Johnsie Lang, PhD (Subject Matter Expert)

Date

1 Introduction

This document is intended to provide guidance to field staff sampling for Per- and Polyfluoroalkyl Substances (PFAS) in potable water. The content in this document describes the intended use, scope and application, personnel qualifications, equipment, cautions, health and safety considerations, procedures, waste management, data recording and management, and quality assurance of PFAS sampling.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

The purpose of this document is to provide guidance on sampling for poly-and perfluorinated alkyl substances (PFASs) from potable water supplies. This protocol was adapted from various sources including the United States (US) Department of Defense, US Army Corp of Engineers (USACE) Omaha, Transport Canada, US Environmental Protection Agency (US EPA), and Michigan Department of Environment, Great Lakes, and Energy.

Given the extremely low detection limits associated with PFAS analysis and the many potential sources of trace levels of PFAS, field personnel are advised to err on the side of caution by strictly following these protocols to mitigate the potential for false detections of PFASs. Specific items related to field sampling for PFASs are discussed in the sections below.

4 Personnel Qualifications

4.1 Sampling Personnel

Field personnel must have current health and safety training, including 40-hour HAZWOPER training, site supervisor training, and site-specific training, as needed. In addition, field personnel must possess the skills and experience necessary to successfully complete the desired field work. The site Health and Safety Plan (HASP) and other documents will identify any other training requirements such as site-specific safety training or access control requirements.

4.2 Laboratories

As of this writing, the preferred method to analyze PFAS in drinking water is USEPA Method 537.1 Version 1.0, issued in November 2018. A laboratory accredited by the relevant federal or state accreditation agency for USEPA Method 537.1 Version 1.0 should be used to conduct the PFAS analysis.

These laboratories are examples of laboratories that may be used to analyze potable water for PFASs:

- TestAmerica Eurofins
- SGS
- Vista
- Pace

Other laboratories may be selected for analysis if they have the appropriate accreditation.

5 Equipment List

The following equipment and materials must be available for sampling:

- Site plan of sampling locations, relevant work plan (or equivalent), and this guidance document;
- Appropriate health and safety equipment, as specified in the site Healthy and Safety Plan (HASP);
- Pens, pencils, and/or fine point Sharpies® for writing;
- Clipboards, field binders, and field note pages that are not waterproof;
- High-density polyethylene (HDPE) sample bottles fitted with polypropylene or HDPE screw cap only;
- Sample labels;
- Ziploc® bags to hold wet ice and samples;
- Laboratory-supplied PFAS-free water;
- Stainless steel or PVC bailer for samples that cannot be collected out of a tap
- Coolers;
- Wet ice;
- Methanol for cleaning reusable sampling equipment (if available);
- Packing and shipping materials; and
- Chain-of-Custody (COC) Forms.

6 Cautions

6.1 Food Packaging

Some food packaging may be treated with PFAS-containing chemicals to prevent permeation of oil and water in the food outside of the packaging. To avoid potential food packaging-related PFAS contact:

- Do not bring any food outside of the field vehicles on site, and eat snacks and meals off site.
- Wash hands after eating.
- Remove any field garments or outer layers prior to eating. Do not put them back on until done eating and hands are washed.

6.2 Field Gear

6.2.1 Clothing

Many types of clothing are treated with PFASs for stain and water resistance, in particular outdoor performance wear under brand names such as Gore-Tex® or eVent™. To avoid potential clothing-related PFAS contact:

- Do not wear any outdoor performance wear that is water or stain resistant, or appears to be. Err on the side of caution.
- Wear pre-laundered (multiple washings, i.e. 6+) clothing that is not stain resistant or water proof. (unless made from the materials listed in Section 5.3.1 of the TGI for PFAS Field Sampling Guidance (Arcadis 2021))
- Natural fabrics such as cotton are preferred. Synthetic fabrics may also be acceptable if there is no indication on the label that the fabric is water and/or stain resistant.
- Most importantly, avoid contacting your clothing with sampling equipment, bottles, and samples.

6.2.2 Personal Protective Equipment

Safety Footwear

Some safety footwear has been treated to provide a degree of waterproofing and increased durability and may represent a source of trace PFAS. If at all possible, Gore-Tex footwear should not be worn and safety footwear without waterproofing should be worn; footwear that provides adequate safety from physical hazards is required and takes precedence over potential PFAS concerns. To avoid any PFAS cross contamination to samples from footwear:

- Do not touch your safety footwear in the immediate vicinity of the sampling port (i.e., within 2 feet).
- Do not allow gloves used for sampling to come in contact with safety footwear.

Nitrile Gloves

Wear disposable, powderless nitrile gloves at all times. Don a new pair of nitrile gloves **before** the following activities at each sample location:

- Contact with sample bottles or “PFAS-free” water bottles;
- Handling of any quality assurance/quality control (QA/QC) samples including field blanks and equipment blanks.

Don a new pair of nitrile gloves **after** the following activities:

- Contacting contaminated surfaces; or
- When judged necessary by field personnel.

6.3 Personal Hygiene

Some personal care products may contain PFASs. To minimize potential for cross-contamination from personal care products:

- Shower at night.
- Do not use personal care products after showering such as lotions, makeup, and perfumes, UNLESS medically necessary.
- Use sunscreen and insect repellent as necessary for health and safety, i.e., if sampling is to occur outdoors in direct sunlight and/or if insect hazards may be present. Specific products that are acceptable for PFAS sampling are listed in Table 1 and in Section 6.1. Apply sunscreen and insect repellent prior to initiating field sampling. If sunscreen and/or repellent need to be reapplied, ensure a safe distance away from the sampling locations and equipment (i.e., more than 10 meters (m) away). Wash hands after application and don new gloves following hand washing.

6.4 Visitors

If possible, visitors to the site are to remain at least 10 m from sampling areas.

7 Health and Safety Considerations

- Field activities must be performed in accordance with the site HASP, a copy of which will be present on site during such activities.
- Use caution when removing well caps as well may be under pressure, cap can dislodge forcefully and cause injury.
- Additional health and safety considerations can be found in TGI for PFAS Field Sampling Guidance (Arcadis 2021)

8 Procedure

The following section details the sample collection procedure for potable water. For additional sampling considerations, reference the TGI for PFAS Field Sampling Guidance (Arcadis 2021).

8.1 Sample Collection

Different laboratories may supply sample collection bottles of varying sizes, however all sample bottles should be made of HDPE plastic with polypropylene or HDPE plastic, unlined lids. The laboratory should specify the amount of sample required for the analysis given the anticipated detection levels.

8.1.1 Sample Containers

- Collect samples in HDPE bottles fitted with an unlined (no Teflon™), polypropylene or HDPE screw cap.
- Sample bottles must contain Trizma® preservative if samples are being collected from a chlorinated water source. The laboratory should specify the amount added to the sample container.
- Complete bottle labels after sample collection, once the caps have been placed back on each bottle.
- Do not use glass bottles due to potential loss of analyte through adsorption to glass.

8.1.2 Potable Water Sampling

Before Sample Collection

- Don a new set of powderless nitrile gloves. Do not use gloved hands to subsequently handle papers, pens, clothes, etc., before collecting samples.
- Use the HDPE bottles that are supplied by the laboratory. Samples bottle caps must remain on the bottle until immediately prior to sample collection, and the bottle must be sealed immediately after sample collection. This will minimize the potential for contamination of the sample. The bottle cap must remain in the other hand of the sampler, until replaced on the bottle. Sample bottles will not be rinsed during sampling.
- Inspect the tap prior to sampling as potable water outfalls and taps are likely to vary.
 - Avoid sampling from any taps fitted with Teflon tape or other PFAS-containing materials. If a sample can only be taken from a tap fitted with PFAS-containing materials, remove these materials prior to sampling if possible. Annotate the presence of these materials in the field notes.
 - Sample from a cold water line only.
 - Whenever possible, remove any attachments from the taps, including aerators, screens, washers, hoses, and water filters. Annotate the presence of these materials in the field notes.
 - Stainless steel and PVC are tap materials that are not expected to bias PFAS results.

During Sample Collection

- If sampling from a tap or port, in accordance with US EPA Method 537.1 sample collection procedures, begin flow from the water source and allow the system to flush for at least 3 minutes. Then, collect the sample under the still running tap.
- If a port or tap is not available to collect the water sample, use a stainless steel or HDPE bailer that has been pre-rinsed with methanol (if available) and PFAS-free water. A pump may be used if needed, but new silicone and/or HDPE outflow tubing should be used for each sample and any wetted pump parts should be decontaminated with methanol (if available) and PFAS-free water.
- Collect the sample into the HDPE bottle until the sample bottle is full to the neck of the bottle. Do not filter and do not overflow the bottle, as the preservative used for chlorinated samples may be lost. Tightly screw on the polypropylene or HDPE cap.

After Sample Collection

- Place each sample bottle in two sealed Ziploc® bags. Another brand of LDPE bag is acceptable.
- Record the sample name and time of sampling on the sample bottle label, in the field notes, and on the COC form. Record notes about the tap, including any attachments, or the conditions of how the sample was collected in the field notes.

- Place samples in coolers that are durable in transportation and keep the temperature between 0 and 4°C with wet ice until transported to the laboratory. The temperature should not exceed 10°C during the first 48 hours after sample collection, per USEPA Method 537.1.
- Treat all disposable sampling materials as single use and dispose of them appropriately after sampling at each location.

8.2 Shipping

- If samples cannot be received at the laboratory the next day (e.g., Friday sample collection), delay shipment until samples can be assured to be received. Note that samples must be extracted within 14 days of sample collection, per USEPA Method 537.1. The laboratory has an additional 28 days before the sample extract must be analyzed.
- If samples cannot be shipped the same day as collected, arrange an appropriate means of keeping the samples cool overnight (e.g., a refrigerated room or extra wet ice) and maintain the temperature between 0 and 4°C. The temperature should not exceed 10°C during the first 48 hours after sample collection, per USEPA Method 537.1.
- For shipping, store labeled samples in coolers suitable with wet ice stored in Ziploc® bags.
- Complete the appropriate procedures for COC handling, packing, and shipping.
- Fill out and check COC forms against the labels on the sample bottles progressively after each sample is collected.

Ship samples via FedEx using priority overnight delivery. Tracking numbers for all shipments should be provided and recorded to ensure their timely delivery.

9 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

If digital data collection isn't possible, waterproof field books should be avoided for field notes. Instead, field notes on loose paper on Masonite, plastic, or aluminum clip boards is preferred. Please note that newer Rite in the Rain® notebooks are approved for PFAS sampling. Other requirements for field notes include:

- Pens, pencils, and fine point Sharpies® may be used.
- Keep field notes and writing implements away from samples and sampling materials.
- Do not write on sampling bottle labels unless the sample bottle covers are tightly closed.
- Complete sampling logs in their entirety.
- Make sure COC forms are properly completed. Verify that the analysis method requested is US EPA Method 537.1 for potable water and includes the appropriate analytes desired for analysis.

10 Quality Assurance

Refer to quality control requirements for the project to ensure that appropriate QA/QC samples are collected. When collecting QA/QC samples, the same guidelines apply as when collecting regular samples – specifically that:

- Samples should be collected in laboratory-supplied HDPE bottles;
- Bottle caps must remain in the hand of the sampler until replaced on the bottle;
- Labels must be completed after the caps have been placed back on each bottle; and
- Samples must be stored in appropriate transport bottles (coolers) with ice (Ziploc® bags for use as ice containers) with appropriate labeling.

10.1 Field Duplicates

Project requirements may include the collection of one or more duplicate samples. If required, one field duplicate for every 20 samples collected or one per day, whichever is more frequent, is a typical collection frequency. Each duplicate sample will be collected immediately after the initial sample of which it is a duplicate into a separate laboratory-provided sample bottle. Do not indicate to the laboratory which sample the duplicate replicates (i.e., it should be given a blind reference on the COC form and given a sample name such as “duplicate”).

10.2 Field Blanks

QA/QC sampling for PFASs typically includes the submission of one laboratory supplied reagent field blank per day or per site. The PFAS-free water used for the reagent field blank sample is brought to the site in a laboratory-supplied bottle. Field staff should transfer the laboratory-supplied PFAS-free water into an empty sample bottle. This reagent field blank should be placed in the same cooler as other PFAS samples.

If a sampling bailer is used to collect the sample, PFAS-free water may be used to take an equipment blank through the sampler and then collected into a new sampling container.

Trip blanks are not needed, as the PFAS to be analyzed are not volatile.

10.3 Matrix Spike/ Matrix Spike Duplicate

Project requirements may include the collection of one or more matrix spikes or matrix spike duplicates. If required, one matrix spike and matrix spike duplicate for every 20 samples collected or one per day, whichever is more frequent, is a typical collection frequency. Each matrix spike sample will be collected immediately after the unspiked sample into a separate laboratory-provided sample bottle; the matrix spike and its duplicate will each require their own containers. The matrix spike and matrix spike duplicate should be clearly indicated on the laboratory COC, however their location should remain blind. The laboratory will add the appropriate chemical spike once the sample returns to the laboratory.

10.4 Laboratory Analytical QA/QC

- Internal laboratory QA/QC should consist of one laboratory blank and one laboratory control sample (or blank spike) per batch of samples, and additional QA/QCs as indicated by the laboratory QA/QC procedures. For potable water, the laboratory should follow the methodology and be accredited for analysis according to US EPA Method 537.1 Version 1. Updated potable water analytical procedures may become available and should be considered at that time.
- As part of the internal QA/QC, relative percent difference (RPD) should be calculated between samples and corresponding field or laboratory duplicates. The laboratory quality assurance portion of the laboratory certificates should be reviewed to verify that all calculations/recoveries were within acceptable limits as established by the laboratory method, typically 20% RPD.

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1.0 USE OF TERMS

Equipment blank: The equipment blank shall include the pump and the pump's tubing. If tubing is dedicated to the well, the equipment blank needs only to include the pump in subsequent sampling rounds. If the pump and tubing are dedicated to the well, the equipment blank is collected prior to its placement in the well. If the pump and tubing will be used to sample multiple wells, the equipment blank is normally collected after sampling from contaminated wells and not after background wells.

Field duplicates: Field duplicates are collected to determine precision of the sampling procedure. For this procedure, collect duplicate for each analyte group in consecutive order (VOC original, VOC duplicate, SVOC original, SVOC duplicate, etc.).

Indicator field parameters: This SOP uses field measurements of turbidity, dissolved oxygen, specific conductance, temperature, pH, and oxidation/reduction potential (ORP) as indicators of when purging operations are sufficient and sample collection may begin.

Matrix Spike/Matrix Spike Duplicates: Used by the laboratory in its quality assurance program. Consult the laboratory for the sample volume to be collected.

Potentiometric Surface: The level to which water rises in a tightly cased well constructed in a confined aquifer. In an unconfined aquifer, the potentiometric surface is the water table.

QAPP: Quality Assurance Project Plan

SAP: Sampling and Analysis Plan

SOP: Standard operating procedure

Stabilization: A condition that is achieved when all indicator field parameter measurements are sufficiently stable (as described in the "Monitoring Indicator Field Parameters" section) to allow sample collection to begin.

Temperature blank: A temperature blank is added to each sample cooler. The blank is measured upon receipt at the laboratory to assess whether the samples were properly cooled during transit.

Trip blank (VOCs): Trip blank is a sample of analyte-free water taken to the sampling site and returned to the laboratory. The trip blanks (one pair) are added to each sample cooler that contains VOC samples.

2.0 SCOPE & APPLICATION

The goal of this groundwater sampling procedure is to collect water samples that reflect the total mobile organic and inorganic loads (dissolved and colloidal sized fractions) transported through the subsurface under ambient flow conditions, with minimal physical and chemical alterations from sampling operations. This standard operating procedure (SOP) for collecting groundwater samples will help ensure that the project's data quality objectives (DQOs) are met under certain low-flow conditions.

The SOP emphasizes the need to minimize hydraulic stress at the well-aquifer interface by maintaining low water-level drawdowns, and by using low pumping rates during purging and sampling operations. Indicator field parameters (e.g., dissolved oxygen, pH, etc.) are monitored during purging in order to determine when sample collection may begin. Samples properly collected using this SOP are suitable for analysis of groundwater contaminants (volatile and semi-volatile organic analytes, dissolved gases, pesticides, PCBs, metals and other inorganics), or naturally occurring analytes. This SOP is based on Puls, and Barcelona (1996).

This procedure is designed for monitoring wells with an inside diameter (1.5-inches or greater) that can accommodate a positive lift pump with a screen length or open interval ten feet or less and with a water level above the top of the screen or open interval (Hereafter, the "screen or open interval" will be referred to only as "screen interval"). This SOP is not applicable to other well-sampling conditions.

While the use of dedicated sampling equipment is not mandatory, dedicated pumps and tubing can reduce sampling costs significantly by streamlining sampling activities and thereby reducing the overall field costs.

The goal of this procedure is to emphasize the need for consistency in deploying and operating equipment while purging and sampling monitoring wells during each sampling event. This will help to minimize sampling variability.

This procedure describes a general framework for groundwater sampling. Other site specific information (hydrogeological context, conceptual site model (CSM), DQOs, etc.) coupled with systematic planning must be added to the procedure in order to develop an appropriate site specific SAP/QAPP. In addition, the site specific SAP/QAPP must identify the specific equipment that will be used to collect the groundwater samples.

This procedure does not address the collection of water or free product samples from wells containing free phase LNAPLs and/or DNAPLs (light or dense non-aqueous phase

liquids). For this type of situation, the reader may wish to check: Cohen, and Mercer (1993) or other pertinent documents.

This SOP is to be used when collecting groundwater samples from monitoring wells at all Superfund, Federal Facility and RCRA sites in Region 1 under the conditions described herein. Request for modification of this SOP, in order to better address specific situations at individual wells, must include adequate technical justification for proposed changes. All changes and modifications must be approved and included in a revised SAP/QAPP before implementation in field.

3.0 BACKGROUND FOR IMPLEMENTATION

It is expected that the monitoring well screen has been properly located (both laterally and vertically) to intercept existing contaminant plume(s) or along flow paths of potential contaminant migration. Problems with inappropriate monitoring well placement or faulty/improper well installation cannot be overcome by even the best water sampling procedures. This SOP presumes that the analytes of interest are moving (or will potentially move) primarily through the more permeable zones intercepted by the screen interval.

Proper well construction, development, and operation and maintenance cannot be overemphasized. The use of installation techniques that are appropriate to the hydrogeologic setting of the site often prevent "problem well" situations from occurring. During well development, or redevelopment, tests should be conducted to determine the hydraulic characteristics of the monitoring well. The data can then be used to set the purging/sampling rate, and provide a baseline for evaluating changes in well performance and the potential need for well rehabilitation. Note: if this installation data or well history (construction and sampling) is not available or discoverable, for all wells to be sampled, efforts to build a sampling history should commence with the next sampling event.

The pump intake should be located within the screen interval and at a depth that will remain under water at all times. It is recommended that the intake depth and pumping rate remain the same for all sampling events. The mid-point or the lowest historical midpoint of the saturated screen length is often used as the location of the pump intake. For new wells, or for wells without pump intake depth information, the site's SAP/QAPP must provide clear reasons and instructions on how the pump intake depth(s) will be selected, and reason(s) for the depth(s) selected. If the depths to top and bottom of the well screen are not known, the SAP/QAPP will need to describe how the sampling depth will be determined and how the data can be used.

Stabilization of indicator field parameters is used to indicate that conditions are suitable for sampling to begin. Achievement of turbidity levels of less than 5 NTU, and stable drawdowns of less than 0.3 feet, while desirable, are not mandatory. Sample collection

may still take place provided the indicator field parameter criteria in this procedure are met. If after 2 hours of purging indicator field parameters have not stabilized, one of three optional courses of action may be taken: a) continue purging until stabilization is achieved, b) discontinue purging, do not collect any samples, and record in log book that stabilization could not be achieved (documentation must describe attempts to achieve stabilization), c) discontinue purging, collect samples and provide full explanation of attempts to achieve stabilization (note: there is a risk that the analytical data obtained, especially metals and strongly hydrophobic organic analytes, may reflect a sampling bias and therefore, the data may not meet the data quality objectives of the sampling event).

It is recommended that low-flow sampling be conducted when the air temperature is above 32°F (0°C). If the procedure is used below 32°F, special precautions will need to be taken to prevent the groundwater from freezing in the equipment. Because sampling during freezing temperatures may adversely impact the data quality objectives, the need for water sample collection during months when these conditions are likely to occur should be evaluated during site planning and special sampling measures may need to be developed. Ice formation in the flow-through-cell will cause the monitoring probes to act erratically. A transparent flow-through-cell needs to be used to observe if ice is forming in the cell. If ice starts to form on the other pieces of the sampling equipment, additional problems may occur.

4.0 HEALTH & SAFETY

When working on-site, comply with all applicable OSHA requirements and the site's health/safety procedures. All proper personal protection clothing and equipment are to be worn. Some samples may contain biological and chemical hazards. These samples should be handled with suitable protection to skin, eyes, etc.

5.0 CAUTIONS

The following cautions need to be considered when planning to collect groundwater samples when the below conditions occur.

If the groundwater degasses during purging of the monitoring well, dissolved gases and VOCs will be lost. When this happens, the groundwater data for dissolved gases (e.g., methane, ethene, ethane, dissolved oxygen, etc.) and VOCs will need to be qualified. Some conditions that can promote degassing are the use of a vacuum pump (e.g., peristaltic pumps), changes in aperture along the sampling tubing, and squeezing/pinching the pump's tubing which results in a pressure change.

When collecting the samples for dissolved gases and VOCs analyses, avoid aerating the groundwater in the pump's tubing. This can cause loss of the dissolved gases and VOCs in

the groundwater. Having the pump's tubing completely filled prior to sampling will avoid this problem when using a centrifugal pump or peristaltic pump.

Direct sun light and hot ambient air temperatures may cause the groundwater in the tubing and flow-through-cell to heat up. This may cause the groundwater to degas which will result in loss of VOCs and dissolved gases. When sampling under these conditions, the sampler will need to shade the equipment from the sunlight (e.g., umbrella, tent, etc.). If possible, sampling on hot days, or during the hottest time of the day, should be avoided. The tubing exiting the monitoring well should be kept as short as possible to avoid the sun light or ambient air from heating up the groundwater.

Thermal currents in the monitoring well may cause vertical mixing of water in the well bore. When the air temperature is colder than the groundwater temperature, it can cool the top of the water column. Colder water which is denser than warm water sinks to the bottom of the well and the warmer water at the bottom of the well rises, setting up a convection cell. "During low-flow sampling, the pumped water may be a mixture of convecting water from within the well casing and aquifer water moving inward through the screen. This mixing of water during low-flow sampling can substantially increase equilibration times, can cause false stabilization of indicator parameters, can give false indication of redox state, and can provide biological data that are not representative of the aquifer conditions" (Vrobesky 2007).

