

**Sampling and Analysis Plan Addendum**  
**Town of Coupeville**  
**Ft Casey Water Treatment Plant**  
**Performance Monitoring**

Whidbey Island

Washington

March 2024

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Worksheet #1—Title and Approval Page

**Sampling and Analysis Plan Addendum**  
**Town of Coupeville**  
**Performance Monitoring**

Whidbey Island  
Washington

March 2024

Prepared for **Town of Coupeville**  
by DCG/Watershed, Inc.  
Freeland, Washington  
Cooperative Agreement Number N44255-23-2-2400



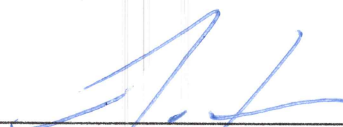
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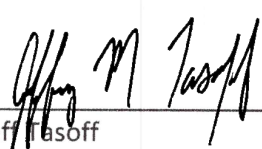
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Janice Horton  
Naval Facilities Engineering Systems Command Northwest  
Remedial Project Manager



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Joe Grogan  
Town of Coupeville  
Public Works Director



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# Executive Summary

The Town of Coupeville (Town) and the Department of the Navy (Navy) are working cooperatively to address drinking water supply and quality issues related to contamination of groundwater supply wells from historical releases of per- and polyfluoroalkyl substances (PFAS) at the Naval Air Station Whidbey Island (NASWI) Outlying Landing Field (OLF) in Coupeville, Washington. PFAS present in groundwater at the OLF are thought to be associated with aqueous film forming foam (AFFF) used for firefighting at Navy installations, although there is no historical documentation of a release of AFFF at OLF Coupeville.

A Sampling and Analysis Plan (SAP) was prepared under the previous Cooperative Agreement N44255-21-2-0001, detailing sampling protocols for performance monitoring of PFAS detected in drinking water supply wells, consistent with the Town's approved and adopted 2009-2014 Water System Plan (WSP) and 2017 Limited WSP Update at the Fort Casey Water Treatment Plant (FCWTP). The objectives of that original SAP were to verify that the granular activated carbon (GAC) treatment system is reducing PFOA and PFOS concentrations to levels below the project action limits (PALs) and to document PFAS concentration in backwash water entering an existing onsite backwash pond. Sampling at FCWTP was then conducted in accordance with the original SAP between May 2021 to November 2023 to meet these objectives.

In February 2023, the Town and the Navy entered into a new Cooperative Agreement, N44255-23-2-2400, to continue operations and maintenance (O&M) on the PFAS treatment process in the Town's drinking water treatment plant, including PFAS performance monitoring. This SAP Addendum will follow the Town's current 2023 WSP and the objectives as noted above.

DCG/Watershed, Inc. (DCG/W) was contracted by the Town to prepare this SAP Addendum in accordance with the Navy's Uniform Federal Policy-SAP policy guidance to help ensure that environmental data collected are scientifically sound, of known and documented quality, and suitable for intended uses. DCG/W is not performing any monitoring detailed in this SAP Addendum but was contracted to prepare the sampling approach to ensure consistency with previous sampling events at FCWTP. The Town will be performing sampling, testing, and monitoring detailed in this SAP Addendum.

This SAP Addendum was developed in accordance with the following guidance documents:

- *Guidance for Quality Assurance Project Plans* (USEPA, 2002)
- *2021 – 2023 Water Treatment Plant Operations and Maintenance Report* (DCG/W, 2023)
- *Uniform Federal Policy for Quality Assurance Project Plans Workbook* (USEPA, 2012)
- *Quality Systems Manual for Environmental Laboratories/Version 5.4* (DoD/DoE, 2021)
- *Guidance on Systematic Planning Using the Data Quality Objectives Process* (USEPA, 2006)
- *Interim Per- and Polyfluoroalkyl Substances (PFAS) Site Guidance for NAVFAC Remedial Project Managers (RPMS)/November 2020 Update* (Navy, 2020)

This SAP Addendum includes 31 updated worksheets, reflecting general process and staff updates since the original SAP as well as changes to applicable USEPA Methods. For worksheets not included in this SAP Addendum, the version in the original SAP then still applies.

Updated tables are embedded within the worksheets. Updated figures are included at the end of the document. Updated field standard operating procedures (FSOPs) are included in **Appendix A**. An updated Department of Defense Environmental Laboratory Accreditation Program Accreditation Letter is included in **Appendix B**. Updated SGS Laboratory Standard Operating Procedures (LSOPs) are included in **Appendix C**.

The laboratory information cited in this SAP Addendum is specific to SGS Laboratory (for PFAS analysis). If additional laboratory services are requested requiring modification to this SAP Addendum, revised SAP worksheets and appendixes will be submitted to the Navy for approval.



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### **Tables**

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- 1 Location Map
- 2 Proposed FCWTP Performance Monitoring Schedule and Decision Flowchart
- 3 Overall Sample Locations
- 4 Sample Location SA-001
- 5 Sample Locations SA-100, SA-111, SA-112
- 6 Sample Location SA-002
- 7 Sample Locations SA-103, SA-107, SA-203, SA-207
- 8 Sample Locations SA-100, SA-111, SA-112
- 9 Sample Locations SA-200, SA-211, SA-212

### **Appendixes**

- A APTIM Field Standard Operating Procedures
- B Department of Defense Environmental Laboratory Accreditation Program Accreditation Letters
- C SGS Laboratory Standard Operating Procedures

# Acronyms and Abbreviations

±	plus or minus
%	percent
>	more than
≤	less than or equal to
°C	degree Celsius
µg/L	microgram per liter
4:2FTS	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid
6:2FTS	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid
8:2FTS	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid
AC	activated carbon
ADONA	4,8-dioxa-3H-perfluorononanoic acid
AFFF	aqueous film-forming foam
CA	corrective action
CAS	Chemical Abstract Service
CCV	continuing calibration verification
CH2M	CH2M HILL, Inc.
CoA	Certificate of Analysis
DCG/W	DCG/Watershed, Inc.
DL	detection limit
DoD	Department of Defense
DoE	Department of Energy
DOH	Department of Health
DQI	data quality indicator
DV	data validation
DW	drinking water
DWTPWW	drinking water treatment process wash water
EDD	electronic data deliverable
ELAP	Environmental Laboratory Accreditation Program
EPA	Environmental Protection Agency
Ext.	Extension
FCWTP	Fort Casey Water Treatment Plant
FD	field duplicate
FTL	Field Team Leader
FRB	Field Reagent Blank
GAC	granular activated carbon
H&S	health and safety
HFPO-DA	Hexafluoropropylene oxide dimer acid
HQ	hazard quotient

ICAL	initial calibration
ID	identification
LCS	laboratory control sample
LCL	lower confidence limit
LFB	laboratory fortified blank
LFSM	laboratory fortified matrix structure
LFSMD	laboratory fortified matrix structure duplicate
LHA	Lifetime Health Advisory
LIMS	laboratory information management system
LOD	limit of detection
LOQ	limit of quantitation
mL	milliliter(s)
MPC	measurement performance criteria
MS	matrix spike
MSD	matrix spike duplicate
N/A	not applicable
NAS	Naval Air Station
NASWI	Naval Air Station Whidbey Island
NAVFAC	Naval Facilities Engineering Systems Command
Navy	Department of the Navy
NEtFOSAA	N-ethyl perfluorooctanesulfonamidoacetic acid
NMeFOSAA	N-methyl perfluorooctanesulfonamidoacetic acid
NFDHA	Nonafluoro-3,6-dioxaheptanoic acid
NWSI	Navy Water Supply Improvements
OLF	Outlying Landing Field
O&M	Operation and maintenance
PAL	project action limit
PFAS	per- and polyfluoroalkyl substances
PFBA	perfluorobutanoic acid
PFBS	perfluorobutanesulfonic acid
PFDA	perfluorodecanoic acid
PFDoA	perfluorododecanoic acid
PFEESA	perfluoro (2-ethoxyethane) sulfonic acid
PFHpA	perfluoroheptanoic acid
PFHpS	perfluoroheptanesulfonic acid
PFHxA	perfluoroheptanoic acid
PFHxS	perfluorohexanesulfonic acid
PFMBA	perfluoro-4-methoxybutanoic acid
PFMPA	perfluoro-3-methoxypropanoic acid
PFNA	perfluorononanoic acid
PFOA	perfluorooctanoic acid
PFOS	perfluorooctanesulfonic acid
PFPeA	perfluoropentanoic acid
PFPeS	perfluoropentanesulfonic acid
PFTA	perfluorotetradecanoic acid

PFTrDA	perfluorotridecanoic acid
PFUnA	perfluoroundecanoic acid
PM	Project Manager
POC	point of contact
PQL	project quantitation limit
PQO	project quality objective
QA	quality assurance
QAO	Quality Assurance Officer
QC	quality control
QSM	Quality Systems Manual
RPD	relative percent difference
RPM	Remedial Project Manager
RSL	regional screening level
SAP	Sampling and Analysis Plan
SOP	standard operating procedure
STC	Senior Technical Consultant
TBD	to be determined
Town	Town of Coupeville
UCL	upper confidence limit
USEPA	United States Environmental Protection Agency
WA	Washington
WSP	Water System Plan

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## Worksheet #2—SAP Identifying Information

**Site Name/Number:** Fort Casey Water Treatment Plant (FCWTP), Coupeville, Washington

**Operable Unit/Solid Waste Management Unit:** Not applicable (N/A)

**Cooperator Name:** Town of Coupeville

**Cooperative Agreement #:** N44255-23-2-2400

**Contract Title:** Environmental Service Cooperative Agreement Between the US Department of the Navy and the Town of Coupeville

**Task Order Number:** N/A

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the following guidance documents:
  - *Guidance for Quality Assurance Project Plans* (USEPA, 2002)
  - *Uniform Federal Policy for Quality Assurance Project Plans* (USEPA, 2005)
  - *Quality Systems Manual for Environmental Laboratories/Version 5.4* (DoD/DoE, 2021)
  - *Guidance on Systematic Planning Using the Data Quality Objectives Process* (USEPA, 2006)
  - *Interim Per- and Polyfluoroalkyl Substances (PFAS) Site Guidance for NAVFAC Remedial Project Managers (RPMS)/November 2020 Update* (Navy, 2020)
2. Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act
3. This document is a project-specific SAP. The approval entities are Naval Facilities Engineering Systems Command (NAVFAC) Northwest Remedial Project Manager (RPM) and the NAVFAC Atlantic Quality Assurance Officer.
4. List dates of scoping sessions that were held: No scoping session was held for this SAP.
5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

<i>Sampling and Analysis Plan Site Inspection for Perfluorinated Compounds in Groundwater Outlying Landing Field Coupeville</i>	January 2017
<i>Final Sampling and Analysis Plan Investigation of Perfluorinated Compounds in Drinking Water Outlying Landing Field Coupeville</i>	January 2017
<i>Final Sampling and Analysis Plan Monitoring of Per- and Polyfluoroalkyl Substances in Drinking Water Ault Field and Outlying Landing Field Coupeville</i>	October 2017
<i>Final Sampling and Analysis Plan Monitoring Well Installation, Aquifer Testing, Drinking Water Sampling, and Groundwater Sampling Outlying Landing Field Coupeville</i>	December 2017
<i>Final Sampling and Analysis Plan Time-Critical Removal Action, Point-of-Use Treatment System Monitoring Ault Field and Outlying Landing Field Coupeville</i>	April 2018
<i>Sampling and Analysis Plan Town of Coupeville Water Supply Improvements Performance Monitoring</i>	July 2019
<i>Sampling and Analysis Plan Town of Coupeville Water Supply Improvements Performance Monitoring</i>	October 2021

## Worksheet #2—SAP Identifying Information (Continued)

6. List organizational partners (stakeholders) and connection with lead organization:
  - NAVFAC Atlantic – Quality Assurance Officer, TBD
  - NAVFAC Northwest – RPM, Kendra Clubb
  - NAVFAC Northwest – RPM, Janice Horton
  - Town of Coupeville – Mayor, Molly Hughes
  - Town of Coupeville – Public Works Director, Joe Grogan
7. Lead organizations (Cooperative Agreement):
  - Department of the Navy (Navy)
  - Town of Coupeville (Town)
8. If any required SAP elements or required information are not applicable (N/A) to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion as follows:
  - Crosswalk table is excluded because all required information is provided in this SAP.



### Worksheet #3—Distribution List

<b>Name of SAP Recipients</b>	<b>Title/Role</b>	<b>Organization</b>	<b>Telephone Number</b>	<b>Email Address</b>
Kendra Clubb	NAVFAC NW RPM	NAVFAC Northwest	(509) 999-6843	kendra.r.clubb.civ@us.navy.mil
Janice Horton	NAVFAC NW RPM	NAVFAC Northwest	(360) 556-0621	janice.l.horton5.civ@us.navy.mil
Heather Wandrey	Laboratory PM	SGS	(407) 425-6700	heather.wandrey@sgs.com
Svetlana Izosimova	Laboratory Quality Assurance Officer	SGS	(407) 425-6700	svetlana.izosimova@sgs.com
Joe Grogan	Public Works Director/ Project Manager (PM)	Town of Coupeville	(360) 678-4461 ext. 110	publicworks@townofcoupeville.org
Jimmy Wadlington	Field Team Leader (FTL)	Town of Coupeville	(360) 678-4461 ext. 113	publicworks5@townofcoupeville.org
Pei Geng	Data Validator	LDC	(760) 827-1100 ext. 141	pgeng@lab-data.com
Jeff Tasoff	Principal Civil Engineer/ Senior Technical Consultant (STC)	DCG/W	(206) 523-0024 ext. 203	jeff@dcgwatershed.com
Carly McArdle	Civil Engineer	DCG/W	(360) 331-4131 ext. 209	carly@dcgwatershed.com

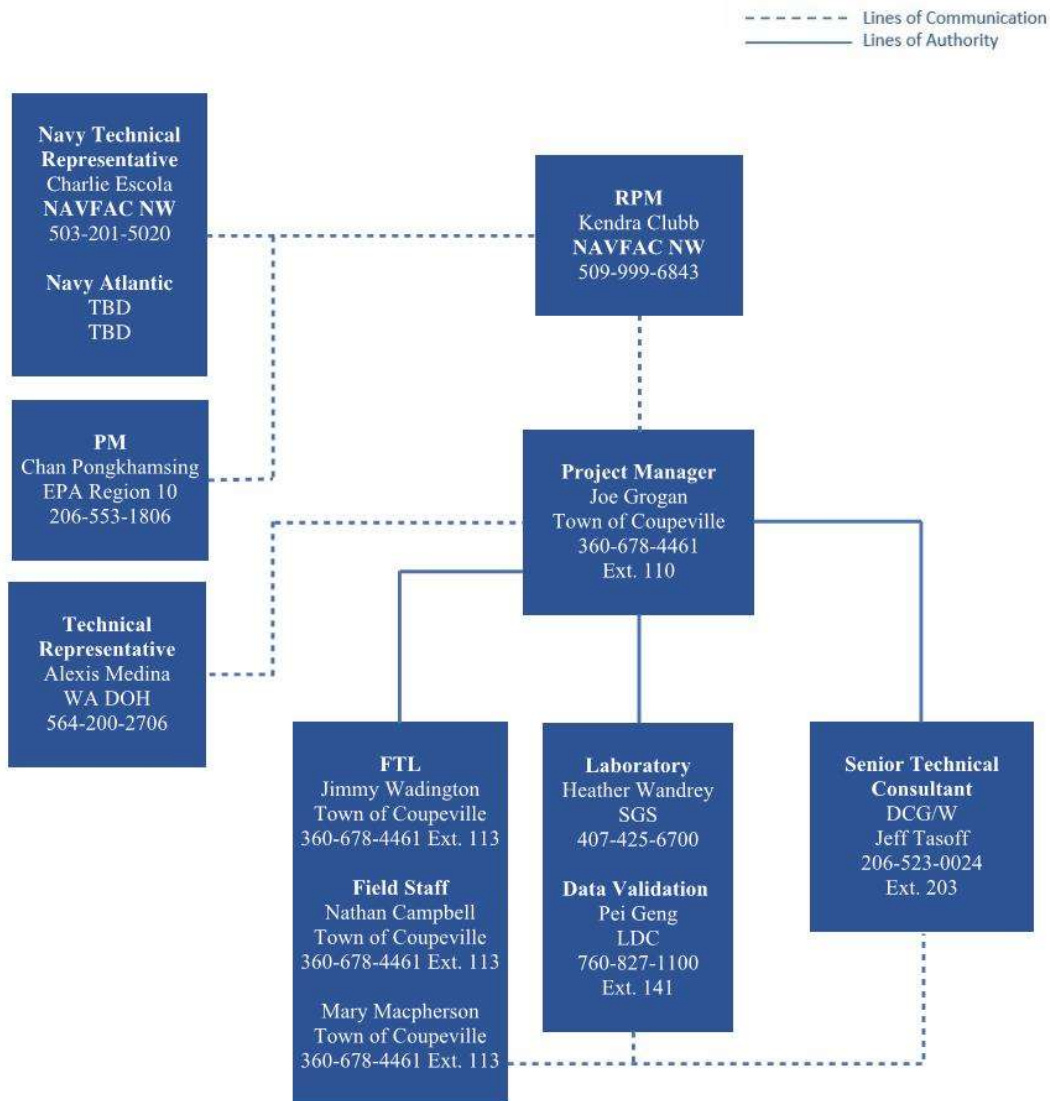
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## Worksheet #4—Project Personnel Sign-off Sheet

Name	Organization/Title/Role	Telephone Number	Signature/Email receipt	Date SAP Read
Heather Wandrey	SGS/Laboratory PM	(407) 425-6700		
Svetlana Izosimova	SGS/Laboratory Quality Assurance Officer	(407) 425-6700		
Joe Grogan	Town/Public Works Director and PM	(360) 678-4461 ext. 110		
Jimmy Wadlington	Town/FTL	(360) 678-4461 ext. 113		
Pei Geng	LDC/Data Validator	(760) 827-1100 ext. 141		
Jeff Tasoff	DCG/W/Principal Civil Engineer and STC	(206) 523-0024 ext. 203		

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## Worksheet #5—Project Organization Chart



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## Worksheet #6—Communication Pathways

Communication Drivers	Responsible Entity	Name	Email and Phone Number	Procedure
Communication with Town PM and NAVFAC Atlantic QAO	RPM	Kendra Clubb  Janice Horton	<a href="mailto:kendra.r.clubb.civ@us.navy.mil">kendra.r.clubb.civ@us.navy.mil</a> (509) 999-6843  <a href="mailto:janice.l.horton5.civ@us.navy.mil">janice.l.horton5.civ@us.navy.mil</a> (360) 556-0621	Primary POC for Navy. Town PM will notify RPM by email or telephone call within 24 hours for field changes affecting the scope or implementation of the SAP.
Communication regarding overall project status and implementation, and primary POC with RPMs and project team	Town	Joe Grogan	<a href="mailto:publicworks@townofcoupeville.org">publicworks@townofcoupeville.org</a> (360) 678-4461 ext. 110	Oversees project. POC for FTL and STC. Will ensure SAP requirements are met by field staff for the duration of the project. If field changes occur, will work with the RPM to communicate in-field changes to the team by email within 24 hours. All data results will be communicated to the project team following data receipt and review. All information and materials about the project will be forwarded to the Navy RPM, as necessary.