Failure to calibrate or perform proper maintenance on the sampling equipment and measurement instruments (e.g., dissolved oxygen meter, etc.) can result in faulty data being collected.

Interferences may result from using contaminated equipment, cleaning materials, sample containers, or uncontrolled ambient/surrounding air conditions (e.g., truck/vehicle exhaust nearby).

Cross contamination problems can be eliminated or minimized through the use of dedicated sampling equipment and/or proper planning to avoid ambient air interferences. Note that the use of dedicated sampling equipment can also significantly reduce the time needed to complete each sampling event, will promote consistency in the sampling, and may reduce sampling bias by having the pump's intake at a constant depth.

Clean and decontaminate all sampling equipment prior to use. All sampling equipment needs to be routinely checked to be free from contaminants and equipment blanks collected to ensure that the equipment is free of contaminants. Check the previous equipment blank data for the site (if they exist) to determine if the previous cleaning procedure removed the contaminants. If contaminants were detected and they are a concern, then a more vigorous cleaning procedure will be needed.

6.0 PERSONNEL QUALIFICATIONS

All field samplers working at sites containing hazardous waste must meet the requirements of the OSHA regulations. OSHA regulations may require the sampler to take the 40 hour OSHA health and safety training course and a refresher course prior to engaging in any field activities, depending upon the site and field conditions.

The field samplers must be trained prior to the use of the sampling equipment, field instruments, and procedures. Training is to be conducted by an experienced sampler before initiating any sampling procedure.

The entire sampling team needs to read, and be familiar with, the site Health and Safety Plan, all relevant SOPs, and SAP/QAPP (and the most recent amendments) before going onsite for the sampling event. It is recommended that the field sampling leader attest to the understanding of these site documents and that it is recorded.

7.0 EQUIPMENT AND SUPPLIES

A. Informational materials for sampling event

A copy of the current Health and Safety Plan, SAP/QAPP, monitoring well construction data, location map(s), field data from last sampling event, manuals for sampling, and the monitoring instruments' operation, maintenance, and calibration manuals should be brought to the site.

B. Well keys.

C. Extraction device

Adjustable rate, submersible pumps (e.g., centrifugal, bladder, etc.) which are constructed of stainless steel or polytetrafluoroethylene (PTFE, i.e. Teflon®) are preferred. PTFE, however, should not be used when sampling for per- and polyfluoroalkyl substances (PFAS) as it is likely to contain these substances.

Note: If extraction devices constructed of other materials are to be used, adequate information must be provided to show that the substituted materials do not leach contaminants nor cause interferences to the analytical procedures to be used. Acceptance of these materials must be obtained before the sampling event.

If bladder pumps are selected for the collection of VOCs and dissolved gases, the pump setting should be set so that one pulse will deliver a water volume that is sufficient to fill a 40 mL VOC vial. This is not mandatory, but is considered a “best practice”. For the proper operation, the bladder pump will need a minimum amount of water above the pump; consult the manufacturer for the recommended submergence. The pump’s recommended submergence value should be determined during the planning stage, since it may influence well construction and placement of dedicated pumps where water-level fluctuations are significant.

Adjustable rate, peristaltic pumps (suction) are to be used with caution when collecting samples for VOCs and dissolved gases (e.g., methane, carbon dioxide, etc.) analyses. Additional information on the use of peristaltic pumps can be found in Appendix A. If peristaltic pumps are used, the inside diameter of the rotor head tubing needs to match the inside diameter of the tubing installed in the monitoring well.

Inertial pumping devices (motor driven or manual) are not recommended. These devices frequently cause greater disturbance during purging and sampling, and are less easily controlled than submersible pumps (potentially increasing turbidity and sampling variability, etc.). This can lead to sampling results that are adversely affected by purging and sampling operations, and a higher degree of data variability.

D. Tubing

PTFE (Teflon®) or PTFE-lined polyethylene tubing are preferred when sampling is to include VOCs, SVOCs, pesticides, PCBs and inorganics. As discussed in the previous section, PTFE tubing should not be used when sampling for PFAS. In this case, a suitable alternative such as high-density polyethylene tubing should be used.

PVC, polypropylene or polyethylene tubing may be used when collecting samples for metal and other inorganics analyses.

Note: If tubing constructed of other materials is to be used, adequate information must be provided to show that the substituted materials do not leach contaminants nor cause interferences to the analytical procedures to be used. Acceptance of these materials must be obtained before the sampling event.

The use of 1/4 inch or 3/8 inch (inside diameter) tubing is recommended. This will help ensure that the tubing remains liquid filled when operating at very low pumping rates when using centrifugal and peristaltic pumps.

Silastic tubing should be used for the section around the rotor head of a peristaltic pump. It should be less than a foot in length. The inside diameter of the tubing used at the pump rotor head must be the same as the inside diameter of tubing placed in the well. A tubing connector is used to connect the pump rotor head tubing to the well tubing. Alternatively, the two pieces of tubing can be connected to each other by placing the one end of the tubing inside the end of the other tubing. The tubing must not be reused.

E. The water level measuring device

Electronic "tape", pressure transducer, water level sounder/level indicator, etc. should be capable of measuring to 0.01 foot accuracy. Recording pressure transducers, mounted above the pump, are especially helpful in tracking water levels during pumping operations, but their use must include check measurements with a water level "tape" at the start and end of each sampling event.

F. Flow measurement supplies

Graduated cylinder (size according to flow rate) and stopwatch usually will suffice.

Large graduated bucket used to record total water purged from the well.

G. Interface probe

To be used to check on the presence of free phase liquids (LNAPL, or DNAPL) before purging begins (as needed).

H. Power source (generator, nitrogen tank, battery, etc.)

When a gasoline generator is used, locate it downwind and at least 30 feet from the well so that the exhaust fumes do not contaminate samples.

I. Indicator field parameter monitoring instruments

Use of a multi-parameter instrument capable of measuring pH, oxidation/reduction potential (ORP), dissolved oxygen (DO), specific conductance, temperature, and coupled with a flow-through-cell is required when measuring all indicator field parameters, except turbidity. Turbidity is collected using a separate instrument. Record equipment/instrument identification (manufacturer, and model number).

Transparent, small volume flow-through-cells (e.g., 250 mLs or less) are preferred. This allows observation of air bubbles and sediment buildup in the cell, which can interfere with the operation of the monitoring instrument probes, to be easily detected. A small volume

cell facilitates rapid turnover of water in the cell between measurements of the indicator field parameters.

It is recommended to use a flow-through-cell and monitoring probes from the same manufacturer and model to avoid incompatibility between the probes and flow-through-cell.

Turbidity samples are collected before the flow-through-cell. A “T” connector coupled with a valve is connected between the pump’s tubing and flow-through-cell. When a turbidity measurement is required, the valve is opened to allow the groundwater to flow into a container. The valve is closed and the container sample is then placed in the turbidimeter.

Standards are necessary to perform field calibration of instruments. A minimum of two standards are needed to bracket the instrument measurement range for all parameters except ORP which use a Zobell solution as a standard. For dissolved oxygen, a wet sponge used for the 100% saturation and a zero dissolved oxygen solution are used for the calibration.

Barometer (used in the calibration of the Dissolved Oxygen probe) and the conversion formula to convert the barometric pressure into the units of measure used by the Dissolved Oxygen meter are needed.

J. Decontamination supplies

Includes (for example) non-phosphate detergent, distilled/deionized water, isopropyl alcohol, etc.

K. Record keeping supplies

Logbook(s), well purging forms, chain-of-custody forms, field instrument calibration forms, etc.

L. Sample bottles

M. Sample preservation supplies (as required by the analytical methods)

N. Sample tags or labels

O. PID or FID instrument

If appropriate, to detect VOCs for health and safety purposes, and provide qualitative field evaluations.

P. Miscellaneous Equipment

Equipment to keep the sampling apparatus shaded in the summer (e.g., umbrella) and from freezing in the winter. If the pump's tubing is allowed to heat up in the warm weather, the cold groundwater may degas as it is warmed in the tubing.

8.0 EQUIPMENT/INSTRUMENT CALIBRATION

Prior to the sampling event, perform maintenance checks on the equipment and instruments according to the manufacturer's manual and/or applicable SOP. This will ensure that the equipment/instruments are working properly before they are used in the field.

Prior to sampling, the monitoring instruments must be calibrated and the calibration documented. The instruments are calibrated using U.S Environmental Protection Agency Region 1 *Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction [ORP], and turbidity)*, March 23, 2017, or latest version or from one of the methods listed in 40CFR136, 40CFR141 and SW-846.

The instruments shall be calibrated at the beginning of each day. If the field measurement falls outside the calibration range, the instrument must be re-calibrated so that all measurements fall within the calibration range. At the end of each day, a calibration check is performed to verify that instruments remained in calibration throughout the day. This check is performed while the instrument is in measurement mode, not calibration mode. If the field instruments are being used to monitor the natural attenuation parameters, then a calibration check at mid-day is highly recommended to ensure that the instruments did not drift out of calibration. Note: during the day if the instrument reads zero or a negative number for dissolved oxygen, pH, specific conductance, or turbidity (negative value only), this indicates that the instrument drifted out of calibration or the instrument is malfunctioning. If this situation occurs the data from this instrument will need to be qualified or rejected.

9.0 PRELIMINARY SITE ACTIVITIES (as applicable)

Check the well for security (damage, evidence of tampering, missing lock, etc.) and record pertinent observations (include photograph as warranted).

If needed, lay out a sheet of clean polyethylene for monitoring and sampling equipment, unless equipment is elevated above the ground (e.g., on a table, etc.).

Remove well cap and if appropriate measure VOCs at the rim of the well with a PID or FID instrument and record reading in field logbook or on the well purge form.

If the well casing does not have an established reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook (consider a photographic record as well). All water level measurements must be recorded relative to this reference point (and the altitude of this point should be determined using techniques that are appropriate to site's DQOs).

If water-table or potentiometric surface map(s) are to be constructed for the sampling event, perform synoptic water level measurement round (in the shortest possible time) before any purging and sampling activities begin. If possible, measure water level depth (to 0.01 ft.) and total well depth (to 0.1 ft.) the day before sampling begins, in order to allow for re-settlement of any particulates in the water column. This is especially important for those wells that have not been recently sampled because sediment buildup in the well may require the well to be redeveloped. If measurement of total well depth is not made the day before, it should be measured after sampling of the well is complete. All measurements must be taken from the established referenced point. Care should be taken to minimize water column disturbance.

Check newly constructed wells for the presence of LNAPLs or DNAPLs before the initial sampling round. If none are encountered, subsequent check measurements with an interface probe may not be necessary unless analytical data or field analysis signal a worsening situation. This SOP cannot be used in the presence of LNAPLs or DNAPLs. If NAPLs are present, the project team must decide upon an alternate sampling method. All project modifications must be approved and documented prior to implementation.

If available check intake depth and drawdown information from previous sampling event(s) for each well. Duplicate, to the extent practicable, the intake depth and extraction rate (use final pump dial setting information) from previous event(s). If changes are made in the intake depth or extraction rate(s) used during previous sampling event(s), for either portable or dedicated extraction devices, record new values, and explain reasons for the changes in the field logbook.

10.0 PURGING AND SAMPLING PROCEDURE

Purging and sampling wells in order of increasing chemical concentrations (known or anticipated) are preferred.

The use of dedicated pumps is recommended to minimize artificial mobilization and entrainment of particulates each time the well is sampled. Note that the use of dedicated sampling equipment can also significantly reduce the time needed to complete each sampling event, will promote consistency in the sampling, and may reduce sampling bias by having the pump's intake at a constant depth.

A. Initial Water Level

Measure the water level in the well before installing the pump if a non-dedicated pump is being used. The initial water level is recorded on the purge form or in the field logbook.

B. Install Pump

Lower pump, safety cable, tubing and electrical lines slowly (to minimize disturbance) into the well to the appropriate depth (may not be the mid-point of the screen/open interval). The Sampling and Analysis Plan/Quality Assurance Project Plan should specify the sampling depth (used previously), or provide criteria for selection of intake depth for each new well. If possible keep the pump intake at least two feet above the bottom of the well, to minimize mobilization of particulates present in the bottom of the well.

Pump tubing lengths, above the top of well casing should be kept as short as possible to minimize heating the groundwater in the tubing by exposure to sun light and ambient air temperatures. Heating may cause the groundwater to degas, which is unacceptable for the collection of samples for VOC and dissolved gases analyses.

C. Measure Water Level

Before starting pump, measure water level. Install recording pressure transducer, if used to track drawdowns, to initialize starting condition.

D. Purge Well

From the time the pump starts purging and until the time the samples are collected, the purged water is discharged into a graduated bucket to determine the total volume of groundwater purged. This information is recorded on the purge form or in the field logbook.

Start the pump at low speed and slowly increase the speed until discharge occurs. Check water level. Check equipment for water leaks and if present fix or replace the affected equipment. Try to match pumping rate used during previous sampling event(s). Otherwise, adjust pump speed until there is little or no water level drawdown. If the

minimal drawdown that can be achieved exceeds 0.3 feet, but remains stable, continue purging.

Monitor and record the water level and pumping rate every five minutes (or as appropriate) during purging. Record any pumping rate adjustments (both time and flow rate). Pumping rates should, as needed, be reduced to the minimum capabilities of the pump to ensure stabilization of the water level. Adjustments are best made in the first fifteen minutes of pumping in order to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover" somewhat as pump flow adjustments are made. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. If the initial water level is above the top of the screen do not allow the water level to fall into the well screen. The final purge volume must be greater than the stabilized drawdown volume plus the pump's tubing volume. If the drawdown has exceeded 0.3 feet and stabilizes, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are collected.

Avoid the use of constriction devices on the tubing to decrease the flow rate because the constrictor will cause a pressure difference in the water column. This will cause the groundwater to degas and result in a loss of VOCs and dissolved gasses in the groundwater samples.

Note: the flow rate used to achieve a stable pumping level should remain constant while monitoring the indicator parameters for stabilization and while collecting the samples.

Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (e.g., bladder, peristaltic), and/or the use of dedicated equipment. For new monitoring wells, or wells where the following situation has not occurred before, if the recovery rate to the well is less than 50 mL/min., or the well is being essentially dewatered during purging, the well should be sampled as soon as the water level has recovered sufficiently to collect the volume needed for all anticipated samples. The project manager or field team leader will need to make the decision when samples should be collected, how the sample is to be collected, and the reasons recorded on the purge form or in the field logbook. A water level measurement needs to be performed and recorded before samples are collected. If the project manager decides to collect the samples using the pump, it is best during this recovery period that the pump intake tubing not be removed, since this will aggravate any turbidity problems. Samples in this specific situation may be collected without stabilization of indicator field parameters. Note that field conditions and efforts to overcome problematic situations must be recorded in order to support field decisions to deviate from normal procedures described in this SOP. If this type of problematic situation persists in a well, then water sample collection should be

changed to a passive or no-purge method, if consistent with the site's DQOs, or have a new well installed.

E. Monitor Indicator Field Parameters

After the water level has stabilized, connect the "T" connector with a valve and the flow-through-cell to monitor the indicator field parameters. If excessive turbidity is anticipated or encountered with the pump startup, the well may be purged for a while without connecting up the flow-through-cell, in order to minimize particulate buildup in the cell (This is a judgment call made by the sampler). Water level drawdown measurements should be made as usual. If possible, the pump may be installed the day before purging to allow particulates that were disturbed during pump insertion to settle.

During well purging, monitor indicator field parameters (turbidity, temperature, specific conductance, pH, ORP, DO) at a frequency of five minute intervals or greater. The pump's flow rate must be able to "turn over" at least one flow-through-cell volume between measurements (for a 250 mL flow-through-cell with a flow rate of 50 mLs/min., the monitoring frequency would be every five minutes; for a 500 mL flow-through-cell it would be every ten minutes). If the cell volume cannot be replaced in the five minute interval, then the time between measurements must be increased accordingly. Note: during the early phase of purging, emphasis should be put on minimizing and stabilizing pumping stress, and recording those adjustments followed by stabilization of indicator parameters. Purging is considered complete and sampling may begin when all the above indicator field parameters have stabilized. Stabilization is considered to be achieved when three consecutive readings are within the following limits:

Turbidity (10% for values greater than 5 NTU; if three Turbidity values are less than 5 NTU, consider the values as stabilized),

Dissolved Oxygen (10% for values greater than 0.5 mg/L, if three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized),

Specific Conductance (3%),

Temperature (3%),

pH (± 0.1 unit),

Oxidation/Reduction Potential (± 10 millivolts).

All measurements, except turbidity, must be obtained using a flow-through-cell. Samples for turbidity measurements are obtained before water enters the flow-through-cell. Transparent flow-through-cells are preferred, because they allow field personnel to watch for particulate build-up within the cell. This build-up may affect indicator field parameter values measured within the cell. If the cell needs to be cleaned during purging operations, continue pumping and disconnect cell for cleaning, then reconnect after cleaning and

continue monitoring activities. Record start and stop times and give a brief description of cleaning activities.

The flow-through-cell must be designed in a way that prevents gas bubble entrapment in the cell. Placing the flow-through-cell at a 45 degree angle with the port facing upward can help remove bubbles from the flow-through-cell (see Appendix B Low-Flow Setup Diagram). Throughout the measurement process, the flow-through-cell must remain free of any gas bubbles. Otherwise, the monitoring probes may act erratically. When the pump is turned off or cycling on/off (when using a bladder pump), water in the cell must not drain out. Monitoring probes must remain submerged in water at all times.

F. Collect Water Samples

When samples are collected for laboratory analyses, the pump's tubing is disconnected from the "T" connector with a valve and the flow-through-cell. The samples are collected directly from the pump's tubing. Samples must not be collected from the flow-through-cell or from the "T" connector with a valve.

VOC samples are normally collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling locations; the SAP/QAPP should list the order in which the samples are to be collected based on the project's objective(s). Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

If the pump's flow rate is too high to collect the VOC/dissolved gases samples, collect the other samples first. Lower the pump's flow rate to a reasonable rate and collect the VOC/dissolved gases samples and record the new flow rate.

During purging and sampling, the centrifugal/peristaltic pump tubing must remain filled with water to avoid aeration of the groundwater. It is recommended that 1/4 inch or 3/8 inch (inside diameter) tubing be used to help ensure that the sample tubing remains water filled. If the pump tubing is not completely filled to the sampling point, use the following procedure to collect samples: collect non-VOC/dissolved gases samples first, then increase flow rate slightly until the water completely fills the tubing, collect the VOC/dissolved gases samples, and record new drawdown depth and flow rate.

For bladder pumps that will be used to collect VOC or dissolved gas samples, it is recommended that the pump be set to deliver long pulses of water so that one pulse will fill a 40 mL VOC vial.

Use pre-preserved sample containers or add preservative, as required by analytical methods, to the samples immediately after they are collected. Check the analytical methods

(e.g. EPA SW-846, 40 CFR 136, water supply, etc.) for additional information on preservation.

If determination of filtered metal concentrations is a sampling objective, collect filtered water samples using the same low flow procedures. The use of an in-line filter (transparent housing preferred) is required, and the filter size (0.45 μm is commonly used) should be based on the sampling objective. Pre-rinse the filter with groundwater prior to sample collection. Make sure the filter is free of air bubbles before samples are collected. Preserve the filtered water sample immediately. Note: filtered water samples are not an acceptable substitute for unfiltered samples when the monitoring objective is to obtain chemical concentrations of total mobile contaminants in groundwater for human health or ecological risk calculations.

Label each sample as collected. Samples requiring cooling will be placed into a cooler with ice or refrigerant for delivery to the laboratory. Metal samples after acidification to a pH less than 2 do not need to be cooled.

G. Post Sampling Activities

If a recording pressure transducer is used to track drawdown, re-measure water level with tape.

After collection of samples, the pump tubing may be dedicated to the well for re-sampling (by hanging the tubing inside the well), decontaminated, or properly discarded.

Before securing the well, measure and record the well depth (to 0.1 ft.), if not measured the day before purging began. Note: measurement of total well depth annually is usually sufficient after the initial low stress sampling event. However, a greater frequency may be needed if the well has a “silting” problem or if confirmation of well identity is needed.

Secure the well.

11.0 DECONTAMINATION

Decontaminate sampling equipment prior to use in the first well, and then following sampling of each subsequent well. Pumps should not be removed between purging and sampling operations. The pump, tubing, support cable and electrical wires which were in contact with the well should be decontaminated by one of the procedures listed below.

The use of dedicated pumps and tubing will reduce the amount of time spent on decontamination of the equipment. If dedicated pumps and tubing are used, only the initial sampling event will require decontamination of the pump and tubing.

Note if the previous equipment blank data showed that contaminant(s) were present after using the below procedure or the one described in the SAP/QAPP, a more vigorous procedure may be needed.

Procedure 1

Decontaminating solutions can be pumped from either buckets or short PVC casing sections through the pump and tubing. The pump may be disassembled and flushed with the decontaminating solutions. It is recommended that detergent and alcohol be used sparingly in the decontamination process and water flushing steps be extended to ensure that any sediment trapped in the pump is removed. The pump exterior and electrical wires must be rinsed with the decontaminating solutions, as well. The procedure is as follows:

Flush the equipment/pump with potable water.

Flush with non-phosphate detergent solution. If the solution is recycled, the solution must be changed periodically.

Flush with potable or distilled/deionized water to remove all of the detergent solution. If the water is recycled, the water must be changed periodically.

Optional - flush with isopropyl alcohol (pesticide grade; must be free of ketones {e.g., acetone}) or with methanol. This step may be required if the well is highly contaminated or if the equipment blank data from the previous sampling event show that the level of contaminants is significant.

Flush with distilled/deionized water. This step must remove all traces of alcohol (if used) from the equipment. The final water rinse must not be recycled.

Procedure 2

Steam clean the outside of the submersible pump.

Pump hot potable water from the steam cleaner through the inside of the pump. This can be accomplished by placing the pump inside a three or four inch diameter PVC pipe with end cap. Hot water from the steam cleaner jet will be directed inside the PVC pipe and the pump exterior will be cleaned. The hot water from the steam cleaner will then be pumped from the PVC pipe through the pump and collected into another container. Note: additives or solutions should not be added to the steam cleaner.

Pump non-phosphate detergent solution through the inside of the pump. If the solution is recycled, the solution must be changed periodically.