Communication Drivers	Responsible Entity	Name	Email and Phone Number	Procedure
<p>Analytical corrective actions (CAs)</p> <p>Data tracking from field collection to database upload</p> <p>Release of analytical data</p> <p>Communications regarding SAP changes</p>	<p>Town</p>	<p>Joe Grogan</p>	<p><a href="mailto:publicworks@townofcoupeville.org">publicworks@townofcoupeville.org</a></p> <p>(360) 678-4461 ext. 110</p>	<p>Any CAs for analytical issues will be determined by the FTL and/or the STC and reported to the Town PM within 4 hours. The Town PM will notify the Navy RPM of any field issues that would negatively affect the schedule or the ability to meet project data quality objectives.</p> <p>No analytical data can be released until the Town PM reviews and approves the data. The PM will review analytical results within 24 hours of receipt for release to the project team. The Town will inform the Navy QAO of any laboratory issues that would prevent the project from meeting project quality objectives (PQOs) or would cause significant delay in project schedule.</p> <p>Changes to the project that would prompt a SAP change that would require Navy QAO approval include: the addition of an analytical suite not previously included in the SAP, the addition of an environmental matrix not previously included in the SAP, laboratory accreditation to a new USEPA Method version, inclusion of a new laboratory into the SAP, or updates to the Conceptual Site Model (CSM) that prompt new DQO's. Updated laboratory LOQ, LOD, and DL Values will not prompt a SAP update for Navy QAO approval unless those updates negatively impact the ability to meet project objectives.</p>



## Worksheet #6—Communication Pathways (Continued)

Communication Drivers	Responsible Entity	Name	Email and Phone Number	Procedure
Reporting laboratory data quality issues	SGS Laboratory Quality Assurance Officer	Svetlana Izosimova	<a href="mailto:svetlana.izosimova@sgs.com">svetlana.izosimova@sgs.com</a> (407) 425-6700	All quality assurance (QA)/quality control (QC) issues with project field samples will be reported within 2 days to the Town PM by the laboratory.
Analytical corrective actions (CAs) Work plan changes in field Field changes/field progress reports	Town	Jimmy Wadlington	<a href="mailto:publicworks5@townofcoupeville.org">publicworks5@townofcoupeville.org</a> (360) 678-4461 ext. 113	Tracks data from sample collection through database upload daily. Any CAs for analytical issues will be determined by the FTL and/or the STC and reported to the Town PM within 4 hours. Deviations from the work plan will be made only with approval from the Town PM and STC. Documentation of field activities and work plan deviations in the field logbooks. Provide daily progress reports to Town PM. The PM will communicate changes to the RPM.
Reporting data quality issues	LDC Data Validator	Pei Geng	<a href="mailto:pgeng@lab-data.com">pgeng@lab-data.com</a> (760) 827-1100 ext. 141	The data validator reviews and qualifies analytical data as necessary. The data, along with a validation narrative, are returned to the Town PM within 7 calendar days.
Field CAs	Town	Jimmy Wadlington	<a href="mailto:publicworks5@townofcoupeville.org">publicworks5@townofcoupeville.org</a> (360) 678-4461 ext. 113	Field issues requiring CA will be determined by the Town FTL and reported to the Town PM; the Town FTL will ensure SAP requirements are met by field staff for the duration of the project. The FTL will notify the Town PM via phone of any need for CA within 4 hours.

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## Worksheet #7—Personnel Responsibilities Table

Name	Title/Role	Organizational Affiliation	Responsibilities
Kendra Clubb/Janice Horton	RPM	NAVFAC Northwest	Oversees project for Navy.
Svetlana Izosimova	Laboratory QAO	SGS	Responsible for audits, CA, and checks of QA performance within the laboratory.
Pei Geng	Data Validator	LDC	Validates laboratory data from an analytical standpoint prior to data use.
Joe Grogan	Public Works Director/PM	Town of Coupeville	Creates SOPs and oversees sampling activities. Communicates all potential changes to SOPs or SAP to Navy RPM.
Jimmy Wadlington	FTL	Town of Coupeville	Coordinates all field activities and sampling.
Jeff Tasoff	Principal Civil Engineer/STC	DCG/W	Technical support for SAP and sampling reporting.
Carly McArdle	Civil Engineer	DCG/W	Technical support for SAP and sampling reporting.

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## Worksheet #9—Project Scoping Session Participants Sheet

<b>Project Name:</b> Town of Coupeville Navy Water Supply Improvements <b>Projected Date(s) of Sampling:</b> Quarterly, beginning in February 2024 <b>PM:</b> Joe Grogan		<b>Site Name:</b> Fort Casey Water Treatment Plant (FCWTP) <b>Site Location:</b> Coupeville, Washington		
<b>Dates of Sessions:</b> N/A <b>Scoping Session Purpose:</b> N/A				
Name	Title/Project Role	Affiliation	Phone #	Email Address

### Comments

- There was no scoping session for this SAP.
- This Sampling and Analysis Plan Addendum was prepared in conformance with the requirements listed in the scope of work in Cooperative Agreement N44255-23-2-2400.

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## Worksheet #10— Conceptual Site Model

### Background

The Town of Coupeville (Town) is located on Whidbey Island in Island County, Washington, approximately 40 miles north of Seattle. The Town and the site layout are shown in **Figure 1**. A description and background summary of the FCWTP associated public and private wells, and the PFAS-impacted groundwater source(s) are presented in this worksheet and **Table 10-1**.

### PFAS Background and Investigation

The following is a summary of the off-Base site investigation activities completed to date.

In 2016, the USEPA established a Lifetime Health Advisory for PFOA and PFOS exposure in drinking water at 70 parts per trillion (ppt), individually or combined. This Lifetime Health Advisory is based on concerns over potential adverse health effects and is intended to provide a margin of protection against cumulative adverse lifetime health effects resulting from exposure to PFOS and PFOA in drinking water.

In September 2016, the Navy conducted on-Base drinking water sampling at OLF Coupeville. PFOA was detected in one on-Base drinking water well at OLF Coupeville below 70 ppt, which indicated a potential previous release of AFFF near Building 2807. Another on-Base drinking water well was sampled (Building 11), but PFAS was not detected. At the time, no previous groundwater investigations had been conducted at OLF Coupeville.

As a result of the on-base drinking water detection and potential on-base release of AFFF, in November 2016, the Navy initiated off-Base drinking water sampling for PFAS. The sampling area was established 1 mile in the estimated direction of groundwater flow from Building 2807. In February 2017, the Navy expanded the off-Base drinking water sampling further to the south of OLF Coupeville based on detections of PFOA and/or PFOS above 70 ppt in the 2016 initial sampling area (CH2M, 2017c). The Navy sampled 118 drinking water wells during this effort. Wells sampled included private residential drinking water wells and the Fort Casey Wellfield which supplies municipal drinking water to the Town of Coupeville. The Fort Casey Wellfield consists of three active water supply wells including the main supply well, Keystone Well 1-08, located just west of the OLF. The results of the sampling efforts indicated that PFOS and PFOA, individually or combined, were above 70 ppt in 8 private drinking water wells that supply 11 residences near the OLF. In addition, PFOA was detected in Keystone Well 1-08 at concentrations above 70 ppt in September of 2018. All other samples at the Keystone Well 1-08 taken by the Navy have been below 70 ppt. In October 2017, the Navy started biannual drinking water sampling for drinking water wells where PFAS were previously detected and for drinking water wells at properties located adjacent to properties with drinking water wells with detections of PFOA and PFOS, individually or combined, above 70 ppt.

The Town has increasingly focused on the Fort Casey Wellfield, including Keystone Well 1-08, as its primary municipal drinking water supply source. Groundwater sourced from the Fort Casey Wellfield is treated at the FCWTP before being distributed through the Town water system as municipal drinking water. The treatment processes did not provide removal of PFOS and PFOA prior to 2019; as it was targeted only at providing basic disinfection and improving aesthetic water quality through removal of naturally-occurring dissolved iron (Fe), manganese (Mn), and hydrogen sulfide (H<sub>2</sub>S). The oxidation filtration backwash was discharged to an infiltration pond adjacent to the FCWTP.

In 2018, the Navy identified the following as the most protective and efficient long-term solution: 1) adding further treatment for PFAS at the FCWTP and 2) connecting the 11 residences currently supplied by private wells above 70 ppt PFOS and/or PFOA to the Town's municipal drinking water system. Design was completed in 2019 by the Navy's engineer, CH2M Hill, for the PFAS treatment improvements and waterline extension (CH2M, 2019a). Two new GAC treatment trains were installed, one brought online in July 2019 and the second in September 2019, which operate in parallel with provisions for a future third train to support system growth (APTIM, 2021b).

## Worksheet #10—Conceptual Site Model (Continued)

With these system modifications in place, samples were scheduled for 1 day, 1 week, and at months 1, 2, 3, 6, 9, 12, 15, and 18 (CH2M, 2019b). Results show that treatment successfully maintains combined PFOS and PFOA concentrations below 70 ppt (APTIM, 2020; APTIM, 2021a; DCG/W, 2023).

In March 2023 the lead GAC tanks underwent media changeout due to concentrations nearing the midpoint PAL as defined in *Sampling and Analysis Plan Town of Coupeville Water Supply Improvements Performance Monitoring* (DCG/W, 2021).

As of January 2024, the Navy has connected 7 of the 11 properties with detections of PFOA and/or PFOS above 70 ppt to the Town’s water system. At this time, the remaining four homes receive alternative water.

**Table 10-1. Fort Casey Water Treatment Plant (FCWTP) Description and Background**  
*Coupeville, Washington*

<b>Site Name</b>	Fort Casey Water Treatment Plant (FCWTP), Coupeville, Washington ( <b>Figure 1</b> )
<b>Study Area Description</b>	The facility to be monitored includes the FCWTP and associated Navy Water Supply Improvements, as described in the <i>Project and Design Development Report Town of Coupeville Navy Water Supply Improvements</i> (CH2M, 2019b). Since installation, operations and maintenance activities have been summarized in three annual operations and maintenance reports (APTIM, 2020; APTIM, 2021a; DCG/W, 2023).
<b>Site History</b>	There is no historical documentation of a release of AFFF at OLF Coupeville. However, the presence of PFAS in groundwater at the OLF are thought to be associated with aqueous film forming foam (AFFF) used for firefighting at Navy installations.
<b>Current Use</b>	The area surrounding the FCWTP and OLF Coupeville is a low-density residential area. Drinking water supply for the area has been supplied by private or municipal drinking water wells. The Town’s drinking water supply is sourced from two groups of wells: the Fort Casey Wellfield which are located approximately 3 miles south of Town, and the In-Town wells located closer to the town center. The In-Town wells are susceptible to sea water intrusion given the local geology and their proximity to Puget Sound. As a result, the Town has increasingly focused on the Fort Casey Wellfield as its primary municipal drinking water supply source.
<b>Site Conditions</b>	A new GAC treatment system was incorporated into the existing treatment plant. See the FCWTP-NAWSI design, provided separately in the <i>Project and Design Development Report Town of Coupeville Navy Water Supply Improvements</i> for more information on the water treatment plant (CH2M, 2019a) and Construction Completion Report (APTIM, 2021b).
<b>Nature and Extent</b>	Performance monitoring in this SAP Addendum addresses PFOA and PFOS in the FCWTP. Ongoing monitoring has been summarized in the three annual operations and maintenance reports (APTIM, 2020; APTIM, 2021a; DCG/W, 2023).  The Navy is conducting a Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) investigation for PFAS at/near OLF Coupeville. The results for associated investigations may be found in separate documents.
<b>Potential Receptors/ Exposure Routes</b>	Current and future off-Base residents: Ingestion of PFAS in public and private drinking water sourced from groundwater.



## Worksheet #10—Conceptual Site Model (Continued)

### Constituents of Potential Concern

The constituents of potential concern are the 29 PFAS as listed in UCMR 5 by EPA Methods 533 and Method 537.1 Version 2 as follows:

#### EPA Method 533

1. Perfluorooctanoic acid (PFOA) (Chemical Abstract Service [CAS] No. 335-67-1)
2. Perfluorooctanesulfonic acid (PFOS) (CAS No. 1763-23-1)
3. Perfluorobutanesulfonic acid (PFBS) (CAS No. 375-73-5)
4. Perfluorodecanoic acid (PFDA) (CAS No. 335-76-2)
5. Perfluorododecanoic acid (PFDoA) (CAS No. 307-55-1)
6. Perfluoroheptanoic acid (PFHpA) (CAS No. 375-85-9)
7. Perfluorohexanesulfonic acid (PFHxS) (CAS No. 355-46-4)
8. Perfluorohexanoic acid (PFHxA) (CAS No. 307-24-4)
9. Perfluorononanoic acid (PFNA) (CAS No. 375-95-1)
10. Perfluoroundecanoic acid (PFUnA) (CAS No. 2058-94-8)
11. 4,8-dioxa-3H-perfluorononanoic acid (ADONA) (CAS No. 919005-14-4)
12. 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS) (CAS No. 756426-58-1)
13. Hexafluoropropylene oxide dimer acid (HFPO-DA) (CAS No. 13252-13-6)
14. 1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2FTS) (CAS No. 757124-72-4)
15. 1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2FTS) (CAS No. 27619-97-2)
16. 1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2FTS) (CAS No. 39108-34-4)
17. Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) (CAS No. 151772-58-6)
18. Perfluorobutanoic acid (PFBA) (CAS No. 375-22-4)
19. Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA) (CAS No. 113507-82-7)
20. Perfluoroheptanesulfonic acid (PFHpS) (CAS No. 375-92-8)
21. Perfluoro-4-methoxybutanoic acid (PFMBA) (CAS No. 863090-89-5)
22. Perfluoro-3-methoxypropanoic acid (PFMPA) (CAS No. 377-73-1)
23. Perfluoropentanoic acid (PFPeA) (CAS No. 2706-90-3)
24. Perfluoropentanesulfonic acid (PFPeS) (CAS No. 2706-91-4)
25. 11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS) (CAS No. 763051-92-9)

#### EPA Method 537.1 Version 2

1. N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA) (CAS No. 2991-50-6)
2. N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA) (CAS No. 2355-31-9)
3. Perfluorotridecanoic acid (PFTTrDA) (CAS No. 72629-94-8)
4. Perfluorotetradecanoic acid (PFTA) (CAS No. 376-06-7)

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## Worksheet #11—Project Quality Objectives/Systematic Planning Process Statements

### Problem Statement and Objectives

PFOA and PFOS have been detected in the Town’s drinking water. The project objectives, environmental questions, general investigation approach, and project quality objectives (PQOs) are presented in **Table 11-1**. The monitoring flow chart for the FCWTP is included on **Figure 2**. The FCWTP design is provided separately in the *Project and Design Development Report Town of Coupeville Navy Water Supply Improvements* (CH2M, 2019b). The detailed sampling approach, including number of samples, sampling frequency, and analyses are included in **Worksheet #17**. The sampling rationale is outlined in **Worksheets #17** and **#18**.

**Table 11-1. Project Quality Objectives**  
*Fort Casey WTP, Coupeville, Washington*

Project Objectives	Environmental Question(s)	General Investigation Approach	PQOs
<ul style="list-style-type: none"> <li>Verify and document GAC treatment effectiveness.</li> <li>Document PFAS concentrations in backwash water routed to the existing onsite pond.</li> <li>Identify/predict GAC media change-out</li> </ul>	<ul style="list-style-type: none"> <li>Is the FCWTP reducing PFOA and PFOS concentrations in WTP effluent to levels less than the effluent Project Action Limit (PAL)<sup>a</sup>?</li> <li>Are the midpoint samples above the midpoint PAL<sup>a</sup> such that GAC media replacement is necessary?</li> <li>Is FCWTP maintenance required to maintain continuously effective treatment?</li> <li>What are PFAS concentrations in backwash water to the pond?</li> </ul>	<ul style="list-style-type: none"> <li>Conduct quarterly water sampling from the three influent locations (i.e., one from FCWTP influent, one from 100-series influent, and one from 200-series influent), two midpoint locations (i.e., one from 100-series and 200-series), four 50% media locations (i.e., one from each tank in the 100-series and 200-series), and three effluent locations (i.e., one from FCWTP effluent, one from 100-series effluent, and one from 200-series effluent) at the FCWTP as specified in <b>Figures 3 through 9</b>.</li> <li>Following installation of fresh GAC media, collect two composite backwash samples, one for GAC backwash flow, and one for media filter backwash flow, from media filter and GAC backwash locations at the FCWTP as specified in <b>Figures 3 through 9</b>. Prepare separate composite samples for each by collecting backwash flow samples of equal volume every 5 minutes during backwash, discarding any samples that contain visible GAC fines or cloudiness within the sample volume, and mixing all remaining samples together.</li> <li>Analyze the water samples for the 29 PFAS constituents as listed in UCMR 5 by EPA Methods 533 and Method 537.1 Version 2 as listed in Worksheet #15. Use the PFOA and PFOS concentrations for decisions specified in <b>Figure 2</b>.</li> </ul>	<ul style="list-style-type: none"> <li>FCWTP monitoring, performance evaluations, and maintenance (i.e., replace all GAC media if effluent samples exceed the effluent PAL<sup>a,b</sup> OR replace lead train’s GAC media if midpoint samples exceed the midpoint PAL<sup>a</sup>).</li> <li>If concentrations of PFOA and/or PFOS in backwash water exceed the effluent PAL<sup>a</sup>, then Town and Navy will discuss measures to address this transport pathway.</li> <li>If concentrations of PFOA and/or PFOS in backwash water do not exceed the effluent PAL<sup>a</sup>, then no additional measures are needed to address this transport pathway under current known requirements.</li> </ul>

<sup>a</sup> Effluent and midpoint PALs are defined in **Worksheet #11**.