Pump potable water through the inside of the pump to remove all of the detergent solution. If the solution is recycled, the solution must be changed periodically.

Pump distilled/deionized water through the pump. The final water rinse must not be recycled.

12.0 FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the groundwater samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation. Quality control samples include field duplicates, equipment blanks, matrix spike/matrix spike duplicates, trip blanks (VOCs), and temperature blanks.

13.0 FIELD LOGBOOK

A field log shall be kept to document all groundwater field monitoring activities (see Appendix C, example table), and record the following for each well:

Site name, municipality, state.

Well identifier, latitude-longitude or state grid coordinates.

Measuring point description (e.g., north side of PVC pipe).

Well depth, and measurement technique.

Well screen length.

Pump depth.

Static water level depth, date, time and measurement technique.

Presence and thickness of immiscible liquid (NAPL) layers and detection method.

Pumping rate, drawdown, indicator parameters values, calculated or measured total volume pumped, and clock time of each set of measurements.

Type of tubing used and its length.

Type of pump used.

Clock time of start and end of purging and sampling activity.

Types of sample bottles used and sample identification numbers.

Preservatives used.

Parameters requested for analyses.

Field observations during sampling event.

Name of sample collector(s).

Weather conditions, including approximate ambient air temperature.

QA/QC data for field instruments.

Any problems encountered should be highlighted.

Description of all sampling/monitoring equipment used, including trade names, model number, instrument identification number, diameters, material composition, etc.

14.0 DATA REPORT

Data reports are to include laboratory analytical results, QA/QC information, field indicator parameters measured during purging, field instrument calibration information, and whatever other field logbook information is needed to allow for a full evaluation of data usability.

Note: the use of trade, product, or firm names in this sampling procedure is for descriptive purposes only and does not constitute endorsement by the U.S. EPA.

15.0 REFERENCES

Cohen, R.M. and J.W. Mercer, 1993, *DNAPL Site Evaluation*; C.K. Smoley (CRC Press), Boca Raton, Florida.

Robert W. Puls and Michael J. Barcelona, *Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures*, April 1996 (EPA/540/S-95/504).

U.S. Environmental Protection Agency, 1992, *RCRA Ground-Water Monitoring: Draft Technical Guidance*; Washington, DC (EPA/530-R-93-001).

U.S. Environmental Protection Agency, 1987, *A Compendium of Superfund Field Operations Methods*; Washington, DC (EPA/540/P-87/001).

U.S. Environmental Protection Agency, Region 1, *Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction [ORP], and turbidity)*, March 23, 2017 or latest version.

U.S. Environmental Protection Agency, EPA SW-846.

U.S. Environmental Protection Agency, 40 CFR 136.

U.S. Environmental Protection Agency, 40 CFR 141.

Vroblesky, Don A., Clifton C. Casey, and Mark A. Lowery, Summer 2007, Influence of Dissolved Oxygen Convection on Well Sampling, *Ground Water Monitoring & Remediation* 27, no. 3: 49-58.

APPENDIX A

PERISTALTIC PUMPS

Before selecting a peristaltic pump to collect groundwater samples for VOCs and/or dissolved gases, (e.g., methane, carbon dioxide, etc.) consideration should be given to the following:

- The decision of whether or not to use a peristaltic pump is dependent on the intended use of the data.
- If the additional sampling error that may be introduced by this device is NOT of concern for the VOC/dissolved gases data's intended use, then this device may be acceptable.
- If minor differences in the groundwater concentrations could affect the decision, such as to continue or terminate groundwater cleanup or whether the cleanup goals have been reached, then this device should NOT be used for VOC/dissolved gases sampling. In these cases, centrifugal or bladder pumps are a better choice for more accurate results.

EPA and USGS have documented their concerns with the use of the peristaltic pumps to collect water sample in the below documents.

- "Suction Pumps are not recommended because they may cause degassing, pH modification, and loss of volatile compounds" *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001, December 1987.
- "The agency does not recommend the use of peristaltic pumps to sample ground water particularly for volatile organic analytes" *RCRA Ground-Water Monitoring Draft Technical Guidance*, EPA Office of Solid Waste, November 1992.
- "The peristaltic pump is limited to shallow applications and can cause degassing resulting in alteration of pH, alkalinity, and volatiles loss", *Low-flow (Minimal drawdown) Ground-Water Sampling Procedures*, by Robert Puls & Michael Barcelona, April 1996, EPA/540/S-95/504.
- "Suction-lift pumps, such as peristaltic pumps, can operate at a very low pumping rate; however, using negative pressure to lift the sample can result in the loss of volatile analytes", USGS Book 9 Techniques of Water-Resources Investigation, Chapter A4. (Version 2.0, 9/2006).

APPENDIX B

SUMMARY OF SAMPLING INSTRUCTIONS

These instructions are for using an adjustable rate, submersible pump or a peristaltic pump with the pump's intake placed at the midpoint of a 10 foot or less well screen or an open interval. The water level in the monitoring well is above the top of the well screen or open interval, the ambient temperature is above 32°F, and the equipment is not dedicated. Field instruments are already calibrated. The equipment is setup according to the diagram at the end of these instructions.

1. Review well installation information. Record well depth, length of screen or open interval, and depth to top of the well screen. Determine the pump's intake depth (e.g., mid-point of screen/open interval).
2. On the day of sampling, check security of the well casing, perform any safety checks needed for the site, lay out a sheet of polyethylene around the well (if necessary), and setup the equipment. If necessary a canopy or an equivalent item can be setup to shade the pump's tubing and flow-through-cell from the sun light to prevent the sun light from heating the groundwater.
3. Check well casing for a reference mark. If missing, make a reference mark. Measure the water level (initial) to 0.01 ft. and record this information.
4. Install the pump's intake to the appropriate depth (e.g., midpoint) of the well screen or open interval. Do not turn-on the pump at this time.
5. Measure water level and record this information.
6. Turn-on the pump and discharge the groundwater into a graduated waste bucket. Slowly increase the flow rate until the water level starts to drop. Reduce the flow rate slightly so the water level stabilizes. Record the pump's settings. Calculate the flow rate using a graduated container and a stop watch. Record the flow rate. Do not let the water level drop below the top of the well screen.

If the groundwater is highly turbid or discolored, continue to discharge the water into the bucket until the water clears (visual observation); this usually takes a few minutes. The turbid or discolored water is usually from the well-being disturbed during the pump installation. If the water does not clear, then you need to make a choice whether to continue purging the well (hoping that it will clear after a reasonable time) or continue to

the next step. Note, it is sometimes helpful to install the pump the day before the sampling event so that the disturbed materials in the well can settle out.

If the water level drops to the top of the well screen during the purging of the well, stop purging the well, and do the following:

Wait for the well to recharge to a sufficient volume so samples can be collected. This may take a while (pump may be removed from well, if turbidity is not a problem). The project manager will need to make the decision when samples should be collected and the reasons recorded in the site's log book. A water level measurement needs to be performed and recorded before samples are collected. When samples are being collected, the water level must not drop below the top of the screen or open interval. Collect the samples from the pump's tubing. Always collect the VOCs and dissolved gases samples first. Normally, the samples requiring a small volume are collected before the large volume samples are collected just in case there is not sufficient water in the well to fill all the sample containers. All samples must be collected, preserved, and stored according to the analytical method. Remove the pump from the well and decontaminate the sampling equipment.

If the water level has dropped 0.3 feet or less from the initial water level (water level measure before the pump was installed); proceed to Step 7. If the water level has dropped more than 0.3 feet, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are be collected.

7. Attach the pump's tubing to the "T" connector with a valve (or a three-way stop cock). The pump's tubing from the well casing to the "T" connector must be as short as possible to prevent the groundwater in the tubing from heating up from the sun light or from the ambient air. Attach a short piece of tubing to the other end of the end of the "T" connector to serve as a sampling port for the turbidity samples. Attach the remaining end of the "T" connector to a short piece of tubing and connect the tubing to the flow-through-cell bottom port. To the top port, attach a small piece of tubing to direct the water into a calibrated waste bucket. Fill the cell with the groundwater and remove all gas bubbles from the cell. Position the flow-through-cell in such a way that if gas bubbles enter the cell they can easily exit the cell. If the ports are on the same side of the cell and the cell is cylindrical shape, the cell can be placed at a 45-degree angle with the ports facing upwards; this position should keep any gas bubbles entering the cell away from the monitoring probes and allow the gas bubbles to exit the cell easily (see Low-Flow Setup Diagram). Note:

make sure there are no gas bubbles caught in the probes' protective guard; you may need to shake the cell to remove these bubbles.

8. Turn-on the monitoring probes and turbidity meter.

9. Record the temperature, pH, dissolved oxygen, specific conductance, and oxidation/reduction potential measurements. Open the valve on the "T" connector to collect a sample for the turbidity measurement, close the valve, do the measurement, and record this measurement. Calculate the pump's flow rate from the water exiting the flow-through-cell using a graduated container and a stop watch, and record the measurement. Measure and record the water level. Check flow-through-cell for gas bubbles and sediment; if present, remove them.

10. Repeat Step 9 every 5 minutes or as appropriate until monitoring parameters stabilized. Note: at least one flow-through-cell volume must be exchanged between readings. If not, the time interval between readings will need to be increased. Stabilization is achieved when three consecutive measurements are within the following limits:

Turbidity (10% for values greater than 5 NTUs; if three Turbidity values are less than 5 NTUs, consider the values as stabilized),

Dissolved Oxygen (10% for values greater than 0.5 mg/L, if three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized),

Specific Conductance (3%),

Temperature (3%),

pH (± 0.1 unit),

Oxidation/Reduction Potential (± 10 millivolts).

If these stabilization requirements do not stabilize in a reasonable time, the probes may have been coated from the materials in the groundwater, from a buildup of sediment in the flow-through-cell, or a gas bubble is lodged in the probe. The cell and the probes will need to be cleaned. Turn-off the probes (not the pump), disconnect the cell from the "T" connector and continue to purge the well. Disassemble the cell, remove the sediment, and clean the probes according to the manufacturer's instructions. Reassemble the cell and connect the cell to the "T" connector. Remove all gas bubbles from the cell, turn-on the probes, and continue the measurements. Record the time the cell was cleaned.

11. When it is time to collect the groundwater samples, turn-off the monitoring probes, and disconnect the pump's tubing from the "T" connector. If you are using a centrifugal or peristaltic pump check the pump's tubing to determine if the tubing is completely filled with water (no air space).

All samples must be collected and preserved according to the analytical method. VOCs and dissolved gases samples are normally collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling locations; the SAP/QAPP should list the order in which the samples are to be collected based on the project's objective(s). Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

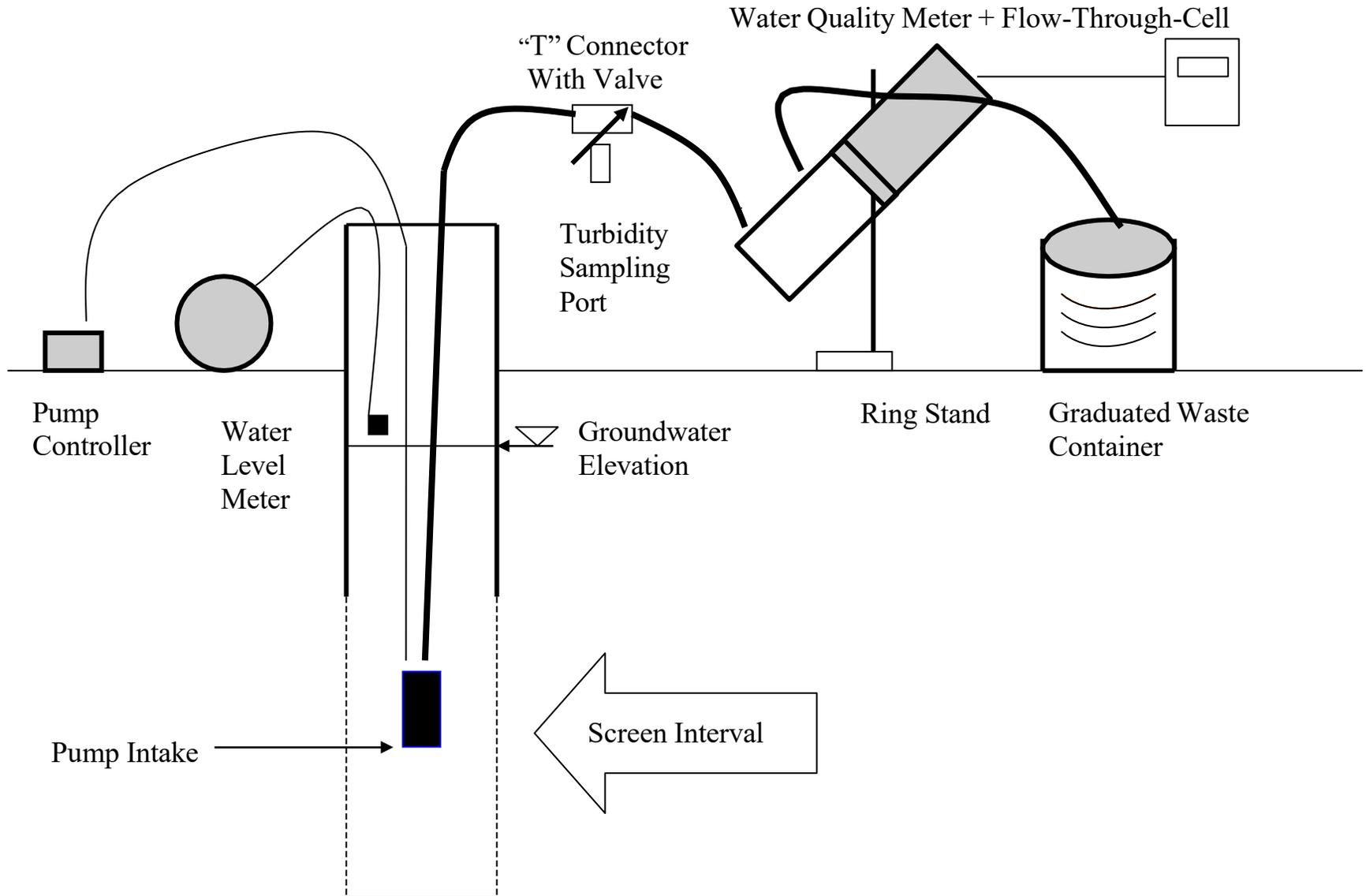
If the pump's tubing is not completely filled with water and the samples are being collected for VOCs and/or dissolved gases analyses using a centrifugal or peristaltic pump, do the following:

All samples must be collected and preserved according to the analytical method. The VOCs and the dissolved gases (e.g., methane, ethane, ethene, and carbon dioxide) samples are collected last. When it becomes time to collect these samples increase the pump's flow rate until the tubing is completely filled. Collect the samples and record the new flow rate.

12. Store the samples according to the analytical method.

13. Record the total purged volume (graduated waste bucket). Remove the pump from the well and decontaminate the sampling equipment.

Low-Flow Setup Diagram



TGI - Manual Water-Level and NAPL Monitoring

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Approval Signatures

Prepared by:



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Everett H. Fortner III, PG (Preparer)

Date

Reviewed by:



3/1/2023

Marc Killingstad, PE (Subject Matter Expert)

Date

1 Introduction

This TGI describes the equipment, field procedures, materials, and documentation procedures to measure and record water-levels using an electronic water-level probe or an oil-water level indicator. This TGI also describes procedures for measuring in-well thicknesses of non-aqueous phase liquid (NAPL), both light and/or dense (LNAPLs and DNAPLs, respectively).

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

The objective of this Technical Guidance Instruction (TGI) is to describe procedures to measure and record water-levels (groundwater and surface-water) using manual water-level meters. Water levels may be measured using an electronic water-level probe or an oil-water level indicator from established reference points (e.g., top of casing). Reference points must be surveyed to evaluate fluid level elevations relative to a vertical datum (e.g., North America Vertical Datum of 1988 [NAVD88] relative to sea level). This TGI also describes procedures for measuring in well thickness of NAPL and DNAPLs.

Surface water-levels can be measured from stilling wells or fixed points (bridges, walls, etc.) and measuring from an established point of reference using a water-level meter. In some cases, surface water water-levels may be determined from a graduated stream gauge, attached to a pole located in open water with known elevation, without the use of a water-level meter.

The use of pressure transducers or other automated devices for the collection of groundwater elevation data will be subject of *TGI – Water-Level Monitoring using Pressure Transducers and TGI – Water-Level Measurements using Sonic Meters*.

4 Personnel Qualifications

Arcadis field sampling personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as specified in the Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. In addition, Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. The HASP and other documents will identify other training requirements or access control requirements.

5 Equipment List

The following field equipment is suggested for water-level measurements:

- Site-specific Health and Safety Plan (HASP).
- Appropriate personal protective equipment (PPE) as specified in the HASP.
- Electronic water-level indicator graduated in 0.01 ft. increments.
- Electronic oil-water (interface) level indicator graduated in 0.01 ft. increments, if necessary.
- Non-phosphate laboratory soap (Alconox or equivalent), brushes, clean buckets or clean wash tubs.
- Distilled or de-ionized (required for some sites) water for equipment decontamination.
- Photoionization detector (PID) and/or organic vapor analyzer (optional).
- 150-foot measuring tape (or sufficient length for the maximum site depth requirement) – if required for total depth measurements of deeper wells.
- Solvent (methanol/acetone/isopropyl alcohol) rinse – optional.
- Spray bottle for solvent - optional
- Plastic drop cloth (e.g. Weatherall Visqueen) to place beneath the buckets or tubs to reduce potential for contamination of the tape or probe.
- Tools and/or keys required for opening wells.
- Well construction summary table and/or well construction logs.
- Summary table of previous water-level measurements.
- Field notebook and/or smart device (phone or tablet) or appropriate field forms (see Attachment 1).
- Indelible ink pen

6 Cautions

Electronic water-level indicators and oil-water interface probes may sometimes produce false-positive readings. For example, if the inside casing surface of the well or stilling tube has condensation above the water level, then an electronic water-level probe may produce a signal by contacting the sidewall of the well, rather than the true water-level surface. For accuracy, the electronic water-level probe and/or interface probe should be raised and lowered several times at the approximate depth where the instrument produces a tone indicating a fluid interface to verify consistent, repeatable results (three or more times). Additionally, some wells may be constructed with a sump. If local/regional groundwater levels have declined such that the water-level is below the base of the well screen, a sump may still contain water and provide an erroneous measurement. Therefore, possessing and comparing measurements with a well construction summary table or well construction log is recommended for proper reporting.

If the presence of a NAPL is known or suspected within specific wells, do not use an electronic water-level indicator. Use an oil-water interface probe instead. If NAPL presents ignition or explosion hazards, an intrinsically safe oil-water interface probe is required to be used with grounding and following the manufacturer's instructions.

If the NAPL is known to be very viscous or problematic to gauge, the data quality will require additional consideration prior to measuring. Staff will consider the data quality objectives for the gauging activity – e.g., if quantifying NAPL thickness is necessary, or if assessing the presence/absence is sufficient.

Alternate NAPL measurement methods (such as using drop pipes or temporary coatings for down-well equipment) may be considered.

When measuring total well depths with an electronic water-level indicator, the measurement must have a correction factor applied for post processing or completed at the time of measurement that is equal to the length of the probe beneath the circuit closing electrodes (if applicable to the instrument). This is necessary because the tape distance markings are referenced to the electrode, rather than the end of the probe. Some newer instruments do not have an offset electrode and this correction factor is needed. In addition, total depth measurements are difficult with wells that have large water columns due to buoyancy issues. In addition, the total depth measurement will include notes that indicate a soft or hard bottom if recognized during the measurement.

Ensure that the type of electronic water-level indicator is compatible with the depth and diameter of the wells to be measured. Some smaller piezometers or larger diameter well stilling tubes will accommodate only smaller diameter probes.

7 Health and Safety Considerations

The HASP will be followed, as appropriate, to ensure the safety of field personnel. Access to wells may expose field personnel to hazardous materials such as contaminated groundwater or oil. Other potential hazards include pressurized wells, stinging insects that may inhabit well heads, other biologic hazards (e.g. ticks in long grass/weeds around well head), and potentially the use of sharp cutting tools (scissors, knife). Appropriate personal protective equipment (PPE) will be worn during these activities. Only use non-toxic peppermint oil spray for stinging insect nests. Open well caps slowly and keep face and body away to allow to vent any built-up pressure. Field personnel will thoroughly review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives.

Obtaining measurements from active pumping wells requires knowledge of the construction and design, as the indicator probe and tape can become intertwined within down-well equipment (such as pump impellers) causing a serious health and safety hazard and equipment damage. Ensure that stilling wells have a perforated end and capped bottom to inhibit tape from extending into the downhole pump depth. If a stilling tube is not present or the still tube construction is not known, determine a conservative “not to exceed” measurement depth based on the top of pump depth with an added safety factor. If all information is not known, a water-level will not be taken from the pumping well until clarification on depths are available.

8 Procedure

Calibration procedures and groundwater level measurement procedures for electronic water-level indicators and oil-water indicators are described in the sections below. Calibration documentation can be requested from the rental or manufacturer.

Calibration Procedures

If the indicator requires length and markings verification is required by project data quality plan or other reasons, then the following steps may be used:

- Measure the lengths between each increment marker on the indicator with a measuring tape. The appropriate length of indicator measuring tape, suitable to cover the depth range for the wells of interest, will be checked for accuracy.
- If the indicator measuring tape is inaccurate, the probe will require to be sent back to the manufacturer or rental company. If a replacement can't immediately be available, then an offset can be measured to correct the measurements.
- If multiple water-level indicators and/or oil-water interface probes are being used for an event, calibration of the multiple devices will be required by measuring a water-level at a single well contemporaneously with all indicators to be used and calculated correction factors provided for data processing (typical corrections are small and range from 0.01 to 0.03 foot).
- Equipment calibration will be recorded in the field logbook and/or smart device.

Water-Level Measurement Procedures

The general procedures to be followed for the collection of fluid level measurements and well depths from the monitoring wells are as follows:

- Check that the water-level/oil-water level indicator battery is functional, before mobilization and prior to each work day (e.g., turn power on and check that meter sounds when probe is lowered into a bucket of water – note that water-level meters will not work with low-electrical-conductivity liquids such as distilled water).
- Record instrument make, model, serial number, and (if present) Arcadis ID number in the field form or electronic field form.
- Don disposable nitrile gloves. Decontaminate the water-level/oil-water indicator, any attached tape and the spool with laboratory-grade soap and distilled water (see Initial Decontamination Procedures below). The spool requires caution with cleaning as it is not water-proof and can be damaged during cleaning.