<sup>b</sup> It is extremely unlikely that the effluent will exceed 70 ppt PFOA/PFOS given the current influent PFOA/PFOS concentration and quarterly midpoint monitoring. However, if it did exceed, the source wells’ contribution would be adjusted to reduce levels below the effluent PAL and/or emergency bottled water would be provided under one of the Navy’s periodic drinking water well sampling and bottled water contract task orders until GAC media replacement is complete.

## Worksheet #11—Project Quality Objectives/Systematic Planning Process Statements (Continued)

### What are the Project Action Limits?

The effluent PALs for PFOA and PFOS are 70 ppt individually or combined (if both chemicals are detected in a sample, then 70 ppt for the summed concentrations of PFOA and PFOS). There are no PALs for the other 27 PFAS being analyzed (Worksheet #10). The results for those PFAS will be archived for future use, if needed.

The midpoint PAL is 35 ppt, PFOS and/or PFOA (one half the effluent PAL). The midpoint PAL will be used to trigger and schedule FCWTP GAC media changeout for this project. Because the sum of the PFOA and PFOS concentrations will be used, if one compound exceeds 35 ppt while the other is non-detect, the sum will still exceed the midpoint PAL. Using the midpoint PAL will conservatively prevent treated water exceeding the effluent PAL for PFOS/PFOA from entering the Town's municipal potable water supply system.

On March 14, 2023, EPA proposed a draft regulatory drinking water standard for certain PFAS, including PFOA and PFOS. In response, DoD has issued the following statement:

“DoD respects and values the public comment process on this proposed nationwide drinking water rule and looks forward to the clarity that a final regulatory drinking water standard for PFAS will provide. In anticipation of the final standard that EPA expects to publish by the end of 2023, the DoD is assessing what actions DoD can take to be prepared to incorporate EPA's final regulatory standard into our current cleanup process, such as reviewing our existing data and conducting additional sampling where necessary. In addition, DoD will incorporate nationwide PFAS cleanup guidance, issued by EPA and applicable to all owners and operators under the federal cleanup law, as to when to provide alternate water when PFAS are present.”

### For what will the data be used?

The data will be used by the Navy, its contractors, and the other stakeholder agencies to address the environmental questions and PQOs listed in **Table 11-1**. Under EPA Methods 533 and 537.1, the data will be analyzed for 29 compounds. Based on current Navy policy, further investigation and removal action decisions are only based on PFOA and PFOS. All other data will be archived in an appendix of the drinking water report for future use.

### What types of data are needed (matrix, target analytes, analytical groups, field screening, onsite analytical or offsite laboratory techniques, sampling techniques)?

**Worksheets #14, #15, and #18** contain detailed information on the types of data needed for this project.

### Are there any special data quality needs, field or laboratory, in order to support environmental decisions?

All off-Base laboratory analytical data will be technically sound and defensible with respect to the aforementioned project objectives. Additionally, laboratory-specific LODs will be less than 70 ppt for PFOA and/or PFOS, individually or combined. QC sample requirements are detailed in **Worksheet #20**. For action decisions, the laboratory will follow the Measurement Performance Criteria (MPC) in **Worksheets #24 and #28** for laboratory QC samples. These MPC are consistent with the latest versions of EPA Method 533 and Method 537.1 Version 2.

### Where, when, and how should the data be collected/generated?

**Worksheets #14 and #18** describe how the drinking water samples will be collected. Detailed information on how data will be collected is provided in **Worksheet #14**, following the SOP listed in **Worksheet #21**. Sampling is based on the rationale presented in **Worksheet #17** and will be in accordance with the project schedule outlined in **Worksheet #16**.

## Worksheet #12-1—Field Quality Control Samples

<b>Matrix:</b> Drinking Water <b>Analytical Group:</b> PFAS <b>Analytical Method:</b> EPA Method 533				
QC Sample <sup>a</sup>	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria
Field Duplicate (FD)	PFAS	One per 10 normal samples	Precision	Relative percent difference (RPD) less than 30% for water. Greater variability may be observed when FDs have analyte concentrations that are within a factor of 2 of the LOQ. At these concentrations, FDs should have RPDs that are less than 50%.
Field Reagent Blank (FRB)		One per normal field sample	Bias/Contamination	If the same PFAS analyte is present in both the sample and FRB, and the concentration of that analyte greater than 1/3 LOQ in the FRB it will require all associated samples to be resampled and/or reanalyzed; however, decision making and/or action (i.e., providing an alternate drinking water source) may proceed in advance of the resampling and reanalysis.
Cooler Temperature Indicator		One per cooler	Representativeness	Samples must be shipped on ice and received at the laboratory at or below 10 degrees Celsius (°C) and stored in the laboratory at or below 6°C. Samples must not be frozen.

<sup>a</sup> Although matrix spike (MS)/matrix spike duplicate (MSD) samples might be considered Field QC, they are shown on **Worksheet #28**.

## Worksheet #12-2—Field Quality Control Samples

<b>Matrix:</b> Drinking Water <b>Analytical Group:</b> PFAS <b>Analytical Method:</b> EPA Method 537.1 Version 2				
QC Sample <sup>a</sup>	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria
FD	PFAS	One per 10 normal samples	Precision	Relative percent difference (RPD) less than 30% for water. Greater variability may be observed when FDs have analyte concentrations that are within a factor of 2 of the LOQ. At these concentrations, FDs should have RPDs that are less than 50%.
FRB		One per normal field sample	Bias/Contamination	If the same PFAS analyte is present in both the sample and FRB, and the concentration of that analyte greater than 1/3 LOQ in the FRB it will require all associated samples to be resampled and/or reanalyzed; however, decision making and/or action (i.e., providing an alternate drinking water source) may proceed in advance of the resampling and reanalysis.
Temperature Blank		One per cooler	Representativeness	Samples must be shipped on ice and received at the laboratory at or below 10 °C and stored in the laboratory at or below 6°C. Samples must not be frozen.

<sup>a</sup> Although MS/MSD samples might be considered Field QC, they are shown on **Worksheet #28**.

## Worksheet #14—Summary of Project Tasks

This worksheet provides a summary of the project tasks. Identification of specific applicable tasks are further detailed in **Worksheets #17** and **#18**. Refer to **Worksheet #21** for the list of field sampling standard operating procedures (SOPs), and Appendix A for the full text of the sampling SOPs.

### Work Planning Tasks

- SAP Preparation and Delivery
- Subcontractor procurement
  - Analytical laboratory
  - Data Validator
- Compile sampling forms
- Fieldwork scheduling
- Sample appointment and scheduling