- The top of the monitoring well will be cleaned with a clean rag to prevent loose particulate matter from falling into the well.
- Perform a well inspection (note that a well inspection form may be required to be filled out along with a photo to document the conditions).
- Place clean plastic sheeting on the ground next to the well.
- Unlock and/or open the monitoring well cover while standing upwind from the well (note that some wells may be under pressure and precaution should be taken with opening well caps – see Section 6).
- Measure the volatile organics present in the monitoring well head space with a PID and record the PID reading (if applicable and requirement for the site).
- Allow the water-level in the well to equilibrate with atmospheric pressure for a few minutes (check previous field forms or field books for equilibration time, if noted).
- Locate the measuring reference point that correlates to the survey point on the well casing. If one is not found, make a reference point by notching the highest and/or north point on the inner casing (or outer if an inner casing is not present) or mark with a permanent mark. All downhole measurements will be taken from the reference point. Document any changes or new reference point addition.
- Measure to the nearest 0.01 foot and record the height of the inner well casing and outer protective casing to ground level (note that some well pads are raised and are not at true ground surface).
- Lower the indicator probe into the center of the well until contact with the water surface is indicated by either an audible alarm or light. The sensitivity of the probe may need adjustment if the alarm or light is not strong signal. Use and install a tape guide (available from some manufacturers) to help with accuracy and provide protection with damaging the measurement tape. If a tape guide is not available, make sure that the tape does not rub on the inner or outer casing which could fray and damage the tape.
- If an oil-water interface probe is being used to measure depth and thickness of NAPL, lower the interface probe into the center of the well until a contact with the NAPL surface is indicated by either audible alarm or light. The sensitivity of the probe may need adjustment if the alarm or light is not strong signal. To gauge the water level in a well which contains LNAPL (LNAPL-water interface), advance the interface probe past the LNAPL-water interface until the probe produces a solid audible alarm indicating water. While slowly retrieving the probe upward, the equipment will produce a different tone when the LNAPL-water interface is reached (typically this is a multiple alarm sound or flashing light). This level should represent the depth to water. The depth indicating the bottom of the water column and top of DNAPL layer, if any, is indicated by the multiple alarm signal or flashing light emitted by the interface probe.
- Hold the tape at the measuring point and repeat the measurement two more times.
- Read and record measurement to the nearest 0.01 foot. Check the measurement with previous measurements, if available, and note any anomalies/discrepancies; if significant, contact the project staff.

- Measure and record total depth of well (see Total Depth Measurement Procedures below); note that measurement of total depth is not always performed at wells containing LNAPL or DNAPL, in order to reduce decontamination of the instrument and reduce potential exposure to NAPL.
- Record all measurements (with date and time collected to the nearest minute) and note any inconsistencies/anomalies and relevant observations in the field notebook and/or smart device or appropriate field forms.
- Follow decontamination procedure outlined below before measuring subsequent wells (see *Decontamination after Water Level and Total Depth Measurements* below).
- Replace cap and lock the well when all activities are completed.

Total Depth Measurement Procedures

- Weighted tape or electronic water-level indicator can be used to measure the total well depth.
- Follow initial procedures noted above in Water-Level Measurements above.
- Lower indicator probe (or tape) until weighted end is resting on the bottom of the well. Raise indicator slowly until there is no slack in the tape. Gently estimate the bottom of the well by slowly raising and lowering the indicator: great care should be taken to avoid damaging the sensor on the probe. The operator may find it easier to allow the weight to touch bottom and then detect the 'tug' on the tape while lifting the weight off the well bottom.
- Because of tape buoyancy and weight effects encountered in deep wells with long water columns, it may be difficult to determine when the probe is in contact the bottom of the well and sediment in the bottom of the well can also make it difficult to determine total depth. Care must be taken in these situations to ensure accurate measurements.
- If total depth measurements are to be collected during low-flow sampling events, the measurement will be made only after low-flow sampling has been completed or at least 12 hours prior to initiating sample collection from the well, in order to minimize: 1) mixing of the stagnant water at the top of the well column with potential formation water underneath; and/or 2) agitation and subsequent entrainment of possible sediment collected at the well bottom).
- Read and record measurement to the nearest 0.1 foot. Please refer to the note regarding total depth measurements described in Section 5 Cautions above.
- Follow decontamination procedure outlined below before gauging the next well (see *Decontamination after Water Level, NAPL Level, and Total Depth Measurements* below).

Initial Decontamination

- Note that there may be project specific decontamination procedure documents that will be followed in lieu of the below procedures.
- Set up a decontamination station consisting of three clean buckets (e.g., 5-gallon buckets). The buckets should not be used to containerize purge water; they will be used for decontamination purposes only.
- Fill the first bucket with one gallon of distilled water (use deionized water if metals are a contaminant at the site) and add non-phosphate laboratory-grade soap. Fill the second bucket

with distilled water (use deionized water if metals are a contaminant at the site) and leave the third bucket empty. Place the drop cloth underneath.

- Unwind the entire tape from the spool into a bucket with non-phosphate laboratory-grade soap and distilled water; Brush the tape carefully to remove dirt and possible contamination, using a brush dedicated to the wash bucket.
- Carefully brush all dirt of the spool and wipe down with a soapy cloth or paper towel.
- Transfer the tape into the second bucket containing rinse water. Carefully brush the tape using a second brush, dedicated to the rinse bucket. Lift the tape out of the bucket and allow rinse water to drip off the tape.
- Transfer the tape to the third bucket. Wind the tape onto the spool while wiping excess water off the tape using a paper towel.

Decontamination after Water Level, NAPL Level, and Total Depth Measurements

- Set up a decontamination station consisting of three clean buckets, fill according to the initial decon procedure.
- Unwind the only the length of tape used for gauging from the spool into a bucket with laboratory-grade soap and distilled water. Brush the tape carefully to remove dirt and possible contamination, using a brush dedicated to the wash bucket.
- Continue as described above.
- Extra care should be taken to clean the probe after a total depth measurement. All sediment or dirt needs to be removed during decontamination.
- If an oil-water interface probe is used to gauge NAPL, a solvent may be necessary to remove all NAPL residue. After decontaminations steps above, use a spray bottle filled with chosen solvent (ex. isopropyl alcohol) and spray across all surfaces of the tape. Use paper towels to wipe off solvent and/or residue. This step may be repeated if necessary.

Notes:

- Collect equipment blanks if required by the work plan (minimum 1 per 20 samples or 1 per sampling event).
- Prepare new wash solution and rinse water when necessary (e.g., every 10 to 20 wells). The spent wash and rinse solution should be discharged according to site practices.
- The decontamination station may be expanded by adding extra rinse and/or detergent stations (i.e., solvent wash station) to the set up. The addition of more stations depends on the requirements of the work plan or the site-specific Field Sampling and Quality Assurance Plan and outlined in the project field plan or kick-off meeting.
- Small crates or washtubs are a possible substitute for the buckets. In any case, it is recommended to use containers with a lid.

9 Waste Management

Decontamination fluids, PPE, and other disposable equipment will be properly stored on site in labeled containers and disposed of properly. Be certain that waste containers are properly labeled and documented in the field log book. Review *TGI – Investigation Derived Waste Handling and Storage*, for additional information and state- or client-specific requirements.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Field personnel will complete all applicable field forms for each test (see attached forms and FieldNow® available forms). Forms will include recommended data file naming protocol per the field implementation plan. Data that has been collected must be transmitted at the end of the day (notes/forms/data file). The files must be transmitted to the appropriate project directory (via direct transfer or email to data manager). Work completed that day and any relevant observations noted during the daily activities as well as copies of the data mentioned above should be summarized and provided in an email. The appropriate team member will review the data for accuracy and provide feedback. Field equipment calibration, decontamination activities, and waste management activities will be recorded in the field notebook, appropriate field form, or daily log.

Refer to the Quality Procedure Data Management, QP 4.09, for additional information on data management.

11 Quality Assurance

Suggested quality control measures are below; project teams may implement some or all of these at their discretion and based on project data quality needs.

- As described in the detailed procedure, the electronic water-level meter and/or oil-water interface probe can be calibrated prior to use versus an engineer's rule to ensure accurate length demarcations on the tape or cable. The results will be recorded.
- Measurements will be completed three times, with the final measurement recorded.
- Fluid interface measurements will be verified by gently raising and lowering the instrument through each interface to confirm repeatable results.
- Field notes will be reviewed by the project team once the field data has been delivered.

12 References

Cunningham, W.L., and Schalk, C.W., comps., 2011. *Groundwater technical procedures of the U.S. Geological Survey: U.S. Geological Survey Techniques and Methods 1–A1*, 151 pp.

U.S. Environmental Protection Agency, 2013. *SESD Operating Procedure, Groundwater level and WellDepth Measurement*. January 29.

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TGI – General Slug Testing

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| | 1 | | | | |
| | 2 | | | | |
| | 3 | | | | |
| | 4 | March 5, 2015 | All | New TGI template | Everett Fortner Marc Killingstad |
| | 5 | April 28, 2022 | All | New TGI template; Updates to pneumatic slug testing guidance; Addition of forms | Everett Fortner Marc Killingstad |
| | 6 | December 22, 2022 | 9, 11 | Clarification on barometric pressure logger and slug | |
| | 7 | March 1, 2023 | All | Annual review completed by SME. Document Revision Number and Document Date Updated. | |

Approval Signatures

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3/1/2023

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Date

Reviewed by:



3/1/2023

Marc Killingstad, PE (Subject Matter Expert)

Date

1 Introduction

Slug testing is a common field method used for estimating hydraulic conductivity. This Technical Guidance Instruction (TGI) document outlines field procedures for conducting such testing.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

This TGI for Slug Testing is intended as general guidance for testing design that applies to all forms of slug testing. Guidance for the specific slug testing method selected based on this overall guidance is attached in four separate documents. The four detailed methods of slug testing covered include:

- **Attachment A – TGI for Solid Slug Testing**
- **Attachment B – TGI for Water Slug (Inflow) Testing**
- **Attachment C – TGI for Baildown Testing**
- **Attachment D – TGI for Pneumatic Slug Testing**

Each of these attachments will be used in conjunction with this general guidance. For more information or questions, please contact the Technical Knowledge and Innovation (TKI) technical lead for Aquifer Testing and Characterization.

The objective of this TGI is to establish uniform procedures for slug testing to estimate the hydraulic conductivity of the groundwater zone near a well. A slug test is completed by “instantaneously” inducing a change in hydraulic head and measuring the rate of the groundwater return to equilibrium (static) conditions. This guidance document provides detailed information on test methodology, planning, and application. Field forms and procedures for conducting tests using solid slugs, inflow (water slug), baildown, and pneumatic testing methods are attached. Also attached is a parts list and as-built drawing for the pneumatic testing manifold.

Please note that the data processing and analysis of the slug testing is not covered in this guidance.

It is strongly advised that, prior to conducting any aquifer testing, the project team contact the TKI Aquifer Testing and Characterization Focus Group to receive assistance with the site-specific technical guidance regarding the design, execution, training, costing, and analysis phases of the proposed testing.

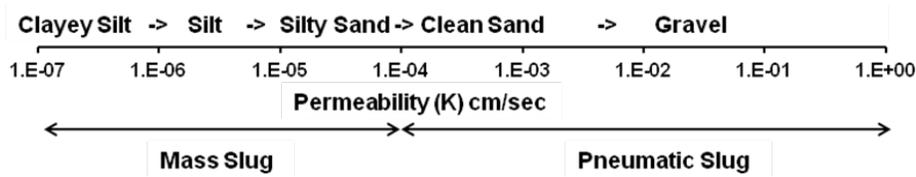
Slug tests are used as an economic, simple, and rapid way to obtain data needed to estimate near-well hydraulic conductivity (Butler 2019). The advantages of slug testing over other testing methods (e.g., pumping tests) is that they are relatively easy and inexpensive to implement and generate little to no investigation-derived waste. These advantages allow for several tests to be completed at a site over a relatively short period of time, which provides a better understanding of the spatial distribution of hydraulic conductivity both horizontally and vertically across the groundwater zone(s) (i.e., aquifer heterogeneity). However, slug tests will not be viewed as a replacement for the larger scale estimates of hydraulic conductivity and additional hydraulic parameters such as storativity derived from multi-well pumping tests (Kruseman and de Ridder 1994). The shorter time frame and limited stress on the groundwater zone from slug tests provides a smaller scale (near-well) hydraulic conductivity estimate than pumping tests. Due to this localized scale of slug testing, the effects of the well filter pack, well development, and well skin are more significant than during pumping tests and have the potential to limit water-level change during testing. Wells that are insufficiently developed can adversely affect the results of the slug test and generally lead to a lower hydraulic conductivity estimate than other conventional methods. Therefore, careful consideration will be given prior testing wells that are expected to have skin damage and where the development history is unknown. Varying the direction and magnitude of the stress and inducing a relatively large, measurable head change can facilitate determination of skin effects and increase the probability that the selected test response is a result of groundwater zone hydraulics and not the well construction hydraulics (Butler 2019).

Slug Test Design

The initial step during the planning stage will consist of determining whether or not slug tests will provide adequate aquifer characterization to meet the specific project objectives. For example, slug testing may be useful in mapping relative changes in hydraulic conductivity (heterogeneities) or used to calibrate high resolution hydraulic testing results provided by direct-push tools. However, slug testing is not an appropriate testing method to obtain aquifer storage estimates. In addition, slug testing may not be appropriate for testing of bedrock, particularly when there is incomplete understanding of the fracture network. A pumping test, packer test, or open borehole test may be more appropriate in bedrock. However, weathered bedrock zones can often be considered hydraulically equivalent to a porous medium and, therefore, slug testing may be appropriate.

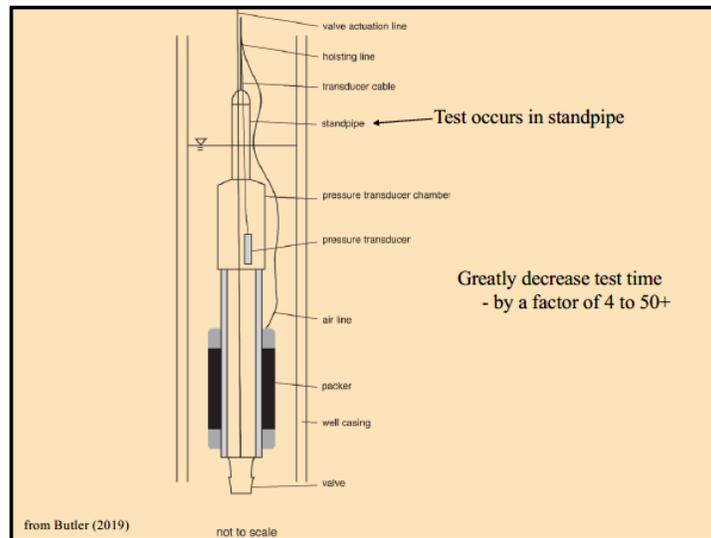
The design phase includes several elements as outlined below. Please note, there may be exceptions, as design specifications need to be tailored to the conceptual site model (CSM) and project objectives.

1. Review of the CSM and site data. This will include a review of boring logs (key elements – lithology and drilling method utilized), well construction details, well development logs, cross-sections, constituent plume information, hydrostratigraphic zones, previous estimates of hydraulic conductivity, and regional estimates from published sources. Special attention will be given to well screen intervals in order to ensure that the tests are conducted in the targeted groundwater zone. If a well is screened across multiple hydrostratigraphic zones, uncertainty associated with estimated hydraulic conductivity values for a given zone is introduced. However, there are exceptions if the permeability of the zones that are crossed by the well screen differ significantly.
2. Review of regulatory program rules or guidance to ensure proper compliance with the criteria outlined in the regulatory documents. In most cases, slug testing using pneumatic or solid slug methods will not require permitting; however, use of in-flow procedures, although unexpected, may require a permit (e.g., Underground Injection Control [UIC] permit).
3. Compile detailed information for each proposed test well:
 - a. Well construction information
 - i. Well casing/screen diameters and lengths
 - ii. Screen type (material and slot design) and slot size
 - iii. Total depth – reviewing the measurement of the test well total depth aids in development assessment. If the total depth has decreased substantially since installation, sediment buildup may be occurring and redevelopment will be completed before testing
 - iv. Filter pack construction (thickness and material)
 - b. Historical and current groundwater levels – this will aid in the determination of the appropriate slug test type (e.g., is the screen submerged)
 - c. Well development record – testing improperly developed wells will result in anomalous results
 - d. Recent well sampling logs – historic sampling data will provide information on the potential yield of the well and groundwater quality (turbidity)
4. Determine appropriate testing method(s): In light of the information researched in items 1 through 3, slug test method(s) will be selected based on the groundwater zone information and data needs for proper analysis:
 - a. **Solid Slug** – used for rising and falling head tests in wells of adequate water column length with fully or partially submerged screens. If screen is partially submerged (i.e., intersects the groundwater table), then a rising head test is the most appropriate test method. Adequate length of water column would allow for placement of transducer at least 1 foot below the anticipated submersion depth of the slug or length of water column to accommodate the just the slug when using manual measurements.
 - b. **Inflow** – used for falling head tests where the water column is too small to accommodate typical slug test equipment or applied as part of development procedures.
 - c. **Baildown** – used for rising head tests especially in cases with partially submerged well screens.
 - d. **Pneumatic** – used for rising head tests with fully submerged well screens. The well is pressurized to depress the water-level and then allowed to equilibrate. The recovery to static conditions is measured after the pressure is released.
 - e. **Groundwater Zone Information** – The initial hypotheses of the groundwater zone characteristics and permeability based on either previous testing, qualitative information (e.g., boring logs and/or grain size analysis), or other studies in the area is also an important consideration when determining the testing methodology. The chart below depicts of test type to be applied in relation to permeability for guidance.



Note: For instance, if boring logs indicate the groundwater zone is predominantly sand and/or gravel and groundwater sampling logs indicate good well yield, then a hypothesis may be made that the formation may have a relatively high permeability. Therefore, if the well screens are fully submerged, pneumatic slug testing would be appropriate.

- f. **Low Permeability** – While pneumatic slug testing may be appropriate for hydrogeologic settings where high hydraulic conductivity is expected, other settings may have very low hydraulic conductivity where recovery may take multiple hours or days. For these types of conditions, a shut-in test can be conducted using a packer and standpipe apparatus to decrease the effective casing radius of an existing well and in turn, reduce the test time. An example of this apparatus is shown below (Butler 2019). Currently, this device type is not available for rental/purchase from known vendors. If this method is needed for a site with expected low hydraulic conductivity, the additional cost for the development and construction of the apparatus will need to be considered.



- g. **Additional Data** – Additional data may be required based on the site setting in order to determine the most appropriate test method (Butler 2019) and to understand external influences on the test. This includes duplicate testing, rising and/or falling head tests, multiple tests with varying initial displacements, and background data to assess background water level trends. Generally, three tests per well is recommended with two duplicate tests at the same displacement and one with double the original displacement. Special cases with expected external influences that induce water level changes (tidal, atmospheric or pumping) may require determination of pre-test water level trends. Arrange to have nearby active pumping wells shut down for at least 48 hours prior to testing or constant rate

extraction during testing, if applicable and possible. Finally, testing will not be completed during periods of significant precipitation.

- h. **Data Acquisition** – To ensure high resolution data acquisition, data-logging pressure transducers are to be used to collect water level data (see TGI – Water-Level Monitoring Using Data Logging Instruments). Change in head measurements are required to be at high resolution at a frequency of 0.5 second or less for high permeability groundwater zones (e.g., gravels and sands). Newer instrumentation has improved memory space; and internal data processing tools are built to maintain the early high frequency data and reduce the later time data. The high frequency is also recommended for lower permeability groundwater zones (e.g., fine sands, silts, and clays) at the start of the tests, then later recovery data (after 1 hour) over longer periods of time can be recorded at a lower frequency by resetting the pressure transducer. Ideally, frequencies should be in the seconds range depending on the permeability and the length of the test. To optimize data collection, the pressure transducer will need to be set to linear data collection, real-time viewing of the results will help with assuring testing quality. Manual water-level measurements are required to also be collected to verify and back-up the electronic data collected using pressure transducers. For low permeability formations with fully submerged well screens, manual data acquisition may be appropriate (i.e., where groundwater recovery occurs at a reduced rate at a magnitude [change in water level of 0.05 feet in 10 to 30 seconds] such that frequent data collection is not required).

4 Personnel Qualifications

Field personnel performing the testing are required to have the following qualifications:

- Sufficient “hands-on” experience necessary to successfully complete the slug test field work. Training requirements for conducting slug tests including the review of this guidance and other applicable documents related to instrumentation.
- Demonstrate familiarity with the electronic data logging equipment (see *TGI – Water-Level Monitoring Using Data Logging Instruments*).
- Completed current health and safety training in accordance with the project health and safety plan (e.g., 40-hour Hazardous Waste Operations training and site-specific training, as appropriate).

5 Equipment List

Refer to the four individual detailed methods of slug testing attachments (Attachments A-D) for specific method equipment lists.

6 Cautions

- Pressure Transducers/Data Loggers (see TGI – Water-Level Monitoring Using Data Logging Instruments)
 - Ensure that all rental instruments and tapes have been calibrated and checked prior to use.
 - Small-diameter pressure transducers (typically 0.5 to 0.75 in) are available that cover a range of pressures. Install the pressure transducer at a reasonable distance below (approximately 3 to 5 feet) the targeted drawdown estimated for the well to prevent noise. Do not install the pressure

transducer closer than 6 inches from the base of the well to eliminate the possibility of fouling the transducer with material accumulated at the bottom of the well. To prevent pressure transducer malfunction or damage, do not submerge pressure transducers in excess of the operating range, and do not insert objects in the sensor opening.

- For vented pressure transducers/data loggers, test functionality with a field test of readings using a bucket or barrel filled with water. Submerge the pressure transducer, accurately measure the water head above the pressure transducer sensor and compare the measurement to the reading. If the measurements don't generally agree, there may be an issue with the instrument.
 - Non-vented transducers, which record a combined pressure of barometric and the water column above the pressure transducer, can be tested in the same fashion as the vented pressure transducer (outlined above). The water column above the pressure transducer can be checked by subtracting out current atmospheric pressure.
 - In general, when testing the pressure transducers, check the pressure transducer general response to changing heads by raising the pressure transducer a certain distance, observing the change in head, and then measuring the distance manually. Additionally, water level meters will be in good working condition and calibrated to true depth and ensuring that there are no breaks or splices in the cable.
 - Pressure transducers will be set in the well at least 15 minutes prior to testing to allow to the instrument to thermally equilibrate with groundwater, collect static water-level measurements, and ensure that the pressure transducer cable will not stretch during testing.
 - Logarithmic or head-change settings will not be used to log data, only linear.
 - Prior to testing, secure pressure transducer cables at the wellhead to prevent movement that may affect measurements. Mark a reference point, such as masking tape, on the down-hole transducer cable or securing line and check regularly to detect slippage.
 - A barometric pressure transducer will be utilized, regardless of in-well pressure transducer type (vented or non-vented) if a slug test is expected to take more than four hours for full recovery. Barometric trends in long-term data sets may need to be evaluated or removed from the data set.
- Data Management
 - Data management is critical to prevent any loss. Use caution not to overwrite any previously recorded files and remember, data backup is always necessary. Multiple tests at the same well do not require for the pressure transducer to be reset and the same log can run throughout the duration of all tests. Upload data collected via smart device by email or cloud server to reduce the risk of data loss (e.g., computer failure).
 - Slug Volume
 - Solid slugs will be calibrated to determine their accurate volume(s) for theoretical displacement. In most cases, rental slugs offer economic and data quality benefits over field-built slugs.
 - When completing baildown or inflow testing, purge or injected volume will need to be measured accurately using a graduated cylinder.
 - The length and diameter of the bailer or solid slug will be measured in the field and noted in the field logs to accurately estimate theoretical displacement.

- Initial Displacement and Recovery
 - When performing slug tests, the general rule of thumb for initial displacement is between 1 and 3 feet and/or generally less than 25% of the effective screen length. For high conductivity formations, initial displacements will be small (0.3 – 0.7 ft) to avoid remobilizing fines and to limit turbulence.
 - Water levels will need to be recorded to within 80% to 95% recovery. In addition, duplicate tests will be completed only after the first test has recovered by at least 95%.
- Equipment Care
 - Keep sensitive electronic equipment away from devices that generate significant magnetic fields. For example, do not place pressure transducers near electric power generators or electric pump motors. Likewise, radio signals may cause pressure transducers or computers to malfunction.
- Decontamination
 - Make sure all equipment that enters the test well (slug, water-level meter, pressure transducer) is decontaminated before use. If testing multiple wells, start with the least contaminated and progress to the most contaminated.
- Non-Aqueous Phase Liquids (NAPL)
 - Slug tests are not recommended in wells where Non-Aqueous Phase Liquids are present. Consult TKI Aquifer Testing and Characterization Focus Group lead for guidance.

7 Health and Safety Considerations

The site-specific HASP will be used to ensure that the tests are conducted in a safe manner and will include a Job Safety Analysis (JSA). The following specific health and safety issues will be considered when conducting slug tests:

- Appropriate PPE with minimum of Level D will be worn to avoid contact with site chemicals of concern during slug test.
- Well covers will be carefully removed to avoid potential contact with insects or animals. Well caps will be vented or tethered to avoid potential eye injury in case of gas buildup in the well.
- Pressurization or vacuum hazards associated with pneumatic slug testing will be considered during test planning and implementation.

8 Procedure

Refer to the four individual detailed methods of slug testing attachments (Attachments A-D) for specific procedures.

9 Waste Management

Rinse water, PPE, and other waste materials generated during equipment decontamination will be placed in appropriate containers and labeled. Containerized waste will be disposed of, consistent with appropriate waste management procedures for investigation-derived waste.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Field personnel will complete a Slug Test Field Log form for each test. The digital field form is available through the FieldNow® Application titled FieldNow – Slug Test Field Log. A hardcopy example is included in the attachments. As previously noted, it is generally recommended to conduct three tests per well (the original displacement, a duplicate, and double original displacement); therefore, one field log will be completed for each test. Multiple tests can be entered into the FieldNow® digital application for each well tested. Field equipment calibration, decontamination activities, and waste management activities will be recorded in the daily field log. Data that has been collected must be transmitted at the end of the day (notes/forms/data file). The files must be transmitted to the appropriate project file storage at the end of each day (via direct transfer or email to data manager). Work completed that day and any relevant observations noted during the daily activities as well as copies of the data mentioned above should be summarized and provided in an email. The appropriate team member will review the data for accuracy and provide feedback.

11 Quality Assurance

Review data collected during field testing to determine reasonableness/quality given site-specific conditions. Again, this can also be completed using the transducer in real-time viewing mode as the test progresses and resulting charts to confirm with project hydrogeologist. Compare the theoretical head displacement calculated from the slug volume or pressure to the observed displacement. If the data are questionable, the field equipment will need to be checked to confirm proper working order and the test may be repeated, if possible. Consult with the project hydrogeologist to work through issues encountered in the field and to help determine test validity.

Any issues that may affect the data will be recorded in the daily field log for consideration by the project hydrogeologist.

12 References

- Butler, J.J., Jr., 2019. The Design, Performance, and Analysis of Slug Tests (2nd Ed), CRC Press, Boca Raton 280p.
- Butler, J.J. Jr., 2020. Slug Test Strategies for Challenging Conditions. Presentation – Midwest Geosciences Webinar. December.
- Kruseman, G.P. and N.A. de Ridder, 1994. Analysis and Evaluation of Pumping Test Data (2nd ed.), Publication 47, Intern. Inst. for Land Reclamation and Improvement, Wageningen, The Netherlands, 370p.

Attachment A

TGI for Solid Slug Testing

TGI – Solid Slug Testing

Rev: 7

Rev Date: March 1, 2023

Version Control

| Issue | Revision No. | Date Issued | Page No. | Description | Reviewed By |
|-------|--------------|----------------|----------|--|-------------------------------------|
| | 1 | | | | |
| | 2 | | | | |
| | 3 | | | | |
| | 4 | March 5, 2015 | All | New TGI template | Everett Fortner Marc Killingstad |
| | 5 | April 28, 2022 | All | New TGI template; Updates to pneumatic slug testing guidance; Addition of forms | Everett Fortner Marc Killingstad |
| | 6 | March 1, 2023 | 6 | Clarification on barometric pressure logger and slug | |
| | 7 | March 1, 2022 | All | Annual review completed by SME. Document Revision Number and Document Date Updated. | |

Approval Signatures

Prepared by:



3/1/2023

Everett H. Fortner III, PG (Preparer)

Date

Reviewed by:



3/1/2023

Marc Killingstad, PE (Subject Matter Expert)

Date

1 Introduction

Slug testing is a common field method used for estimating hydraulic conductivity. This Technical Guidance Instruction (TGI) document outlines field procedures for conducting such testing using a solid slug.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

The use of a solid slug allows for both falling- and rising-head slug tests to be completed. Solid slug(s) of a known volume are inserted and removed from the water column in a well in a near-instantaneous manner. The water level response is observed using a data-logging pressure transducer with manual measurement backup or using just a manual water level meter for slow recovering wells with fully submerged screens.

4 Personnel Qualifications

Field personnel performing the testing are required to have the following qualifications:

- Sufficient “hands-on” experience necessary to successfully complete the slug test field work. Training requirements for conducting slug tests including the review of this guidance and other applicable documents related to instrumentation.
- Demonstrate familiarity with the electronic data logging equipment (see *TGI – Water-Level Monitoring Using Data Logging Instruments*).
- Completed current health and safety training in accordance with the project health and safety plan (e.g., 40-hour Hazardous Waste Operations training and site-specific training, as appropriate)

5 Equipment List

The following materials will be available, as required, during slug testing using a solid slug:

- Job safety analysis and site Health and Safety Plan
- Related project-specific requirements and plans
- Personal protective equipment, as required by the site Health and Safety Plan
- Solid slug(s) of known volume
- Pressure transducer and barometric pressure logger (if necessary)
- Pressure transducer software
- Laptop computer, smart device (phone or tablet), and/or data transfer device
- Rope or cables (for deep wells) (chemical resistant, low stretch [stainless steel or Kevlar] is optimal)
- Water level meter
- Measuring tape
- Spring-loaded clamps and zip ties
- Decontamination equipment
- Slug test field form (paper or digital)
- Field tablet and/or daily logs
- Waterproof marker

6 Cautions

- Pressure Transducers/Data Loggers (see *TGI – Water-Level Monitoring Using Data Logging Instruments*)
 - Ensure that all rental instruments and tapes have been calibrated and checked prior to use.
 - Small-diameter pressure transducers (typically 0.5 to 0.75 in) are available that cover a range of pressures. Install the pressure transducer at a reasonable distance below (approximately 3 to 5 feet) the targeted drawdown estimated for the well to prevent noise. Do not install the pressure transducer closer than 6 inches from the base of the well to eliminate the possibility of fouling the transducer with material accumulated at the bottom of the well. To prevent pressure transducer malfunction or damage, do not submerge pressure transducers in excess of the operating range, and do not insert objects in the sensor opening.

- For vented pressure transducers/data loggers, test functionality with a field test of readings using a bucket or barrel filled with water. Submerge the pressure transducer, accurately measure the water head above the pressure transducer sensor and compare the measurement to the reading. If the measurements don't generally agree, there may be an issue with the instrument.
- Non-vented transducers, which record a combined pressure of barometric and the water column above the pressure transducer, can be tested in the same fashion as the vented pressure transducer (outlined above). The water column above the pressure transducer can be checked by subtracting out current atmospheric pressure.
- In general, when testing the pressure transducers, check the pressure transducer general response to changing heads by raising the pressure transducer a certain distance, observing the change in head, and then measuring the distance manually. Additionally, water level meters will be in good working condition and calibrated to true depth and ensuring that there are no breaks or splices in the cable.
- Pressure transducers will be set in the well at least 15 minutes prior to testing to allow to the instrument to thermally equilibrate with groundwater, collect static water-level measurements, and ensure that the pressure transducer cable will not stretch during testing.
- Logarithmic or head-change settings will not be used to log data, only linear.
- Prior to testing, secure pressure transducer cables at the wellhead to prevent movement that may affect measurements. Mark a reference point, such as masking tape, on the down-hole transducer cable or securing line and check regularly to detect slippage.
- A barometric pressure transducer will be utilized, regardless of in-well pressure transducer type (vented or non-vented) if a slug test is expected to take more than four hours for full recovery. Barometric trends in long-term data sets may need to be evaluated or removed from the data set.
- Data Management
 - Data management is critical to prevent any loss. Use caution not to overwrite any previously recorded files and remember, data backup is always necessary. Multiple tests at the same well do not require for the pressure transducer to be reset and the same log can run throughout the duration of all tests. Upload data collected via smart device by email or cloud server to reduce the risk of data loss (e.g., computer failure).
- Slug Volume
 - Solid slugs will be calibrated to determine their accurate volume(s) for theoretical displacement. In most cases, rental slugs offer economic and data quality benefits over field-built slugs. The length and diameter of the solid slug will be measured in the field and noted in the field logs to accurately estimate theoretical displacement.
- Initial Displacement and Recovery
 - When performing slug tests, the general rule of thumb for initial displacement is between 1 and 3 feet and/or generally less than 25% of the effective screen length. For high conductivity formations, initial displacements will be small (0.3 – 0.7 ft) to avoid remobilizing fines and to limit turbulence.
 - Water levels will need to be recorded to within 80% to 95% recovery. In addition, duplicate tests will be completed only after the first test has recovered by at least 95%.

- Equipment Care
 - Keep sensitive electronic equipment away from devices that generate significant magnetic fields. For example, do not place pressure transducers near electric power generators or electric pump motors. Likewise, radio signals may cause pressure transducers or computers to malfunction.
- Decontamination
 - Make sure all equipment that enters the test well (slug, water-level meter, pressure transducer) is decontaminated before use. If testing multiple wells, start with the least contaminated and progress to the most contaminated.
- Non-Aqueous Phase Liquids (NAPL)
 - Slug tests are not recommended in wells where Non-Aqueous Phase Liquids are present. Consult TKI Aquifer Testing and Characterization Focus Group lead for guidance.

7 Health and Safety Considerations

The site-specific HASP will be used to ensure that the tests are conducted in a safe manner and will include a Job Safety Analysis (JSA). The following specific health and safety issues will be considered when conducting slug tests:

- Appropriate PPE with minimum of Level D will be worn to avoid contact with site chemicals of concern during slug test.
- Well covers will be carefully removed to avoid potential contact with insects or animals. Well caps will be vented or tethered to avoid potential eye injury in case of gas buildup in the well.

8 Procedure

1. Decontaminate all down-well equipment: pressure transducer and cable, slug(s), rope or cable, water level meter in accordance with project specific requirements. In general, wells will be tested from least contaminated to more contaminated, if possible or applicable.
2. Select a solid slug according to a target initial displacement using the table below. A general guideline is that initial displacements are between 1 and 3 feet, but will depend on the anticipated response (i.e., smaller initial displacements will be chosen for formations with high hydraulic conductivity). When utilizing the FieldNow® – Slug Test Field Log, the bailer size and length and the well diameter can be entered, and a theoretical displacement will be automatically calculated.
3. Measure depth to water and well total depth. Determine the water column length.
 - a. Multiple depth to waters will be measured and any trends will be noted.
 - b. The "static" depth to water will be representative of the water level after the well equalizes with atmosphere.
4. Review the well construction log to determine screened interval and confirm depth to bottom. If discrepancies exist, consult with project hydrogeologist.
5. Program the pressure transducer to record water levels at the following suggested frequencies. Note that the lithologic descriptions and datalogger memory will be used to select the highest measurement frequency possible.

- a. In hydrogeologic settings where high hydraulic conductivity is expected, water levels will be measured at 0.5-second intervals or the highest frequency available. This measurement frequency will be selected for gravels and sands.
- b. In hydrogeologic settings where low hydraulic conductivity is expected, water levels may be measured at 1-second intervals. This measurement frequency will be selected for silts and clays.
6. If applicable, program the barometric pressure logger to record barometric pressure. The logger will be placed in the headspace of an adjacent well, or on grade, adjacent to the well being tested and kept protected from the elements (e.g., rain or sun)
7. Install the pressure transducer deep enough within the water column to not interfere with the testing equipment. Ideally the transducer will be 3 to 5 feet lower than the maximum depth of the slug testing equipment not closer than 6 inches above the well bottom. Remember to use measurements and not the well bottom as silt can clog the pressure transducer sensor. Secure the pressure transducer cable to the well casing or other static object.
8. View the measured water level in real time. Wait for the water levels to stabilize. Note that the temperature of the pressure transducer will require to equilibrate to groundwater temperatures to ensure accurate water-level measurements (at least 15 minutes).
9. After static measurements and equilibration (15 minutes), re-measure the depth to water.
10. Measure the slug and rope assembly length and mark the rope at a length as follows:
 - Rope Mark #1 = Depth to Potentiometric Surface from TOC
 - Rope Mark #2 = Depth to Potentiometric Surface from TOC + Length of Slug + Safety Factor (Safety Factor = 10% of the Length of Slug)When deployed, Rope Mark #2 will be at the well top of casing, and the slug will be totally submerged. If insufficient water column is available to cover the slug assembly top, note the theoretical length of the slug to be inserted into the water column. Upon removal, measure the wet slug length.
11. Slowly insert the slug assembly into the well and stop just above the potentiometric surface Rope Mark #1.
12. With slack in the rope and the slug being suspended above the water column, place the Rope Mark #2 at the top of casing. Clamp the non-slug end of the rope to a static object.
13. Quickly drop the slug into the water column.
14. Observe the water level response on the laptop computer and/or measure depth to water, being careful not to interfere with the pressure transducer cable. Several manual depth to water measurements will be made throughout the test (typically 2 to 3 in the first 30 seconds to 1 minute, one reading a minute for the next 5 to 10 minutes, and every 2 to 5 minutes thereafter). If the water level meter is used as the primary measurement technique, the measurement frequency will need to be increased as practicable.
15. Allow sufficient time for water level to recover to static level. If completing one test (just a falling head test or just a rising head test), then 80% recovery is sufficient. Duplicate tests are highly recommended, and the next test will be completed after the first test has recovered to greater than 95%. A third test at a displacement of twice the initial is recommended.

$$\text{Percent Recovery} = \left(\frac{\text{current displacement}}{\text{maximum displacement}} \right) * 100$$

This calculation can be used to determine the depth to water that will be needed before conducting a duplicate test. For example, if your theoretical maximum displacement is 1.92 ft and 95% recovery is needed then 1.82 feet of recovery is necessary to conduct a duplicate test which means the depth to water or be less than or equal to 0.10 feet below the static water level.

16. Quickly remove the slug assembly from the water column. The slug assembly will be left in the well above the static water level in order to limit pressure cable disturbance until the testing is complete and the water levels have equilibrated to the target level.
17. Repeat both the falling- and rising-head slug tests for data reproducibility by repeating steps 12 and 13, if applicable. If possible, complete a third test with a slug or combination of slugs that equates to twice or half the volume as the original.
18. Save all data files to the laptop and finalize any field notes.
19. Review the data collected to determine the reasonableness of the preliminary results and compare the pressure transducer results to the water level meter results. The observation of apparently anomalous results will be discussed with senior project staff prior to additional testing or leaving the field site. The water level record for each test will show static conditions, the insertion or removal of the slug(s), and the water level response. Make notes on the field form and notebook concerning any irregularities.
20. Decontaminate all down-well equipment in accordance with project plans.

9 Waste Management

Rinse water, PPE, and other waste materials generated during equipment decontamination will be placed in appropriate containers and labeled. Containerized waste will be disposed of, consistent with appropriate waste management procedures for investigation-derived waste.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Field personnel will complete a Slug Test Field Log form for each test. The digital field form is available through the FieldNow® Application titled *FieldNow – Slug Test Field Log*. A hardcopy example is included in the attachments. As previously noted, it is generally recommended to conduct three tests per well (the original displacement, a duplicate, and double original displacement); therefore, one field log will be completed for each test. Multiple tests can be entered into the FieldNow® digital application for each well tested. Field equipment calibration, decontamination activities, and waste management activities will be recorded in the daily field log. Data that has been collected must be transmitted at the end of the day (notes/forms/data file). The files must be transmitted to the appropriate project file storage at the end of each day (via direct transfer or email to data manager). Work completed that day and any relevant observations noted during the daily activities as well as copies of the data mentioned above should be summarized and provided in an email. The appropriate team member will review the data for accuracy and provide feedback.

11 Quality Assurance

Review data collected during field testing to determine reasonableness/quality given site-specific conditions. Again, this can also be completed using the transducer in real-time viewing mode as the test progresses and resulting charts to confirm with project hydrogeologist. Compare the theoretical head displacement calculated from the slug volume or pressure to the observed displacement. If the data are questionable, the field equipment will need to be checked to confirm proper working order and the test may be repeated, if possible. Consult with the project hydrogeologist to work through issues encountered in the field and to help determine test validity. Any issues that may affect the data will be recorded in the daily field log for consideration by the project hydrogeologist.

12 References

- Butler, J.J., Jr., 2019. The Design, Performance, and Analysis of Slug Tests (2nd Ed), CRC Press, Boca Raton, 280p.
- Butler, J.J. Jr., 2020. Slug Test Strategies for Challenging Conditions. Presentation – Midwest Geosciences Webinar. December.
- Kruseman, G.P. and N.A. de Ridder, 1994. Analysis and Evaluation of Pumping Test Data (2nd ed.), Publication 47, Intern. Inst. for Land Reclamation and Improvement, Wageningen, The Netherlands, 370p.

Attachment B

TGI for Water Slug (Inflow) Testing

TGI – Water Slug (Inflow) Testing

Rev: 7

Rev Date: March 1, 2023

Version Control

| Issue | Revision No. | Date Issued | Page No. | Description | Reviewed By |
|-------|--------------|-------------------|----------|--|-------------------------------------|
| | 1 | | | | |
| | 2 | | | | |
| | 3 | | | | |
| | 4 | March 5, 2015 | All | New TGI template | Everett Fortner Marc Killingstad |
| | 5 | April 28, 2022 | All | New TGI template; Updates to pneumatic slug testing guidance; Addition of forms | Everett Fortner Marc Killingstad |
| | 6 | December 22, 2022 | 6 | Clarification on barometric pressure logger and slug | |
| | 7 | March 1, 2023 | All | Annual review completed by SME. Document Revision Number and Document Date Updated. | Everett Fortner |

Approval Signatures

Prepared by:



3/1/2023

Everett H. Fortner III, PG (Preparer)

Date

Reviewed by:



3/1/2023

Marc Killingstad, PE (Subject Matter Expert)

Date

1 Introduction

Slug testing is a common field method used for estimating hydraulic conductivity. This Technical Guidance Instruction (TGI) document outlines field procedures for conducting such testing use a known volume of potable water (slug).

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

A known volume of potable water (slug) may be used to complete falling-head slug tests. Water of a known volume is poured into a well in a near-instantaneous manner. The water level response is observed using a pressure transducer. Slug tests using a water slug are most appropriate for fully submerged well screen (i.e., water table above the well screen top), and where a relatively slow response is anticipated. These constraints limit the probability of slug water entering the filter pack and vadose zone, thereby ensuring slug insertion to be near instantaneous relative to the observed response. This type of test is sometimes used to evaluate well development by performing a slug test before and after well development.

Consult with local regulatory requirements concerning underground injections. The injection volume and injectate are typically innocuous enough that a permit-by-rule authorization is granted in lieu of an underground injection control permit.

4 Personnel Qualifications

Field personnel performing the testing are required to have the following qualifications:

- Sufficient “hands-on” experience necessary to successfully complete the slug test field work. Training requirements for conducting slug tests including the review of this guidance and other applicable documents related to instrumentation.
- Demonstrate familiarity with the electronic data logging equipment (*see TGI – Water-Level Monitoring Using Data Logging Instruments*).
- Completed current health and safety training in accordance with the project health and safety plan (e.g., 40-hour Hazardous Waste Operations training and site-specific training, as appropriate).

5 Equipment List

The following materials will be available during slug testing using a water slug:

- Job safety analysis and site Health and Safety Plan
- Related project-specific requirements and plans
- Personal protective equipment, as required by the site Health and Safety Plan
- Potable water (do not use distilled water, as it is not conductive and will not work with electronic water level meters)
- Pressure transducer and cable
- Pressure transducer software
- Laptop computer, smart device (phone or tablet), and/or data transfer device
- Graduated cylinder or similar measuring device
- Funnel with large neck and opening (wide mouth)
- Water level meter
- Spring-loaded clamp and zip ties
- Decontamination equipment
- Slug test field form (paper or digital)
- Field tablet and/or daily logs[Click to enter text]

6 Cautions

- Pressure Transducers/Data Loggers (*see TGI – Water-Level Monitoring Using Data Logging Instruments*)
 - Ensure that all rental instruments and tapes have been calibrated and checked prior to use.