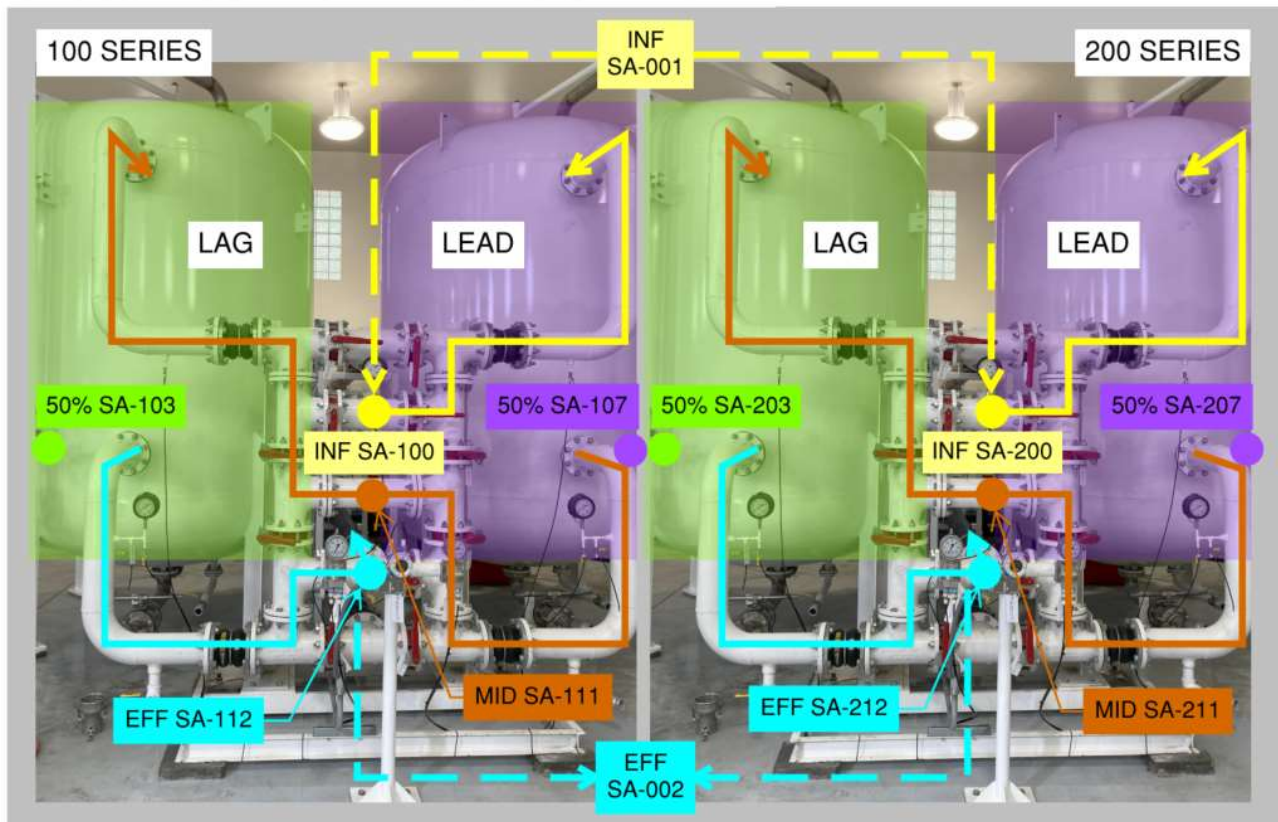
### Performance Monitoring Tasks

- Performance monitoring will occur quarterly as outlined in the monitoring flow diagram on **Figure 2**. Monitoring will continue on a routine basis for the duration the FCWTP is in use. The regular monitoring frequency is based on the sampling results from the first two years of system operation (i.e., July 2019 to July 2021). A sample event is any event where 12 process monitoring water samples and/or two backwash samples are collected. Collection of the 12 process monitoring water samples will follow the schedule outlined in **Figure 2**. Collection of the two backwash water samples will occur following GAC changeout, which is independent of the schedule outlined in **Figure 2**.
- Twelve process monitoring water samples will be collected at the following locations at the FCWTP, as shown in **Figures 3 through 9** and the photo in this worksheet:
  1. Influent SA-001 (After the supply well network and before the raw water tank)
  2. The GAC Tanks are lead/lag. The lead/lag operational configuration does not affect whether sample locations are “midpoint” or “effluent” sample locations.
    - a. Five samples will be collected from the GAC Tank 101 and 102 configuration (100-series GAC Tanks).
      - Influent SA-100 (GAC Treatment Train 1 between GAC Tank 101 and 102)
      - Water at 50% media SA-103 (GAC Treatment Train 1 Tank 101)
      - Midpoint SA-111 (GAC Treatment Train 1, mid point between lead tank and lag tank, regardless of whether Tank 101 or Tank 102 is lead tank)
      - Water at 50% media SA-107 (GAC Treatment Train 1 Tank 102)
      - Effluent SA-112 (GAC Treatment Train 1 after GAC lag tank)
    - b. Five samples will be collected from the GAC Tank 201 and 202 configurations (200-series GAC Tanks).
      - Influent SA-200 (GAC Treatment Train 2 between GAC Tank 201 and 202)
      - Water at 50% media SA-203 (GAC Treatment Train 2 Tank 201)
      - Midpoint SA-211 (GAC Treatment Train 2, mid point between lead tank and lag tank, regardless of whether Tank 201 or Tank 202 is lead tank)
      - Water at 50% media SA-207 (GAC Treatment Train 2 Tank 202)
      - Effluent SA-212 (GAC Treatment Train 2 after GAC lag tank)
  3. Effluent SA-002 (After the treated water tank and before transfer and distribution pumps)

## Worksheet #14—Summary of Project Tasks (Continued)

4. Two backwash samples for the media filter and GAC will be collected at ‘backwash waste to pond’ as shown in **Figures 3 through 9**. These samples will be collected immediately after each GAC changeout, which does not have a set schedule.
  - Media Filter BW-003
  - GAC BW-004
- Water samples will be collected in accordance with **Worksheet #18** and with the SOPs listed in **Worksheet #21** and provided in **Appendix A**.
- Water samples will be collected at the FCWTP following the sampling protocol as specified in **Worksheet #18**.
- Field measurements of pH and conductivity will be collected from influent samples. This field data will be used for qualitative system troubleshooting and not for project GAC media change-out triggers.
- Applicable PFAS-free field book and forms should be filled out completely each day.

### GAC CONFIGURATION: MARCH 2023 - PRESENT



### Sample Shipment

All analytical samples and equipment will be shipped by FedEx. All samples will be shipped in accordance with the SOPs referenced in **Worksheet #21**.



## Worksheet #14—Summary of Project Tasks (Continued)

### Sample Analysis and Testing Tasks

- The subcontracted analytical laboratory will process and prepare samples for analyses and will analyze all samples for the PFAS compounds listed in **Worksheet #15**, in accordance with **Worksheets #18 and #19**.

### Quality Control, Assessment, and Audit Tasks

- Implement SOPs for field and laboratory activities being performed.
- Collect QC samples as described on **Worksheets #12 and #20**.
- Perform field performance audits, safe work observations, and field document reviews described in **Worksheets #31 and #32**.

### Data Review and Use Tasks

- Preliminary, non-validated, analytical data will be reviewed upon receipt from the laboratory to determine if PFAS treatment at the time of sampling was effective or whether the Project Action Limits (PALs) have been exceeded. If the midpoint PAL, as defined in **Worksheet #11**, has been exceeded at either Midpoint location SA-111 or SA-211, the supplier will be contacted to change out the GAC within 5-business days. In the unlikely event that the data shows the effluent PAL, as defined in **Worksheet #11**, has been exceeded at the Effluent location EFF-SA-002, community residents would be advised to use bottled water.

### Secondary Data Review

- See **Worksheet #13**.

### Data Validation, Review, and Management Tasks

- See **Worksheets #34 through #36** for discussion of data management procedures.

### Documentation and Reporting

- Following the completion of the last quarterly sampling event, a summary of field activities for FCWTP sampling will be documented in one Operations and Maintenance (O&M) report submitted to the Navy RPM for review and approval.
- FCWTP performance monitoring sample results will be communicated after each sampling event to the Navy RPM by email.

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## Worksheet #15-1—Reference Limits and Evaluation Table

One of the objectives of this SAP Addendum is to provide guidance for selecting the appropriate analytical methods and laboratories that will provide data that satisfy the project objectives and general investigation approach established in **Worksheet #11**. The table below summarizes the analytical method, target analytes, PALs, and laboratory reference limits for the project laboratory that will analyze the water samples for this project.

<b>Analytical Group:</b> PFAS (EPA Method 533) <b>Matrix:</b> Drinking Water									
Analyte	CAS	PALs <sup>a</sup> (ng/L)		Laboratory-Specific Limits (ng/L)			Accuracy Control Limit (%R) <sup>b</sup>		Precision Control Limit
		Midpoint	Effluent	LOQs (ng/L)	LODs (ng/L)	DLs (ng/L)	LCL	UCL	(%RPD)
Perfluorooctanoic acid (PFOA)	335-67-1	35	70	2	1.6	0.6	70	130	30
Perfluorooctanesulfonic acid (PFOS)	1763-23-1	35	70	2	1.6	0.6	70	130	30
Perfluorobutanesulfonic acid (PFBS)	375-73-5	NC	NC	2	1.6	0.6	70	130	30
Perfluorodecanoic acid (PFDA)	335-76-2	NC	NC	2	1.6	0.64	70	130	30
Perfluorododecanoic acid (PFDoA)	307-55-1	NC	NC	2	1.6	0.6	70	130	30
Perfluoroheptanoic acid (PFHpA)	375-85-9	NC	NC	2	1.6	0.68	70	130	30
Perfluorohexanesulfonic acid (PFHxS)	355-46-4	NC	NC	2	1.6	0.6	70	130	30
Perfluorohexanoic acid (PFHxA)	307-24-4	NC	NC	2	1.6	0.6	70	130	30
Perfluorononanoic acid (PFNA)	375-95-1	NC	NC	2	1.6	0.6	70	130	30
Perfluoroundecanoic acid (PFUnA)	2058-94-8	NC	NC	2	1.6	0.65	70	130	30
4,8-dioxo-3H-perfluorononanoic acid (ADONA)	919005-14-4	NC	NC	4	1.6	0.6	70	130	30
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	756426-58-1	NC	NC	4	1.6	0.6	70	130	30
Hexafluoropropylene oxide dimer acid (HFPO-DA)	13252-13-6	NC	NC	4	2	1	70	130	30
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2FTS)	757124-72-4	NC	NC	4	1.6	0.6	70	130	30
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2FTS)	27619-97-2	NC	NC	4	1.6	1.1	70	130	30
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2FTS)	39108-34-4	NC	NC	4	1.6	0.6	70	130	30
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)	151772-58-6	NC	NC	4	1.6	0.6	70	130	30

## Worksheet #15-1—Reference Limits and Evaluation Table (Continued)

<b>Analytical Group:</b> PFAS (EPA Method 533)									
<b>Matrix:</b> Drinking Water									
Analyte	CAS	PALs <sup>a, b</sup> (ng/L)		Laboratory-Specific Limits (ng/L) <sup>c</sup>			Accuracy Control Limit (%R) <sup>d</sup>		Precision Control Limit
		Midpoint	Midpoint	LOQs (ng/L)	LODs (ng/L)	DLs (ng/L)	LCL	UCL	(%RPD)
Perfluorobutanoic acid (PFBA)	375-22-4	NC	NC	4	1.6	0.6	70	130	30
Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)	113507-82-7	NC	NC	4	1.6	0.6	70	130	30
Perfluoroheptanesulfonic acid (PFHpS)	375-92-8	NC	NC	2	1.6	0.6	70	130	30
Perfluoro-4-methoxybutanoic acid (PFMBA)	863090-89-5	NC	NC	4	1.6	0.6	70	130	30
Perfluoro-3-methoxypropanoic acid (PFMPA)	377-73-1	NC	NC	4	1.6	0.64	70	130	30
Perfluoropentanoic acid (PFPeA)	2706-90-3	NC	NC	4	1.6	0.68	70	130	30
Perfluoropentanesulfonic acid (PFPeS)	2706-91-4	NC	NC	2	1.6	0.77	70	130	30
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	763051-92-9	NC	NC	4	1.6	0.68	70	130	30
PFOA + PFOS (calculated)	-	35	70	-	-	-	-	-	-

<sup>a</sup> Refer to **Worksheet #11** for a detailed discussion on the development of PALs.