- Small-diameter pressure transducers (typically 0.5 to 0.75 in) are available that cover a range of pressures. Install the pressure transducer at a reasonable distance below (approximately 3 to 5 feet) the targeted drawdown estimated for the well to prevent noise. Do not install the pressure transducer closer than 6 inches from the base of the well to eliminate the possibility of fouling the transducer with material accumulated at the bottom of the well. To prevent pressure transducer malfunction or damage, do not submerge pressure transducers in excess of the operating range, and do not insert objects in the sensor opening.
- For vented pressure transducers/data loggers, test functionality with a field test of readings using a bucket or barrel filled with water. Submerge the pressure transducer, accurately measure the water head above the pressure transducer sensor and compare the measurement to the reading. If the measurements don't generally agree, there may be an issue with the instrument.
- Non-vented transducers, which record a combined pressure of barometric and the water column above the pressure transducer, can be tested in the same fashion as the vented pressure transducer (outlined above). The water column above the pressure transducer can be checked by subtracting out current atmospheric pressure.
- In general, when testing the pressure transducers, check the pressure transducer general response to changing heads by raising the pressure transducer a certain distance, observing the change in head, and then measuring the distance manually. Additionally, water level meters will be in good working condition and calibrated to true depth and ensuring that there are no breaks or splices in the cable.
- Pressure transducers will be set in the well at least 15 minutes prior to testing to allow to the instrument to thermally equilibrate with groundwater, collect static water-level measurements, and ensure that the pressure transducer cable will not stretch during testing.
- Logarithmic or head-change settings will not be used to log data, only linear.
- Prior to testing, secure pressure transducer cables at the wellhead to prevent movement that may affect measurements. Mark a reference point, such as masking tape, on the down-hole transducer cable or securing line and check regularly to detect slippage.
- Data Management
 - Data management is critical to prevent any loss. Use caution not to overwrite any previously recorded files and remember, data backup is always necessary. Multiple tests at the same well do not require for the pressure transducer to be reset and the same log can run throughout the duration of all tests. Upload data collected via smart device by email or cloud server to reduce the risk of data loss (e.g., computer failure).
- Slug Volume
 - When completing baildown or inflow testing, purge or injected volume will need to be measured accurately using a graduated cylinder.
- Initial Displacement and Recovery
 - When performing slug tests, the general rule of thumb for initial displacement is between 1 and 3 feet and/or generally less than 25% of the effective screen length. For high conductivity formations, initial displacements will be small (0.3 – 0.7 ft) to avoid remobilizing fines and to limit turbulence.

- Water levels will need to be recorded to within 80% to 95% recovery. In addition, duplicate tests will be completed only after the first test has recovered by at least 95%.
- Investigative-Derived Waste (IDW)
 - Containerize all purged water as specified in the project plans. Discharge water will be disposed of according to all applicable laws, regulations, and project guidelines. Contact the governing agencies to determine which restrictions apply. Arcadis will not be responsible for signing manifests and will not "take possession" of purged water.
- Equipment Care
 - Keep sensitive electronic equipment away from devices that generate significant magnetic fields. For example, do not place pressure transducers near electric power generators or electric pump motors. Likewise, radio signals may cause pressure transducers or computers to malfunction.
- Decontamination
 - Make sure all equipment that enters the test well (slug, water-level meter, pressure transducer) is decontaminated before use. If testing multiple wells, start with the least contaminated and progress to the most contaminated.
- Non-Aqueous Phase Liquids (NAPL)
 - Slug tests are not recommended in wells where Non-Aqueous Phase Liquids are present. Consult TKI Aquifer Testing and Characterization Focus Group lead for guidance.

7 Health and Safety Considerations

The site-specific HASP will be used to ensure that the tests are conducted in a safe manner and will include a Job Safety Analysis (JSA). The following specific health and safety issues will be considered when conducting slug tests:

- Appropriate PPE with minimum of Level D will be worn to avoid contact with site chemicals of concern during slug test.
- Well covers will be carefully removed to avoid potential contact with insects or animals. Well caps should be vented or tethered to avoid potential eye injury in case of gas buildup in the well.

8 Procedure

1. Decontaminate all down-well equipment: pressure transducer, pressure transducer cable, and water level meter in accordance with project specific requirements. In general, wells will be tested from least contaminated to more contaminated, if possible or applicable.
2. Measure depth to water and well total depth. Determine the water column length.
 - a. Multiple depth to waters should be measured and any trends will be noted.
 - b. The "static" depth to water will be representative of the water level after the well equalizes with atmosphere.
3. Review the well construction log to determine screened interval and confirm depth to bottom. If discrepancies exist, consult with project hydrogeologist.

4. Program the pressure transducer to record water levels at the following suggested frequencies. Note that the lithologic descriptions and datalogger memory will be used to select the highest measurement frequency possible.
 - a. In hydrogeologic settings where high hydraulic conductivity is expected, water levels should be measured at 0.5-second intervals, or the highest frequency available. This measurement frequency should be selected for gravels and sands.
 - b. In hydrogeologic settings where low hydraulic conductivity is expected, water levels should be measured at 1-second intervals. This measurement frequency should be selected for silts and clays.
5. If applicable, program the barometric pressure logger to record barometric pressure. The logger will be placed in the headspace of an adjacent well, or on grade, adjacent to the well being tested.
6. Install the pressure transducer deep enough within the water column to not interfere with the testing equipment. Ideally, the transducer should be 3 to 5 feet lower than the water level, not closer than 6 inches above the well bottom. Remember to use measurements and not the well bottom, as silt can clog the pressure transducer. Secure the pressure transducer cable to the well casing or other static object.
7. View the measured water level in real time. Wait for the water levels to stabilize. Note that the temperature of the pressure transducer will require to equilibrate to groundwater temperatures to ensure accurate water-level measurements (at least 15 minutes).
8. Determine the volume of the water slug. A general guideline is that initial displacements are generally between 1 to 3 feet, but will depend on the well diameter. The large the well diameter, the larger volume will be required.

| Slug Volume (gal) | Slug Volume (mL) | Casing Diameter (in) | Theoretical Initial Displacement (ft) |
|-------------------|------------------|----------------------|---------------------------------------|
| 0.25 | 946 | 2 | 1.56 |
| 0.5 | 1893 | 2 | 3.13 |
| 1 | 3785 | 2 | 6.25 |
| 0.5 | 1893 | 4 | 0.77 |
| 1 | 3785 | 4 | 1.54 |
| 2 | 7570 | 4 | 3.08 |
| 1 | 3785 | 6 | 0.68 |
| 2 | 7570 | 6 | 1.36 |
| 3 | 11355 | 6 | 2.04 |

Notes:

- gal = gallons (U.S.)
- mL = milliliters
- in = inches
- ft = feet

9. Measure the slug volume and place in a container that is easy to quickly pour from. Note the measured volume.
10. Insert the wide mouth funnel into the well casing.

11. Quickly pour the slug through the funnel and into the well. Note the approximate time required to insert the slug.
12. Observe the water level response on the real time view or use water level meter.
13. Measure depth to water, being careful not to interfere with the pressure transducer cable. Several manual depth to water measurements should be made throughout the test.
14. Allow sufficient time for water level to recover to static level. If completing one test, then 80% recovery is sufficient. Duplicate tests are highly recommended, and the next test should be completed after the first test has recovered to greater than 95%. A third test at a displacement of twice the initial is recommended.

$$\text{Percent Recovery} = ((\text{current displacement})/(\text{maximum displacement})) * 100$$

This calculation can be used to determine the depth to water that will be needed before conducting a duplicate test. For example, if your theoretical maximum displacement is 1.92 ft and 95% recovery is needed then 1.82 ft of recovery is necessary to conduct a duplicate test which means the depth to water to be less than or equal to 0.10 feet below the static water level.

15. Repeat steps 9 through 14.
16. Save all data files and finalize any field notes.
17. Review the data collected to determine the reasonableness of the preliminary results. The observation of apparently anomalous results will be discussed with senior project staff prior to additional testing or leaving the field site. The water level record for each test will show static conditions, the insertion or removal of the slug(s), and the water level response. Make notes on the field form and notebook concerning any irregularities.
18. Decontaminate all down-well equipment in accordance with project plans.

9 Waste Management

Rinse water, PPE, and other waste materials generated during equipment decontamination will be placed in appropriate containers and labeled. Containerized waste will be disposed of, consistent with appropriate waste management procedures for investigation-derived waste.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Field personnel will complete a Slug Test Field Log form for each test. The digital field form is available through the FieldNow® Application titled FieldNow – Slug Test Field Log. A hardcopy example is included in the attachments. As previously noted, it is generally recommended to conduct three tests per well (the original

displacement, a duplicate, and double original displacement); therefore, one field log will be completed for each test. Multiple tests can be entered into the FieldNow® digital application for each well tested. Field equipment calibration, decontamination activities, and waste management activities should be recorded in the daily field log. Data that has been collected must be transmitted at the end of the day (notes/forms/data file). The files must be transmitted to the appropriate project file storage at the end of each day (via direct transfer or email to data manager). Work completed that day and any relevant observations noted during the daily activities as well as copies of the data mentioned above should be summarized and provided in an email. The appropriate team member will review the data for accuracy and provide feedback.

11 Quality Assurance

Review data collected during field testing to determine reasonableness/quality given site-specific conditions. Again, this can also be completed using the transducer in real-time viewing mode as the test progresses and resulting charts to confirm with project hydrogeologist. Compare the theoretical head displacement calculated from the slug volume or pressure to the observed displacement. If the data are questionable, the field equipment will need to be checked to confirm proper working order and the test may be repeated, if possible. Consult with the project hydrogeologist to work through issues encountered in the field and to help determine test validity. Any issues that may affect the data will be recorded in the daily field log for consideration by the project hydrogeologist.

12 References

Butler, J.J., Jr., 2019. The Design, Performance, and Analysis of Slug Tests (2nd Ed), CRC Press, Boca Raton, 280p.

Butler, J.J. Jr., 2020. Slug Test Strategies for Challenging Conditions. Presentation – Midwest Geosciences Webinar. December.

Kruseman, G.P. and N.A. de Ridder, 1994. Analysis and Evaluation of Pumping Test Data (2nd ed.), Publication 47, Intern. Inst. for Land Reclamation and Improvement, Wageningen, The Netherlands, 370p.[Click to enter text]

Attachment C

TGI for Baildown Testing

TGI – Baildown Slug Testing

Rev: 7

Rev Date: March 1, 2023

Version Control

| Issue | Revision No. | Date Issued | Page No. | Description | Reviewed By |
|-------|--------------|-------------------|----------|---|-------------------------------------|
| | 1 | | | | |
| | 2 | | | | |
| | 3 | | | | |
| | 4 | March 5, 2015 | All | New TGI template | Everett Fortner Marc Killingstad |
| | 5 | April 28, 2022 | All | New TGI template; Updates to pneumatic slug testing guidance; Addition of forms | Everett Fortner Marc Killingstad |
| | 6 | December 22, 2022 | 6 | Clarification on barometric pressure logger and slug | |
| | 7 | March 1, 2023 | All | Annual Review completed by SME. Document revision number and document date update. | Everett Fortner |

Approval Signatures

Prepared by:



3/1/2023

Everett H. Fortner III, PG (Preparer)

Date

Reviewed by:



3/1/2023

Marc Killingstad, PE (Subject Matter Expert)

Date

1 Introduction

Slug testing is a common field method used for estimating hydraulic conductivity. This Technical Guidance Instruction (TGI) document outlines field procedures for conducting such testing using a bailer.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

A bailer is used to remove a volume of water (slug) to complete rising-head tests. A bailer removes water from a well in a near-instantaneous manner. The water level response is observed using a pressure transducer.

4 Personnel Qualifications

Field personnel performing the testing are required to have the following qualifications:

- Sufficient “hands-on” experience necessary to successfully complete the slug test field work. Training requirements for conducting slug tests including the review of this guidance and other applicable documents related to instrumentation.

- Demonstrate familiarity with the electronic data logging equipment (see *TGI – Water-Level Monitoring Using Data Logging Instruments*).
- Completed current health and safety training in accordance with the project health and safety plan (e.g., 40-hour Hazardous Waste Operations training and site-specific training, as appropriate)

5 Equipment List

The following materials should be available during slug testing using a bailer slug:

- Job safety analysis and site Health and Safety Plan
- Related project-specific requirements and plans
- Personal protective equipment, as required by the site Health and Safety Plan
- Bailers of known size/capacity
- Pressure transducer and cable
- Pressure transducer software
- Graduated cylinder or similar measuring device
- Rope or cables (for deep wells) (chemical resistant, low stretch is optimal)
- Laptop computer, smart device (phone or tablet), and/or data transfer device
- Water level meter
- Measuring tape
- Decontamination equipment
- Spring-loaded clamps or zip ties
- Slug test field forms (paper or digital)
- Field tablet and/or daily logs
- Waterproof marker.

6 Cautions

- Pressure Transducers/Data Loggers (see *TGI – Water-Level Monitoring Using Data Logging Instruments*)
 - Ensure that all rental instruments and tapes have been calibrated and checked prior to use.
 - Small-diameter pressure transducers (typically 0.5 to 0.75 in) are available that cover a range of pressures. Install the pressure transducer at a reasonable distance below (approximately 3 to 5 feet) the targeted drawdown estimated for the well to prevent noise. Do not install the pressure transducer closer than 6 inches from the base of the well to eliminate the possibility of fouling the transducer with material accumulated at the bottom of the well. To prevent pressure transducer malfunction or damage, do not submerge pressure transducers in excess of the operating range, and do not insert objects in the sensor opening.
 - For vented pressure transducers/data loggers, test functionality with a field test of readings using a bucket or barrel filled with water. Submerge the pressure transducer, accurately measure the water head above the pressure transducer sensor and compare the measurement to the reading. If the measurements don't generally agree, there may be an issue with the instrument.

- Non-vented transducers, which record a combined pressure of barometric and the water column above the pressure transducer, can be tested in the same fashion as the vented pressure transducer (outlined above). The water column above the pressure transducer can be checked by subtracting out current atmospheric pressure.
- In general, when testing the pressure transducers, check the pressure transducer general response to changing heads by raising the pressure transducer a certain distance, observing the change in head, and then measuring the distance manually. Additionally, water level meters will be in good working condition and calibrated to true depth and ensuring that there are no breaks or splices in the cable.
- Pressure transducers will be set in the well at least 15 minutes prior to testing to allow to the instrument to thermally equilibrate with groundwater, collect static water-level measurements, and ensure that the pressure transducer cable will not stretch during testing.
- Logarithmic or head-change settings will not be used to log data, only linear.
- Prior to testing, secure pressure transducer cables at the wellhead to prevent movement that may affect measurements. Mark a reference point, such as masking tape, on the down-hole transducer cable or securing line and check regularly to detect slippage.
- A barometric pressure transducer will be utilized, regardless of in-well pressure transducer type (vented or non-vented) if a slug test is expected to take more than four hours for full recovery. Barometric trends in long-term data sets may need to be evaluated or removed from the data set.
- Data Management
 - Data management is critical to prevent any loss. Use caution not to overwrite any previously recorded files and remember, data backup is always necessary. Multiple tests at the same well do not require for the pressure transducer to be reset and the same log can run throughout the duration of all tests. Upload data collected via smart device by email or cloud server to reduce the risk of data loss (e.g., computer failure).
- Slug Volume
 - When completing baildown or inflow testing, purge or injected volume will need to be measured accurately using a graduated cylinder.
 - The length and diameter of the bailer will be measured in the field and noted in the field logs to accurately estimate theoretical displacement.
- Initial Displacement and Recovery
 - When performing slug tests, the general rule of thumb for initial displacement is between 1 and 3 feet and/or generally less than 25% of the effective screen length. For high conductivity formations, initial displacements will be small (0.3 – 0.7 ft) to avoid remobilizing fines and to limit turbulence.
 - Water levels will need to be recorded to within 80% to 95% recovery. In addition, duplicate tests will be completed only after the first test has recovered by at least 95%.
- Equipment Care
 - Keep sensitive electronic equipment away from devices that generate significant magnetic fields. For example, do not place pressure transducers near electric power generators or electric pump motors. Likewise, radio signals may cause pressure transducers or computers to malfunction.
- Decontamination
 - Make sure all equipment that enters the test well (slug, water-level meter, pressure transducer) is decontaminated before use. If testing multiple wells, start with the least contaminated and progress to the most contaminated.

- Non-Aqueous Phase Liquids (NAPL)
 - Slug tests are not recommended in wells where Non-Aqueous Phase Liquids are present. Consult TKI Aquifer Testing and Characterization Focus Group lead for guidance.[Click to enter text]

7 Health and Safety Considerations

The site-specific HASP will be used to ensure that the tests are conducted in a safe manner and will include a Job Safety Analysis (JSA). The following specific health and safety issues will be considered when conducting slug tests:

- Appropriate PPE with minimum of Level D should be worn to avoid contact with site chemicals of concern during slug test.
- Well covers should be carefully removed to avoid potential contact with insects or animals. Well caps will be vented or tethered to avoid potential eye injury in case of gas buildup in the well.

8 Procedure

1. Select a bailer according to a target initial displacement using the table below. A general guideline is that initial displacements are between 1 and 3 feet, but should depend on the anticipated response (i.e., smaller initial displacements will be chosen for formations with high hydraulic conductivity). When utilizing the FieldNow® – Slug Test Field Log, the bailer size and length and the well diameter can be entered, and a theoretical displacement will be automatically calculated.

| Bailer Volume (gal) | Bailer Volume (mL) | Casing Diameter (in) | Theoretical Initial Displacement (ft) |
|---------------------|--------------------|----------------------|---------------------------------------|
| 0.25 | 946 | 2 | 1.56 |
| 0.5 | 1893 | 2 | 3.13 |
| 1 | 3785 | 2 | 6.25 |
| 0.5 | 1893 | 4 | 0.77 |
| 1 | 3785 | 4 | 1.54 |
| 2 | 7570 | 4 | 3.08 |
| 1 | 3785 | 6 | 0.68 |
| 2 | 7570 | 6 | 1.36 |
| 3 | 11355 | 6 | 2.04 |

Notes:

gal = gallons, U.S. liquid
 mL = milliliters
 in = inches
 ft = feet

2. Decontaminate all down-well equipment: pressure transducer and cable, slug(s), rope or cable, water level meter in accordance with project-specific requirements. In general, wells will be tested from least contaminated to more contaminated, if possible or applicable.
3. Measure depth to water and well total depth. Determine the water column length.
 - a. Multiple depth to waters will be measured and any trends will be noted.
 - b. The "static" depth to water should be representative of the water level after the well equalizes with atmosphere.
4. Measure depth to water and well total depth. Total depth should be taken using a weighted tag line. Determine the water column length.
5. Review the well construction log to determine screened interval and confirm depth to bottom. If discrepancies exist, consult with project hydrogeologist.
6. Program the pressure transducer to record water levels at the following suggested frequencies. Note that the lithologic descriptions and datalogger memory will be used to select the highest measurement frequency possible.
 - a. In hydrogeologic settings where high hydraulic conductivity is expected, water levels will be measured at 0.5-second intervals, or the highest frequency available. This measurement frequency should be selected for gravels and sands.
 - b. In hydrogeologic settings where low hydraulic conductivity is expected, water levels will be measured at 1- to 2-second intervals. This measurement frequency should be selected for silts and clays.
7. If applicable, program the barometric logger to record barometric pressure. The logger will be placed in the headspace of an adjacent well, or on grade, adjacent to the well being tested.
8. Install the pressure transducer deep enough within the water column to not interfere with the testing equipment. Ideally, the transducer should be 5 to 10 feet lower than the maximum depth of the slug testing equipment not closer than 6 inches above the well bottom. Remember to use measurements and not the well bottom, as silt can clog the pressure transducer. Clamp the pressure transducer cable to the well casing or other static object.
9. View the measured water level in real time on the laptop computer. Wait for the water levels to stabilize. Note that the temperature of the pressure transducer should be permitted to equilibrate to groundwater temperatures to ensure accurate water-level measurements.

Measure the bailer and rope assembly length and mark the rope at a length as follows: Rope Mark #1 = Depth to Potentiometric Surface from TOC

Rope Mark #2 = Depth to Potentiometric Surface from TOC + Length of Bailer + Safety Factor (Safety Factor = 10% of the Length of Slug)
10. When deployed, Rope Mark #2 should ensure that the bailer is fully submerged. If a sufficient water column is not available to obtain a full bailer, measure the volume removed upon removal.
11. Slowly insert the bailer into the well and stop just above the potentiometric surface Rope Mark #1.
12. With slack in the rope and the bailer being suspended above the water column, lower the bailer and place the Rope Mark #2 at the top of casing. Clamp the non-bailer end of the rope to a static object to keep in place.
13. Wait for water level to equilibrate using response from the pressure transducer or from water level meter.
14. Quickly remove the bailer from the water column and carefully pull it to surface. Pour the removed water into an empty bucket.

15. Observe the water level response on the laptop computer and/or measure depth to water, being careful not to interfere with the pressure transducer cable. Several manual depth to water measurements will be made throughout the test.
16. Allow sufficient time for water level to recover to static level. If completing one test, then 80% recovery is sufficient. Duplicate tests are highly recommended, and the next test will be completed after the first test has recovered to greater than 95%. A third test at a displacement of twice or half the initial is recommended.
$$\text{Percent Recovery} = \left(\frac{\text{current displacement}}{\text{maximum displacement}} \right) * 100$$

This calculation can be used to determine the depth to water that will be needed before conducting a duplicate test. For example, if your theoretical maximum displacement is 1.92 ft and 95% recovery is needed then 1.82 ft of recovery is necessary to conduct a duplicate test which means the depth to water would need to be less than or equal to 0.10 feet below the static water level.
17. Measure the volume of water removed by the bailer that was poured into the empty bucket using a graduated cylinder.
18. Save or transmit all data files and finalize any field notes.
19. Review the data collected to determine the reasonableness of the preliminary results. The observation of apparently anomalous results should be discussed with senior project staff prior to additional testing or leaving the field site. The water level record for each test will show static conditions, the insertion or removal of the slug(s), and the water level response. Make notes on the field form and notebook concerning any irregularities.
20. Decontaminate all down-well equipment.