<sup>b</sup> NC = No criteria for this compound.

<sup>c</sup> The project laboratory is SGS North America located in Vallejo, CA (see **Worksheet #30**).

<sup>d</sup> Limits shown are for spikes greater than LOQ. Limits are 50 to 150 percent for spikes at or below the LOQ. These limit requirements follow EPA Method 533.

%R = percent recovery

CAS = Chemical Abstract Service Number

PAL = Project Action Limit

LOQ = Limit of Quantitation

LOD = Limit of Detection

DL = Detection Limit

LCL = Lower Control Limit

UCL = Upper Control Limit

RPD = Relative Percent Difference

## Worksheet #15-2—Reference Limits and Evaluation Table

Analytical Group: PFAS (EPA Method 537.1 Version 2)									
Matrix: Drinking Water									
Analyte	CAS	PALs <sup>a, b</sup> (ng/L)		Laboratory-Specific Limits (ng/L) <sup>c</sup>			Accuracy Control Limit (%R) <sup>d</sup>		Precision Control Limit
		Midpoint	Midpoint	LOQs (ng/L)	LODs (ng/L)	DLs (ng/L)	LCL	UCL	(%RPD)
N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	2991-50- 6	NC	NC	4	2	1	70	130	30
N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	2355-31- 9	NC	NC	4	2	1	70	130	30
Perfluorotridecanoic acid (PFTrDA)	72629- 94-8	NC	NC	2	1.6	0.8	70	130	30
Perfluorotetradecanoic acid (PFTA)	376-06-7	NC	NC	2	1.6	0.8	70	130	30

<sup>a</sup> Refer to **Worksheet #11** for a detailed discussion on the development of PALs.

<sup>b</sup> NC = No criteria for this compound.

<sup>c</sup> The project laboratory is SGS North America located in Vallejo, CA (see **Worksheet #30**).

<sup>d</sup> Limits shown are for spikes greater than LOQ. Limits are 50 to 150 percent for spikes at or below the LOQ. These limit requirements follow EPA Method 537.1 Version 2.

%R = percent recovery

CAS = Chemical Abstract Service Number

PAL = Project Action Limit

LOQ = Limit of Quantitation

LOD = Limit of Detection

DL = Detection Limit

LCL = Lower Control Limit

UCL = Upper Control Limit

RPD = Relative Percent Difference

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## Worksheet #16—Project Schedule/Timeline Table

This worksheet provides a schedule summary of the project tasks listed in **Worksheet #14**.

Activities	Organization	Dates (MM/DD/YYYY)		Deliverable
		Anticipated Date of Initiation	Anticipated Date of Completion	
<b>Work Planning</b>				
<b>SAP Preparation and Delivery</b>				
Draft SAP Addendum preparation	DCG/W, Town	10/30/2023	12/29/2023	Draft SAP Addendum
Navy SAP Addendum review	NAVFAC Northwest/Atlantic	01/01/2024	01/18/2024	Comments
Response to Comments	DCG/W, Town	01/19/2024	02/01/2024	Comment Responses
Final SAP Addendum preparation	DCG/W, Town	02/02/2024	02/21/2024	Final SAP Addendum
<b>Sample Collection, Analysis, and Data Review</b>				
Quarterly sample collection	Town	February 2024, May 2024, August 2024	August 2024	Water samples
Sample analysis	Project Laboratory Subcontractor	February 2024, May 2024, August 2024	August 2024	Preliminary, non-validated, analytical sample data
Data validation, review, and management	Third Party Data Validator Subcontractor	February 2024, May 2024, August 2024	August 2024	Final, validated, analytical sample data
<b>Quality Control, Assessment, and Testing</b>				
Implement SOPs for field and laboratory activities	DCG/W, Town	February 2024, May 2024, August 2024	August 2024	N/A
Collect and analyze QC samples	Town	February 2024, May 2024, August 2024	August 2024	Analytical QC sample data
Perform safe work observations, and field document reviews	Town	February 2024, May 2024, August 2024	August 2024	Performance audit, safe work, and field review reports and findings.
Lab Accreditation Performance Testing	Town	May 2024	May 2024	Report

Worksheet #16—Project Schedule/Timeline Table (Continued)

Activities	Organization	Dates (MM/DD/YYYY)		Deliverable
		Anticipated Date of Initiation	Anticipated Date of Completion	
<b>Documentation and Reporting</b>				
<b>Monitoring Data</b>				
Submit performance monitoring sample results	Town	February 2024, May 2024, August 2024	August 2024	Email containing a table of validated, analytical sample data.
<b>O&amp;M Report</b>				
Draft O&M report preparation	DCG/W, Town	10/15/2024	12/23/2024	Draft report
Draft O&M report review	NAVFAC Northwest	12/24/2024	01/10/2025	Comments
Response to Comments	DCG/W, Town	01/13/2025	01/24/2025	Comment Responses
Submit final O&M report	DCG/W, Town	01/27/2025	02/13/2025	Final report



## Worksheet #17—Sampling Design and Rationale

**Table 17-1** presents the sampling strategy and rationale.

**Table 17-1. Performance Monitoring Sampling Strategy and Rationale**

*Fort Casey WTP, Coupeville, Washington*

<b>Matrix</b>	Drinking Water and Drinking Water Treatment Process Backwash Water
<b>Analysis and Method <sup>a</sup></b>	PFAS by USEPA Method 537.1 Version 2 and USEPA Method 533
<b>Sampling Frequency</b>	Collection events will occur quarterly and following any GAC media replacements. The collection events will follow FCWTP commissioning as outlined in the monitoring flow diagram on <b>Figure 2</b> . After which, collection events will be conducted based on an updated and revised schedule. Media Filter and GAC Backwash samples will be taken once on the same day following GAC Media replacement. Quarterly collection events and GAC changeout will not necessarily occur simultaneously. Backwash collection events have separate sampling frequencies. The sample plan is outlined in <b>Worksheet #11</b> and <b>Figure 2</b> .
<b>Number of Samples <sup>b</sup></b>	12 process monitoring samples per collection event 2 composite backwash samples per each GAC changeout
<b>No. of Field Duplicates <sup>b</sup></b>	1 field duplicate per up to 10 parent samples
<b>No. of MS/MSDs pairs <sup>b</sup></b>	1 MS/MSD pair per up to 20 parent samples
<b>No. of Field Reagent Blanks <sup>b</sup></b>	4 field reagent blank per sample set
<b>Rationale</b>	Water samples will be collected from the three influent, two midpoint, four 50% media, and three effluent location(s) at the FCWTP as specified in <b>Figures 3 through 9</b> . Initial Media Filter and GAC Backwash samples will be collected at the FCWTP location as specified in <b>Figures 3 through 9</b> during backwashing and on the same day following GAC Media replacement. The backwash samples will consist of equal volume samples collected at 5-minute intervals during the duration of the backwash, discarding any sample volumes that contain visible GAC fines or cloudiness, and then mixing together remaining samples to create separate representative composite samples from GAC and media filter backwash flows. Collected samples will be analyzed for PFAS compounds to: 1) ensure effectiveness of the FCWTP to continuously reduce PFOA and PFOS concentrations to levels less than the effluent PAL in municipal potable water outflowing from the WTP, and 2) implement a long-term monitoring and maintenance plan to verify and document treatment effectiveness ( <b>Worksheets #11 and #15</b> ).

<sup>a</sup> The 29 PFAS compounds listed in UCMR 5 will be analyzed. EPA Methods 533 and 537.1 Version 2 are used in tandem to provide data for all 29 PFAS compounds required. EPA Method 537.1 Version 2 is needed to report four compounds which are not provided by EPA Method 533.

<sup>b</sup> Samples will be collected as detailed in **Worksheets #14, #17, and #18**. Field QA/QC samples will be collected as detailed in **Worksheet #12**.

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## Worksheet #18—Sampling Locations and Methods/SOP Requirements Table

Water samples will be collected from the influent, midpoint, 50% media, and effluent of the FCWTP as shown in **Figures 3 through 9**. Collected water samples will be analyzed for the PFAS compounds listed in **Worksheet #15-1** and **Worksheet #15-2** using USEPA Methods 533 and 537.1 Version 2, respectively. The table below provides the station location ID, sample name ID, matrix, analytical group, sample quantity, collection frequency, and applicable field SOP reference.