9 Waste Management

Rinse water, PPE, and other waste materials generated during equipment decontamination will be placed in appropriate containers and labeled. Containerized waste will be disposed of, consistent with appropriate waste management procedures for investigation-derived waste.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Field personnel will complete a Slug Test Field Log form for each test. The digital field form is available through the FieldNow® Application titled FieldNow – Slug Test Field Log. A hardcopy example is included in the attachments. As previously noted, it is generally recommended to conduct three tests per well (the original displacement, a duplicate, and double original displacement); therefore, one field log will be completed for each test. Multiple tests can be entered into the FieldNow® digital application for each well tested. Field equipment

calibration, decontamination activities, and waste management activities should be recorded in the daily field log. Data that has been collected must be transmitted at the end of the day (notes/forms/data file). The files must be transmitted to the appropriate project file storage at the end of each day (via direct transfer or email to data manager). Work completed that day and any relevant observations noted during the daily activities as well as copies of the data mentioned above should be summarized and provided in an email. The appropriate team member will review the data for accuracy and provide feedback.

11 Quality Assurance

Review data collected during field testing to determine reasonableness/quality given site-specific conditions. Again, this can also be completed using the transducer in real-time viewing mode as the test progresses. Compare the theoretical head displacement calculated from the slug volume or pressure to the observed displacement. If the data are questionable, the field equipment will be checked to confirm proper working order and the test may be repeated, if possible. Consult with the project hydrogeologist to work through issues encountered in the field and to help determine test validity. Any issues that may affect the data should be recorded in the daily field log for consideration by the project hydrogeologist.

12 References

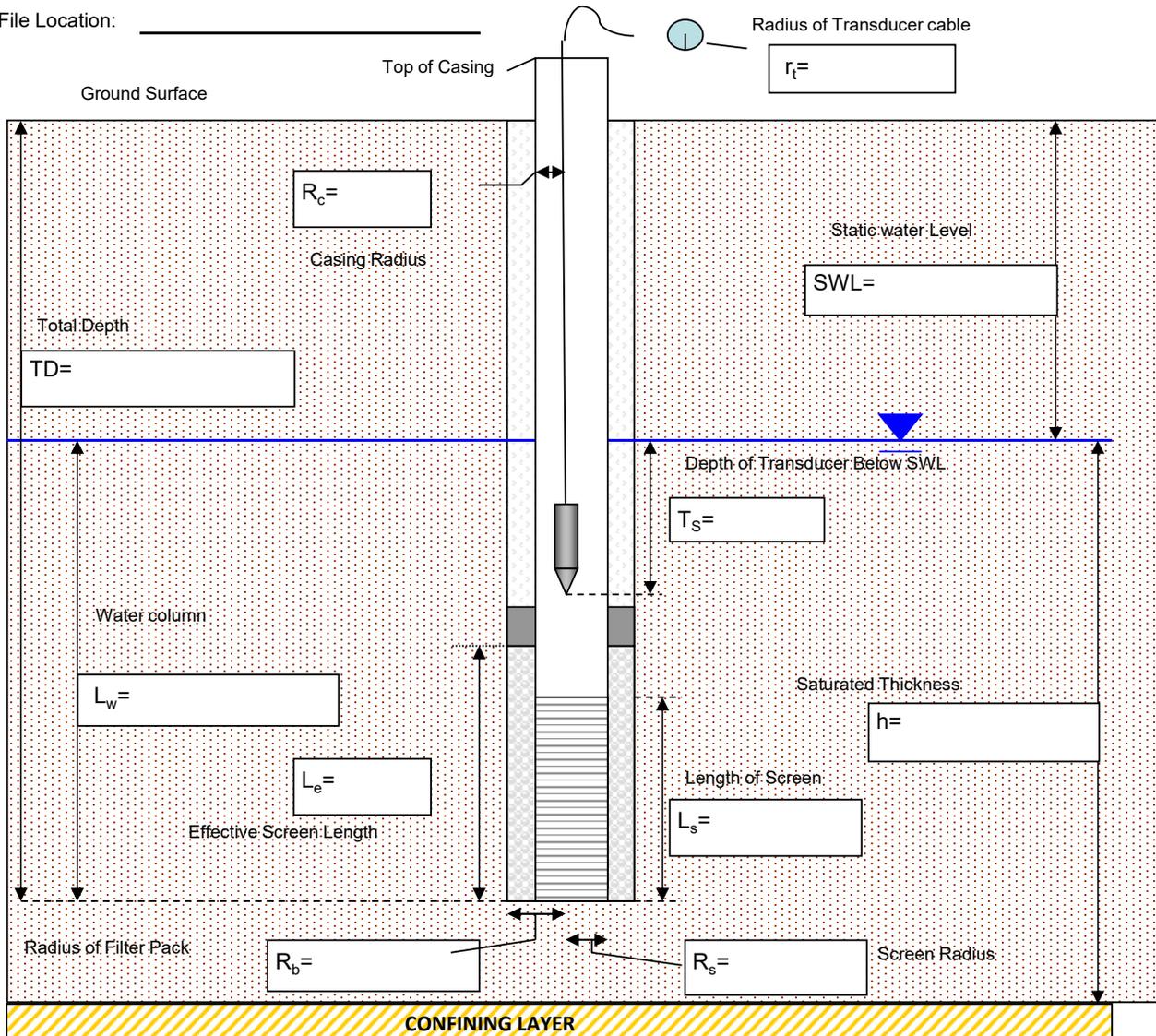
- Butler, J.J., Jr., 2019. The Design, Performance, and Analysis of Slug Tests (2nd Ed), CRC Press, Boca Raton, 280p.
- Butler, J.J. Jr., 2020. Slug Test Strategies for Challenging Conditions. Presentation – Midwest Geosciences Webinar. December.
- Kruseman, G.P. and N.A. de Ridder, 1994. Analysis and Evaluation of Pumping Test Data (2nd ed.), Publication 47, Intern. Inst. for Land Reclamation and Improvement, Wageningen, The Netherlands, 370p.

Attachment

Slug Test Field Log

SLUG TEST LOG

Site Name: _____ Project No: _____ Page: ___ of _____
 Test Well ID: _____ Observation Well ID (if different): _____ Date: _____
 Completed By: _____ Weather: _____
 Test Type & No: Rising _____ Falling _____ Pressure Transducer SN: _____
 Data File Name(s): _____ Barologger Serial No (if used): _____
 File Location: _____



WELL PARAMETERS REQUIRED FOR CALCULATING HYDRAULIC CONDUCTIVITY:

- L_e = Effective screen length, including the sand pack
- L_s = True screen length
- L_w = Length of water column in Well (TD-SWL)
- R_s = Screen radius
- R_b = Radius of filter Pack or borehole
- R_c = Casing radius
- r_t = Radius of the transducer cable (can be ignored if less than 1/8 inch)
- T_s = Depth the transducer is submerged below the SWL
- SWL = Static water level
- TD = Total depth of well/screen from reference point
- h = Saturated thickness of aquifer
- H_0 = Initial head change at instant the slug test is started.
- Aquifer Type = Confined or unconfined

COMMON CONVERSIONS:

- 1 foot = 12 inches
- 1 foot³ ≈ 7.48 gallons
- 1 gallon ≈ 3.785 Liters
- 1 psi ≈ 2.3 feet of water

Attachment D

TGI for Pneumatic Slug Testing

TGI – Pneumatic Slug Testing

Rev: 7

Rev Date: March 1, 2023

Version Control

| Issue | Revision No. | Date Issued | Page No. | Description | Reviewed By |
|-------|--------------|-------------------|----------|---|-------------------------------------|
| | 1 | | | | |
| | 2 | | | | |
| | 3 | | | | |
| | 4 | March 5, 2015 | All | New TGI template | Everett Fortner Marc Killingstad |
| | 5 | April 28, 2022 | All | New TGI template; Updates to pneumatic slug testing guidance; Addition of forms | Everett Fortner Marc Killingstad |
| | 6 | December 22, 2022 | 6 | Clarification on barometric pressure logger and slug | |
| | 7 | March 1, 2023 | All | Annual review completed by SME. Document Revision Number and Date Updated. | Everett Fortner |

Approval Signatures

Prepared by:



3/1/2023

Everett H. Fortner III, PG (Preparer)

Date

Reviewed by:



3/1/2023

Marc Killingstad, PE (Subject Matter Expert)

Date

1 Introduction

Slug testing is a common field method used for estimating hydraulic conductivity. This Technical Guidance Instruction (TGI) document outlines field procedures for conducting such testing using pneumatic methods.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

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3 Scope and Application

Pneumatic slug tests are conducted by sealing the well head and applying air pressure to depress the water level. As air pressure is increased in the well, the water level falls until the water pressure and the air pressure return to equilibrium. After the water level is stable, air is released from the sealed well head by opening an air release valve. The water level recovery (i.e., rising head) typically produces very high-quality data with little interference. A pressure transducer is used to monitor and record the change of the water level in the well during the pneumatic slug test.

4 Personnel Qualifications

Field personnel performing the testing are required to have the following qualifications:

- Sufficient “hands-on” experience necessary to successfully complete the slug test field work. Training requirements for conducting slug tests including the review of this guidance and other applicable documents related to instrumentation.
- Demonstrate familiarity with the electronic data logging equipment (see *TGI – Water-Level Monitoring Using Data Logging Instruments*).
- Completed current health and safety training in accordance with the project health and safety plan (e.g., 40-hour Hazardous Waste Operations training and site-specific training, as appropriate)

5 Equipment List

The following materials should be available during slug testing using a water slug:

- Job safety analysis and site Health and Safety Plan
- Related project-specific requirements and plans
- Personal protective equipment, as required by the site Health and Safety Plan
- Pneumatic slug test manifold (see attached Figure 1 for suggested configuration)
- Pressure transducer and cable
- Pressure transducer software
- Air pressurization source (compressed or pump) and appropriate hoses
- Leak prevention supplies (Teflon pipe sealant, plumbers putty or similar product)
- Laptop computer, smart device (phone or tablet), and/or data transfer device
- Water level meter
- Measuring tape
- Decontamination equipment
- Slug test field forms (paper or digital)
- Field tablet and/or daily logs
- Waterproof marker.

6 Cautions

- Pressure Transducers/Data Loggers (see *TGI – Water-Level Monitoring Using Data Logging Instruments*)
 - Ensure that all rental instruments and tapes have been calibrated and checked prior to use.
 - Small-diameter pressure transducers (typically 0.5 to 0.75 in) are available that cover a range of pressures. Install the pressure transducer at a reasonable distance below (approximately 3 to 5 feet) the targeted drawdown estimated for the well to prevent noise. Do not install the pressure transducer closer than 6 inches from the base of the well to eliminate the possibility of fouling the transducer with material accumulated at the bottom of the well. To prevent pressure transducer malfunction or damage, do not submerge pressure transducers in excess of the operating range, and do not insert objects in the sensor opening.
 - For vented pressure transducers/data loggers, test functionality with a field test of readings using a bucket or barrel filled with water. Submerge the pressure transducer, accurately measure the

- water head above the pressure transducer sensor and compare the measurement to the reading. If the measurements don't generally agree, there may be an issue with the instrument.
- Non-vented transducers, which record a combined pressure of barometric and the water column above the pressure transducer, can be tested in the same fashion as the vented pressure transducer (outlined above). The water column above the pressure transducer can be checked by subtracting out current atmospheric pressure.
 - In general, when testing the pressure transducers, check the pressure transducer general response to changing heads by raising the pressure transducer a certain distance, observing the change in head, and then measuring the distance manually. Additionally, water level meters will be in good working condition and calibrated to true depth and ensuring that there are no breaks or splices in the cable.
 - Pressure transducers will be set in the well at least 15 minutes prior to testing to allow to the instrument to thermally equilibrate with groundwater, collect static water-level measurements, and ensure that the pressure transducer cable will not stretch during testing.
 - Logarithmic or head-change settings will not be used to log data, only linear.
 - Prior to testing, secure pressure transducer cables at the wellhead to prevent movement that may affect measurements. Mark a reference point, such as masking tape, on the down-hole transducer cable or securing line and check regularly to detect slippage.
 - A barometric pressure transducer will be utilized, regardless of in-well pressure transducer type (vented or non-vented) if a slug test is expected to take more than four hours for full recovery. Barometric trends in long-term data sets may need to be evaluated or removed from the data set.
- Data Management
 - Data management is critical to prevent any loss. Use caution not to overwrite any previously recorded files and remember, data backup is always necessary. Multiple tests at the same well do not require for the pressure transducer to be reset and the same log can run throughout the duration of all tests. Upload data collected via smart device by email or cloud server to reduce the risk of data loss (e.g., computer failure).
 - Slug Volume
 - Manifold pressure just prior to release will be noted in the field logs for comparison to actual displacement recorded in the data set.
 - Initial Displacement and Recovery
 - When performing slug tests, the general rule of thumb for initial displacement is between 1 and 3 feet and/or generally less than 25% of the effective screen length. For high conductivity formations, initial displacements will be small (0.3 – 0.7 ft) to avoid remobilizing fines and to limit turbulence.
 - Water levels will need to be recorded to within 80% to 95% recovery. In addition, duplicate tests will be completed only after the first test has recovered by at least 95%.
 - Equipment Care
 - Keep sensitive electronic equipment away from devices that generate significant magnetic fields. For example, do not place pressure transducers near electric power generators or electric pump motors. Likewise, radio signals may cause pressure transducers or computers to malfunction.
 - Decontamination
 - Make sure all equipment that enters the test well (slug, water-level meter, pressure transducer) is decontaminated before use. If testing multiple wells, start with the least contaminated and progress to the most contaminated.

- Non-Aqueous Phase Liquids (NAPL)
 - Slug tests are not recommended in wells where Non-Aqueous Phase Liquids are present. Consult TKI Aquifer Testing and Characterization Focus Group lead for guidance.

7 Health and Safety Considerations

The site-specific HASP will be used to ensure that the tests are conducted in a safe manner and will include a Job Safety Analysis (JSA). The following specific health and safety issues will be considered when conducting slug tests:

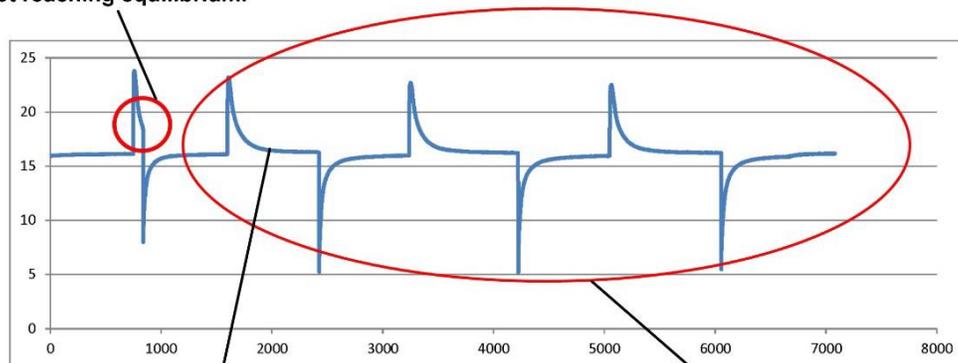
- Appropriate PPE with minimum of Level D should be worn to avoid contact with site chemicals of concern during slug test.
- Well covers should be carefully removed to avoid potential contact with insects or animals. Well caps will be vented or tethered to avoid potential eye injury in case of gas buildup in the well.
- Pressurization hazards associated with pneumatic slug testing should be considered during test planning and implementation.

8 Procedure

1. Decontaminate all down-well equipment: pressure transducer, cable, and water level meter in accordance with project-specific requirements. In general, wells will be tested from least contaminated to more contaminated, if possible or applicable.
2. Measure depth to water and well total depth. Determine the water column length.
 - a. Multiple depth to waters will be measured and any trends will be noted.
 - b. The "static" depth to water should be representative of the water level after the well equalizes with atmosphere.
3. Review the well construction log to determine screened interval and confirm depth to bottom. If discrepancies exist, consult with project hydrogeologist.
4. Attach the pneumatic slug test manifold onto the top of the well casing. Tighten the rubber connector to ensure an airtight seal.
5. Set up the air compressor or compressed air source with associated regulators.
6. If applicable, program the barometric logger to record barometric pressure. The logger should be placed in the headspace of an adjacent well, or on grade, adjacent to the well being tested.
7. Program the pressure transducer to record water levels at the following frequencies. Note that the lithologic descriptions and datalogger memory will be used to select the highest measurement frequency possible.
 - a. In hydrologic settings where high hydraulic conductivity is expected, water levels will be measured at 0.5-second intervals, or the highest frequency available. This measurement frequency will be selected for gravels and sands.
 - b. In hydrologic settings where low hydraulic conductivity is expected, water levels will be measured at 1-second intervals. This measurement frequency should be selected for silts and clays.

8. Install the pressure transducer deep enough to be below the maximum pressurization planned. Ideally, the transducer will be placed 3 to 5 feet below the water level. Secure the pressure transducer cable at the top of the manifold with the airtight fitting.
9. View the measured water level in real time. Wait for the water levels to stabilize (at least 15 minutes). Note that the temperature of the pressure transducer will be permitted to equilibrate to groundwater temperatures to ensure accurate water-level measurements.
10. Re-measure the depth to water.
11. Close the air release valve.
12. Close the inlet air valve with the pressure regulator closed.
13. Verify that the incoming pressure from compressor or air supply is less than safe operating pressure of the manifold pressure regulator (<10 psi is necessary) before attaching air hose (not applicable for hand pump). Note that hand pumps are applicable to small diameter wells only and may have complications if the manifold or well casing has leaks. A compressor or compressed air source is recommended.
14. Attach air hose and open regulator to verify incoming pressure.
15. Close regulator and open the inlet air valve.
16. Slowly open the manifold pressure regulator to pressurize well head and depress water level a pre-determined distance (2.31 feet of water is equal to 1 psi). Keep to the rule of thumb of 1 to 3 feet displacement and follow best practices with two duplicate tests and a third test with double or half the displacement. For high conductivity formations, initial displacements should be small (0.3 – 0.7 ft) to avoid remobilizing fines and to limit turbulence. Begin with a low pressure and gradually increase the pressure in order to obtain the desired displacement. **DO NOT OVER PRESSURIZE THE WELL** (do not exceed ~2 psi). If using a hand pump, pressurize the well head with pump with regulator open.
17. Close the regulator and leak check the system with leak detection fluid and fix any leaks (plumbers putty). If the leak is very slow, or down the well, the manifold regulator may be used to maintain a constant pressure head.
18. Check the pressure transducer response and air pressure to verify system is stable. This may take several minutes as the pressure transducer is equalizing to both the pressurization in the well and the displaced water column (see below figure). Stabilization is reached once the reported pressure returns to near the original, static, pressure. If it is stable proceed to the next step, if not check the seals.

Example showing the first test not reaching equilibrium.



Pressure is allowed to equilibrate and return to original reading. Several minutes of baseline are recorded before air slug release.

Good Tests

19. Record a baseline pressure for a minimum of 5 minutes. Record all applicable data on the electronic or field form.
20. Close inlet valve and quickly open the release valve to initiate the test.
21. Allow sufficient time for water level to recover to static level. If completing one test (just a falling head test or just a rising head test), then 80% recovery is sufficient. Duplicate tests are highly recommended, and the next test should be completed after the first test has recovered to greater than 95%. A third test at a displacement of twice the initial is recommended.
22. Save all data files to the laptop, email or upload data, and finalize any field notes.
23. Review the data collected to determine the reasonableness of the preliminary results. The observation of apparently anomalous results should be discussed with senior project staff prior to proceeding. The water level record for each test should show static conditions, pressurization of the well column, and the recovery response. Make notes on the field form and notebook concerning any irregularities.
24. Decontaminate all down-well equipment.

9 Waste Management

Rinse water, PPE, and other waste materials generated during equipment decontamination should be placed in appropriate containers and labeled. Containerized waste should be disposed of, consistent with appropriate waste management procedures for investigation-derived waste.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Field personnel will complete a Slug Test Field Log form for each test. The digital field form is available through the FieldNow® Application titled *FieldNow – Slug Test Field Log*. A hardcopy example is included in the attachments. As previously noted, it is generally recommended to conduct three tests per well (the original displacement, a duplicate, and double original displacement); therefore, one field log should be completed for each test. Multiple tests can be entered into the FieldNow® digital application for each well tested. Field equipment calibration, decontamination activities, and waste management activities should be recorded in the daily field log. Data that has been collected must be transmitted at the end of the day (notes/forms/data file). The files must be transmitted to the appropriate project file storage at the end of each day (via direct transfer or email to data manager). Work completed that day and any relevant observations noted during the daily activities as well as copies of the data mentioned above should be summarized and provided in an email. The appropriate team member will review the data for accuracy and provide feedback.

11 Quality Assurance

Review data collected during field testing to determine reasonableness/quality given site-specific conditions. Again, this can also be completed using the transducer in real-time viewing mode as the test progresses. Compare the theoretical head displacement calculated from the slug volume or pressure to the observed displacement. If the data are questionable, the field equipment should be checked to confirm proper working order and the test may be repeated, if possible. Consult with the project hydrogeologist to work through issues encountered in the field and to help determine test validity. Any issues that may affect the data should be recorded in the daily field log for consideration by the project hydrogeologist.

12 References

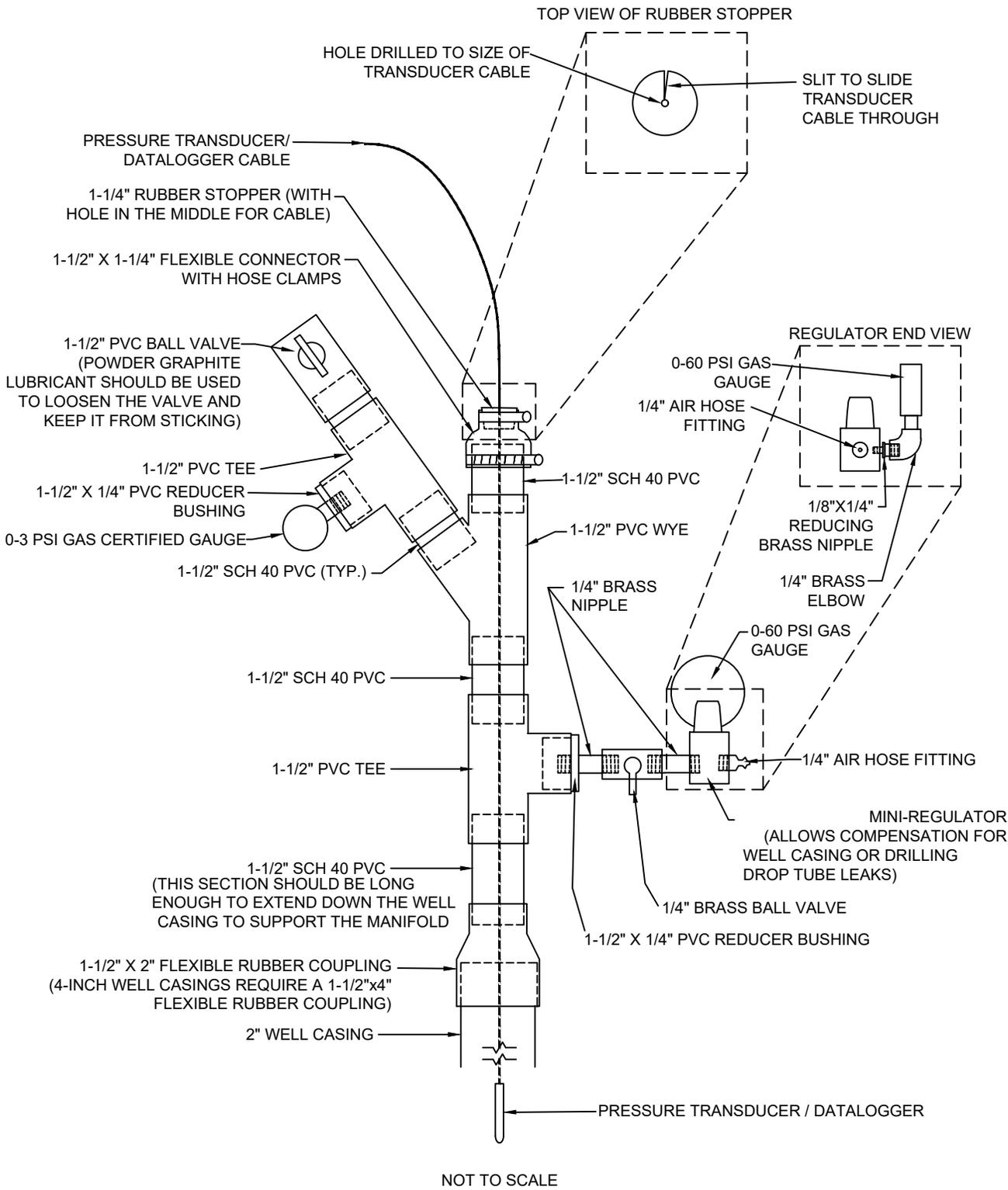
- Butler, J.J., Jr., 2019. The Design, Performance, and Analysis of Slug Tests (2nd Ed), CRC Press, Boca Raton, 280p.
- Butler, J.J. Jr., 2020. Slug Test Strategies for Challenging Conditions. Presentation – Midwest Geosciences Webinar. December.
- Kruseman, G.P. and N.A. de Ridder, 1994. Analysis and Evaluation of Pumping Test Data (2nd ed.), Publication 47, Intern. Inst. for Land Reclamation and Improvement, Wageningen, The Netherlands, 370p.

13 Attachments

- Figure 1 Slug Test Manifold
Pneumatic Slug Test Field Log

Attachments

CITY: (Columbus, Ohio) DIV: (GROUP: (env)) DB: (R. Smith) LD: (Opt) PIC: (Opt) PM: (T. Forthner) TM: (Opt) LVR: (Option: OFF=REF*)
 C:\0-TEMP\TGI2021\SLUG TEST MANIFOLD.dwg LAYOUT: FIG 1 SAVED: 11/19/2021 11:26 AM ACADVER: 23.1S (LMS TECH) PAGES: 1 BY: SMITH, BOB
 XREFS:



| | |
|-------------------------------------|--------------------|
| Aquifer Testing Focus Group | |
| PNEUMATIC SLUG TEST MANIFOLD | |
| | FIGURE 1 |

ARCADIS

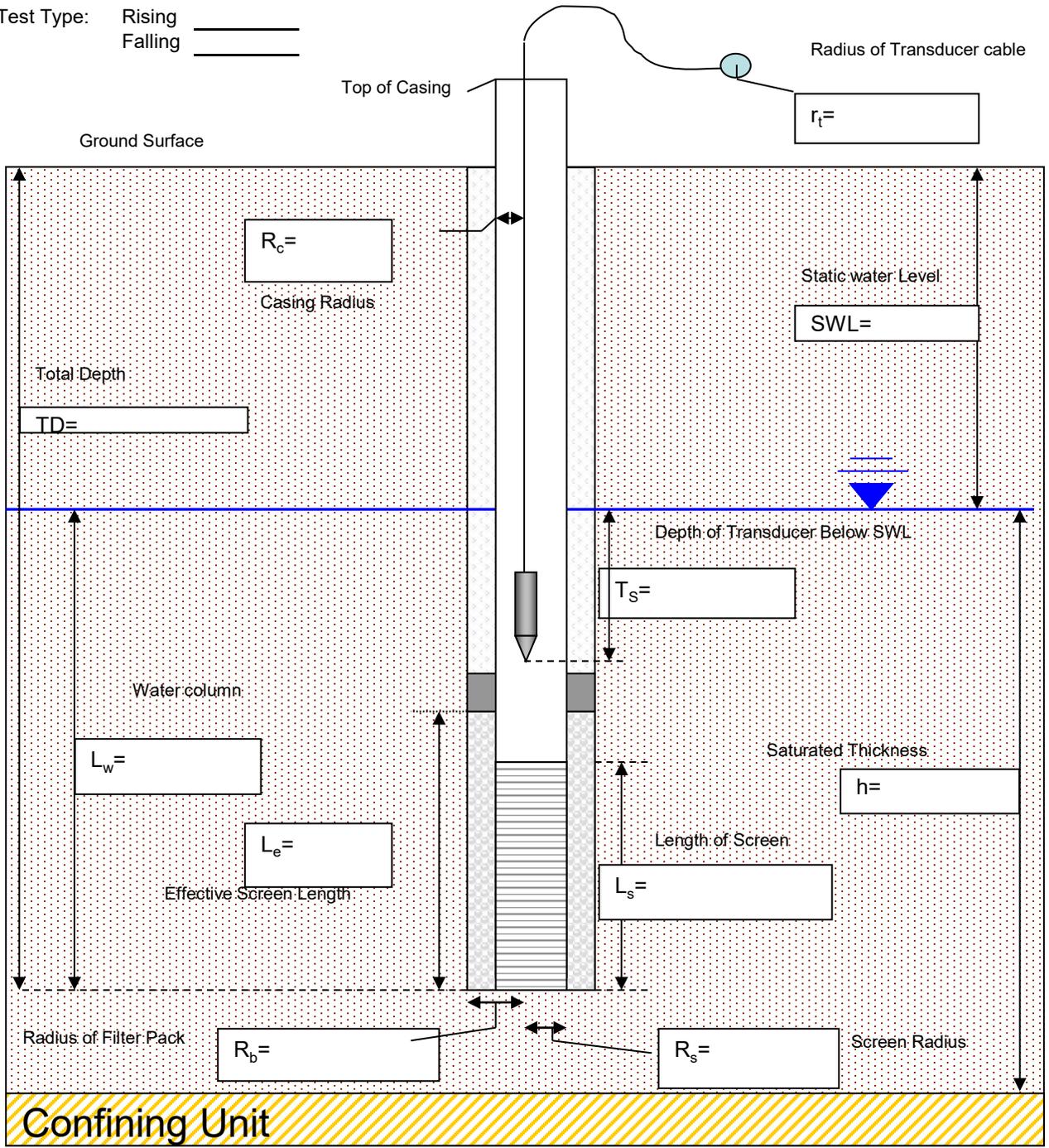
Pneumatic Slug Test Log

Site Name: _____ Project No: _____ Page: _____ of _____

Well No: _____ Prepared By: _____ Date: _____ Time: _____

Completed By: _____

Test Type: Rising _____
 Falling _____



ARCADIS

Pneumatic Slug Test Log

Site Name: _____ Project No: _____ Page: ___ of ___

Well No: _____ Prepared By: _____ Date: _____ Time: _____

TESTS

Number of Tests: _____ Data File Name: _____ Data File Location: _____

Input Pressure: _____ Pressure Transducer SN: _____ r_t : _____

Test: T_s Baseline: _____ Manifold Pressure Reading: _____
 _____ H_o : _____ Test Start _____ Test End _____

Test: T_s Baseline: _____ Manifold Pressure Reading: _____
 _____ H_o : _____ Test Start _____ Test End _____

Test: T_s Baseline: _____ Manifold Pressure Reading: _____
 _____ H_o : _____ Test Start _____ Test End _____

Notes:

- H_o Initial change in head at instant the slug test is started
- r_t Radius of transducer cable
- T_s Depth of transducer below static water level

Theoretical Change in Head - 2.307 feet = 1 psi

| (Feet) | (psi) | (Feet) | (psi) | (Feet) | (psi) |
|--------|-------|--------|-------|--------|-------|
| 0.50 | 0.22 | 1.50 | 0.65 | 2.50 | 1.08 |
| 0.75 | 0.33 | 1.75 | 0.76 | 2.75 | 1.19 |
| 1.00 | 0.43 | 2.00 | 0.87 | 3.00 | 1.30 |
| 1.25 | 0.54 | 2.25 | 0.98 | 3.25 | 1.41 |

Well Parameters Required for Calculating Hydraulic Conductivity

- L_e Effective screen length, including the sand pack
- L_s True screen length
- L_w Length of water column in Well (TD-SWL)
- R_s Screen radius
- R_b Radius of filter Pack or borehole
- R_c Casing radius
- r_t Radius of the transducer cable
- T_s Depth the transducer is submerged below the SWL
- SWL Static water level
- TD Total depth of well/screen from reference point
- h Saturated thickness of aquifer
- H_o Initial head change at instant the slug test is started.
- Aquifer Type Confined or unconfined

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TGI –Collection and Logging of Bedrock Chips

Rev: 0

Rev Date: November 4, 2022

DRAFT

Version Control

| Issue | Revision No. | Date Issued | Page No. | Description | Reviewed By |
|-------|--------------|---------------------|----------|-------------|------------------|
| | 0 | November 4, 2022 | All | New TGI | Marc Killingstad |
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| | | | | | |

Approval Signatures

Prepared by:



Click or tap to
enter a date.

Jeremy Franz (Preparer)

Date

Reviewed by:

Click or tap to
enter a date.

Marc Killingstad (Subject Matter Expert)

Date

1 Introduction

This Technical Guidance Instruction (TGI) describes the procedures to be used to collect and describe bedrock chip samples. The approach is applicable to subsurface investigations employing standard air rotary, mud rotary and reverse circulation drilling methods.

2 Intended Use and Responsibilities

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

3 Scope and Application

The bedrock chip collection and description procedures presented in this TGI are applicable for subsurface investigations where the project objectives include describing general lithology and bedrock characteristics including:

- Overburden-bedrock interface
- Top of competent/unweathered rock
- Rock type – dominant and secondary
- Depositional environment specific texture descriptions (e.g., volcanic glass, aphanitic or phaneritic textures, foliation, schistosity, gneissic banding, ect.)

- Grainsize, grain shape, etc. in clastic sediments such as sand and gravel.
- Full description of cohesive materials such as silt and clay
- Color
- Chemical alteration of rock minerals
- Veining, including nature of vein filling materials
- Secondary mineralization

The approach and level of detail presented is appropriate for most environmental-site subsurface investigations. Given the diverse nature of bedrock, and variety of potential project objectives, the project team will review site-specific data needs prior to starting work and, if needed, adapt the field procedures.

The scope of this TGI is specific to bedrock chip collection and description; it does not encompass the broader suite of tasks associated with bedrock drilling or well construction (see relevant SOP and TGIs, as needed). Note that bedrock drilling is often combined with related bedrock characterization techniques, including packer-testing, geophysical logging, FLUTE™ profiling and whole-core rock sampling. These tasks are outside of the scope of this TGI; however, if such additional work is part of the project scope, planning and sequencing of drilling tasks will consider the requirements of auxiliary tasks.

4 Personnel Qualifications

Arcadis field personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40- hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as specified in the Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed.

In addition, Arcadis field personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. The HASP and other documents will identify other training requirements or access control requirements.

Bedrock chip logging will only be performed by Arcadis personnel or authorized subcontractors with a bachelor's degree in geology or a geology-related discipline. Field personnel will complete training on this TGI in the office and/or in the field under the guidance of an experienced field geologist with at least 2 years of prior experience with bedrock core description.

Note that this TGI is written specifically for site characterization and remediation projects. When bedrock core samples are to be used for engineering purposes (e.g., foundation design, rock mechanics, design of excavation support), field staff will work under the direction of a geotechnical engineer.

5 Equipment List

Typically Provided by Geologist:

- Approved site-specific Health and Safety Plan (HASP)

- Approved site-specific field implementation plan (FIP)/work plan which will include boring location map and drilling plan
- Required PPE (see site-specific HASP)
- Handheld sieve
- Field logbook and/or boring logs
- Camera and/or smart device (phone or tablet)
- Pen knife (to test rock hardness)
- Munsell rock color chart
- Rock hammer
- Photoionization detector (PID) or Flame ionization detector (FID) (as appropriate, depending on site-specific constituents of concern)
- Air monitoring equipment (as required)
- Hand lens (optional)
- 10% Hydrochloric acid solution (appropriately labeled eyedropper for carbonate identification [optional])

6 Cautions

- **Review relevant guidance:** Utility avoidance, drilling, decontamination, management of investigation derived waste and related tasks will be completed in accordance with a project- specific FIP/work plan and/or applicable SOPs or TGIs.
- **Use a trusted, experienced driller:** The quality of bedrock borings often depends on the skill of the driller (e.g., at selecting the correct tooling, down-pressure and spin-rate for the type of rock and depth). An experienced driller is less likely to drill a borehole that is smooth, straight, and true. A quality borehole will result in better data quality if bedrock drilling tasks are combined with other related bedrock characterization techniques (e.g., downhole geophysics, packer testing, ect.)
- **Choose a clean water supply for drilling fluid:** Whether for mixing mud or used as dust suppression during air rotary drilling, water used for drilling will be of sufficient quality to meet project objectives. Testing of water supply will be considered.
- **Understand your driller’s plans for recirculation of drilling fluids:** Recirculation is common practice in when using mud rotary drilling methods, to limit generation of large quantities of investigation-derived waste (IDW). A mud mixture is pumped down the inside of the core barrel (or outer rods if using reverse circulation methods) to cool the bit and carry rock cuttings back to the surface. The cuttings and drilling fluids spill into a mud tub, often designed with several baffles to help cuttings fall out of suspension. This drilling fluids are then pumped back down the drill tooling, or recirculated, until the sediment load is too great, then drilling fluids must be replaced. Additionally, an inline desander/cyclone may be put in place upstream to the mud tub. The desander will help reduce sediment load significantly and cuttings can be deposited directly into a roll-off container. Recirculation can increase the risk of cross-contamination, so caution is needed. However, drilling without recirculation can quickly generate very large quantities of IDW and is often not practicable.

- **Avoid cross-contamination:** Bedrock drilling often involves creating long open boreholes that may, at least temporarily, penetrate confining beds or create artificial connections between fracture zones at different depths. If cross-contamination is a concern at a site, work will be planned to limit the length of open sections (e.g., by telescoping casing), and limit the duration that a borehole stands open. Field crews will stop-work if dense-non-aqueous phase liquid (DNAPL) is encountered (e.g., if sheens are observed on drilling return water).

7 Health and Safety Considerations

Conduct drilling and related tasks in accordance with a site-specific Health and Safety Plan (HASP). Review all site-specific and procedural hazards as they are provided in the HASP, and review Job Safety Analysis (JSA) documents in the field each day prior to beginning work. Appropriate personal protective equipment (PPE) will be worn at all times in line with the task and the site-specific HASP.

Use appropriate hand protection when conducting carbonate-rock test (using dilute acid) and hardness tests (using a penknife). If site- or client-specific health and safety requirements prohibit use of fixed/folding-blade knives, an alternative steel object (e.g., nail) may be substituted.

8 Procedure

Rotary drilling methods, including air rotary, mud rotary, and reverse circulation, are some of the most common methods for installation of bedrock monitoring wells. The nature of these drilling methods pulverizes the formation under the drill string resulting in drill cuttings consisting of fine pieces (i.e., chips) of the formation. Adequate characterization of drill cuttings ensures data quality objectives (e.g., accurate well placement) of the project are maintained. The procedures for collecting and characterizing chips samples are outlined below.

8.1 Chip Collection

Accurate chip logging requires the supervising geologist be at the rig at all times during drilling. Often, changes in lithology result in corresponding changes in drilling characteristics (e.g., drilling rate, feed pressure, and audible feedback from the drills string). Additionally, time lag for cuttings to reach the surface is typically minimal unless drilling to significant depths. Therefore, accurate formation logging (e.g., fracture sets resulting in secondary mineralization or changes in rock type) requires immediate access to cuttings in order to correlate variations in drilling characteristics with formation changes.

Chips will be collected as close to the discharge near the drill string (e.g., at the cyclone outlet, if applicable, or prior to drilling fluids entering the mud tub) as safely possible. Depending on the rig setup the driller may need to collect the sample. Chip collection will be done using a handheld sieve/strainer.

8.2 Chip Description

It is recommended that geological cross-sections, including surface geophysical results, be made available while drilling and geological interpretation be commenced while at the rig. Also, field staff will consult interpreted logs from adjacent boreholes, including borehole geophysical logs, if available.

In addition to the visual description of chip samples discussed below, the following details will be recorded in the geological log as the drilling progresses being sure to note both time of day and depth drill string at noted events:

- rate of penetration during full down force based on regular notation of depth and time of day at beginning and end of drill rod as well as several intermediate locations, particularly if conditions change rapidly;
- audible changes in drilling tone and audible volume;
- notable jumps or rapid increases in downward movement of drill string which may be indicative of voids, fractures, faults, flow tops, geologic contacts, etc.;
- indications of rock falls evidenced by ejection of rock samples of unusual size (not chips)
- dampness on samples,
- water inflows including an estimate of water produced – if significant inflow is found, stop drilling and have the driller conduct a test of the volume of water ejected by air lift after a pause of a noted amount of time under quiet conditions.
- odors and visual discolorations of samples
- PID/FID readings
- drilling problems

Perform geological logging on at least a one meter by one meter basis and record in the log as such. As a minimum, the following features must be logged:

| What to Record | How to Describe |
|------------------|--|
| Depth | Note top of an interval being described, relative to ground surface. Avoid referencing depths relative to the position in the drill string run. |
| Rock type | Describe based on observation. Use terminology consistent with local mapping, if available. If the specific type cannot be determined in the field, use a more general descriptor (e.g., metamorphic). |
| Color | Reference Munsell rock color chart. Describe matrix color and major clast color separately, if applicable. |
| Weathering state | General weathering descriptors (e.g., fresh or weathered) may be applicable based geologic environment. Small chip size can make weathering determination difficult |

Other observations may also be made, if appropriate to the rock type. Common supplemental observations include:

- Overburden-bedrock interface
- Top of competent/unweathered rock
- Depositional environment specific texture descriptions (e.g., volcanic glass, aphanitic or phaneritic textures, foliation, schistosity, gneissic banding, ect.)
- Grainsize, grain shape, etc. in clastic sediments such as sand and gravel.
- Full description of cohesive materials such as silt and clay

- Color
- Chemical alteration of rock minerals
- Veining, including nature of vein filling materials
- Secondary mineralization
- Evidence of any structural features such as slickensides, breccia, stylolites, and folds
- Effervesce, e.g., if testing for limestone or dolomite using a hydrochloric acid solution
- Observations of porosity, pitting, vugs, or cavities

Take photographs of chips regularly during drilling and as geologic conditions change. For clarity, it is recommended that chips be deposited on a clean white background with identifying descriptors (locations, depth, date, time, ect) written above the sample.

8.3 Contaminant Screening

Methods for screening for contamination while drilling depend on the nature of impacts suspected. As noted above, air-monitoring at the ground surface of the borehole, and continuous visual observation of the return water while drilling generally provide the first indication of an impact.

Specific procedures for screening chips will be identified in the project FIP/work plan. Common approaches include the following:

- Though field staff will NOT intentionally sniff the cuttings, obvious odors are sometimes useful indicators. Field descriptions of odors will be general, and not attempt to specify what contaminant it smells like.
- If screening core for volatile organic compounds (VOCs) with a photo-ionization detector (PID), focus stained cuttings.
- If NAPL is suspected (e.g., based on high PID hits, or sheens in the return water), one of several commercially available NAPL-detection kits (using hydrophobic dye) may be applied to the cuttings as a supplemental test.

As noted above, when NAPL is observed in a borehole, drilling will almost always stop to avoid dragging the impacts down—drilling deeper will occur ONLY when necessitated by the project objectives, and ONLY after consulting project leadership.

9 Waste Management

Bedrock drilling may generate several types of investigation derived waste (IDW):

- Bedrock drilling typically generates substantial quantities of drilling fluid. It is typically a mixture of water and suspended fine sediment. In most cases, this is drummed. For large jobs, roll-off or “sludge” boxes may be more economical.
- Solid rock cuttings also accumulate in the mud tub. These are typically shoveled into drums.
- Other waste streams include decontamination liquids, and disposable materials (well material packages, personal protective equipment [PPE], etc.).

Waste will be managed in accordance with the TGI – Investigation-Derived Waste Handling and Storage, the procedures identified in the FIP or QAPP as well as state-, federal- or client-specific requirements. Be certain that all IDW will be placed in clearly labeled, appropriate containers and documented in the field logbook.

10 Data Recording and Management

Digital data collection is the Arcadis standard using available FieldNow® applications that enable real-time, paperless data collection, entry, and automated reporting. Paper forms should only be used as backup to FieldNow® digital data collection and/or as necessary to collect data not captured by available FieldNow® applications. The Field Now® digital form applications follow a standardized approach, correlate to most TGIs and are available to all projects accessible with a PC or capable mobile device. Once the digital forms are saved within FieldNow®, the data is instantly available for review on a web interface. This facilitates review by project management team members and SMEs enabling error or anomalous data detection for correction while the staff are still in the field. Continual improvements of FieldNow® applications are ongoing, and revisions are made as necessary in response to feedback from users and subject matter experts.

Records generated as a result of this TGI will be controlled and maintained in the project record files in accordance with project requirements as outlined in the FIP/work plan and/or QAPP.

Field forms, logs/notes (including daily field and relevant calibration logs), and digital records will be maintained by the field team lead.

Records will be transmitted to the Arcadis Project Manager and/or Task Manager, as appropriate, at the end of each day or as specified in the FIP/work plan.

Electronic data files will be sent to the project team and uploaded to the electronic project folder daily or as specified in the FIP/work plan.

Management of the original documents from the field will be completed in accordance with the site-specific QAPP

11 Quality Assurance

Bedrock chip descriptions will be completed only by appropriately trained personnel, and descriptions will be reviewed by an experienced field geologist for content, format and consistency. Edited boring logs should be reviewed by the original author to assure that content has not changed.

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