Sample Type <sup>a</sup>	Station Location Identification (ID) <sup>a, b</sup>	Sample Name ID <sup>b</sup>	Matrix <sup>c</sup>	Analytical Group	Quantity of Samples	Collection Frequency	Field SOP Reference
FCWTP Influent	FCWTP-INF-SA-001	FCWTP-INF-SA-001-MM/DD/YY	DW	PFAS (Method 533 and Method 537.1 Version 2)	1 per method	One per method per event	Worksheet #21
Train 1 Influent	FCWTP-INF-SA-100	FCWTP-INF-SA-100-MM/DD/YY			1 per method	One per method per event	
Tank 101 50% Media	FCWTP-50%-SA-103	FCWTP-50%-SA-103-MM/DD/YY			1 per method	One per method per event	
Train 1 Midpoint	FCWTP-MID-SA-111	FCWTP-MID-SA-111-MM/DD/YY			1 per method	One per method per event	
Tank 102 50% Media	FCWTP-50%-SA-107	FCWTP-50%-SA-107-MM/DD/YY			1 per method	One per method per event	
Train 1 Effluent	FCWTP-EFF-SA-112	FCWTP-EFF-SA-112-MM/DD/YY			1 per method	One per method per event	
Train 2 Influent	FCWTP-INF-SA-200	FCWTP-INF-SA-200-MM/DD/YY			1 per method	One per method per event	
Tank 201 50% Media	FCWTP-50%-SA-203	FCWTP-50%-SA-203-MM/DD/YY			1 per method	One per method per event	
Train 2 Midpoint	FCWTP-MID-SA-211	FCWTP-MID-SA-211-MM/DD/YY			1 per method	One per method per event	
Tank 202 50% Media	FCWTP-50%-SA-207	FCWTP-50%-SA-207-MM/DD/YY			1 per method	One per method per event	
Train 2 Effluent	FCWTP-EFF-SA-212	FCWTP-EFF-SA-212-MM/DD/YY			1 per method	One per method per event	
FCWTP Effluent	FCWTP-EFF-SA-002	FCWTP-EFF-SA-002-MM/DD/YY			1 per method	One per method per event	
Media Filter Backwash	FCWTP-MF-BW-003	FCWTP-MF-BW-003-MM/DD/YY	DWTPWW	1 per method	One per method per event: composite sample following backwash sampling composite protocol <sup>e</sup>		
GAC Filter Backwash	FCWTP-GAC-BW-004	FCWTP-GAC-BW-004-MM/DD/YY		1 per method			

Sample Type <sup>a</sup>	Station Location Identification (ID) <sup>a, b</sup>	Sample Name ID <sup>b</sup>	Matrix <sup>c</sup>	Analytical Group	Quantity of Samples	Collection Frequency	Field SOP Reference
Influent Field Duplicate	FCWTP-INF-SA-001	FCWTP-P-INF-SA-001-MM/DD/YY	QC		1 per method	One per method per 10 samples	
Train 1 Midpoint Field Duplicate	FCWTP-MID-SA-111	FCWTP-P-MID-SA-111-MM/DD/YY					
Train 2 Midpoint Field Duplicate	FCWTP-MID-SA-211	FCWTP-P-MID-SA-211-MM/DD/YY					
Effluent Field Duplicate	FCWTP-EFF-SA-002	FCWTP-P-EFF-SA-002-MM/DD/YY			1 per method	One per method per 20 samples	
Effluent (MS/MDS)	FCWTP-EFF-SA-002	FCWTP-MS-EFF-SA-002-MM/DD/YY					
		FCWTP-MSD-EFF-SA-002-MM/DD/YY					
Influent Field Reagent Blank	FCWTP-INF-SA-001	FCWTP-FRB-INF-SA-001-MM/DD/YY			1 per method	One per method per event	
Train 1 Midpoint Field Reagent Blank	FCWTP-MID-SA-111	FCWTP-FRB-MID-SA-111-MM/DD/YY			1 per method	One per method per event	
Train 2 Midpoint Field Reagent Blank	FCWTP-MID-SA-211	FCWTP-FRB-MID-SA-211-MM/DD/YY			1 per method	One per method per event	
Effluent Field Reagent Blank	FCWTP-EFF-SA-002	FCWTP-FRB-EFF-SA-002-MM/DD/YY			1 per method	One per method per event	

## Worksheet #18—Sampling Locations and Methods/SOP Requirements Table (Continued)

### Notes:

- <sup>a</sup> Potential samples to be collected from multiple station locations at the FCWTP including the three GAC treatment process influent, two GAC process midpoint, four 50% media, and three effluent location(s).
- <sup>b</sup> Station location ID and sample name ID nomenclature is based on the following:
- FCWTP = Fort Casey Water Treatment Plant
  - INF = Influent Location
  - MID = Midpoint Location
  - EFF = Effluent Location
  - P = Sample is a field duplicate
  - SA = Sample station
  - FRB = Field blank
  - MM/DD/YY = Two-digit month, day, and year
  - MS = Sample is a matrix spike
  - MSD = Sample is a matrix spike duplicate
  - 50 = 50% media location
- <sup>c</sup> Drinking water and drinking water treatment process backwash water samples will be collected as described in **Worksheet #14** and **#17**. Field QA/QC samples will be collected as detailed in **Worksheet #12**.
- <sup>d</sup> One field duplicate sample (FD) will be collected for every 10 parent samples collected from any station location sampled (i.e. Influent, Midpoint, or Effluent). Field duplicate samples will be named with a “P” after the station location ID.
- <sup>e</sup> Collect equal volume samples at a 5-minute interval during the backwash, discarding any sample volumes that contain visible GAC fines or cloudiness, then mixing all remaining samples (minimum of five) together to create combined composite samples that are representative of the backwash flow.

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Worksheet #19—Analytical SOP Requirements Table

Matrix	Analytical Group	Analytical and Preparation Method/ SOP Reference	Container(s) (number, size & type per sample)	Sample Volume	Preservation Requirements	Maximum Holding Time <sup>a</sup> (preparation/analysis)
Drinking Water	PFAS	EPA Method 533/ SOPs MS022.6	Two 250-milliliter (mL) polypropylene caps w/o Teflon liner	250 mL	Temperature must be above freezing and less than or equal to 10°C when received at the laboratory. Samples stored in the laboratory must be held at or below 6 °C until extraction but should not be frozen. Preserve with Ammonium Acetate	28 days from sampling to extraction/28 days from extraction to analysis
Drinking Water	PFAS	EPA Method 537.1 Version 2/ SOPs MS017.2	Two 250-milliliter (mL) polypropylene bottles w/o Teflon liners	250 mL	Temperature must be above freezing and less than or equal to 10°C when received at the laboratory. Samples stored in the laboratory must be held at or below 6 °C until extraction but should not be frozen. Preserve with Trizma.	14 days from sampling to extraction/28 days from extraction to analysis

<sup>a</sup> Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

<sup>b</sup> For both Method 533 and Method 537.1 Version 2, the same lot of preservative must be used for the FRBs as for the field samples.

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## Worksheet #20—Field Quality Control Sample Summary Table

Matrix	Analytical Group	Analytical Method	Number of Sampling Locations <sup>1</sup>	Number of Field Duplicates <sup>1</sup>	Number of MS/MSDs <sup>1</sup>	Number of Field Reagent Blanks <sup>1</sup>	Total Number of Samples to Lab <sup>1</sup>
Drinking Water	PFAS	EPA Method 533	12 process monitoring samples	One per 10 samples	One pair (2 samples) per 20 samples	Four per sample set	19-21 <sup>2</sup>
Drinking Water Treatment Process Backwash Water	PFAS		2 composited backwash samples per each GAC changeout				
Drinking Water	PFAS	EPA Method 537.1 Version 2	12 process monitoring samples	One per 10 samples	One pair (2 samples) per 20 samples	Four per sample set	19-21 <sup>2</sup>
Drinking Water Treatment Process Backwash Water	PFAS		2 composited backwash samples per each GAC changeout				

Notes:

- <sup>1</sup> The listed sample quantities are per each individual sampling event. Multiple sampling events are scheduled and expected. The total number of samples to be sent to the lab per sampling event will consist of 12 samples plus QC samples (up to 7 QC samples including a field duplicate, a matrix spike, a matrix spike duplicate, and field reagent blanks for each sample). Samples will be collected as detailed in **Worksheets #14, #17, and #18** of this SAP. Field QA/QC samples will be collected as detailed in **Worksheet #12**.
- <sup>2</sup> Not every process monitoring sample will require backwash samples, backwash samples are only required following GAC media changeouts. When no GAC media change out has occurred, only 19 samples will be sent to the lab. Following GAC changeout, 21 samples will be sent to the lab.

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Worksheet #22—Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Activity	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference	Comments
Horiba U-22 (or equivalent) Temperature Probe	Verification of calibration <sup>a</sup>	Verify daily, before use, using stable room temperature water and traceable digital pocket thermometer	Consistent with the current ambient temperature	Do not use instrument if not operating properly	FTL	EID-FS-204	<b>Worksheet #21 and Appendix A</b>
Traceable Digital Pocket Thermometer	Calibration not required with current calibration certificate date valid within last year	N/A	N/A	N/A	N/A	N/A	

<sup>a</sup> Temperature is not a parameter that can be calibrated in the field.

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Worksheet #23—Laboratory SOP References Table

SOP #	Title, Date, and URL (if available)	Date Last Revisited if Not Revised	Definitive or Screening Data	Matrix/Analytical Group	Instrument	Organization Performing Analysis	Variance to QSM (Yes/No)	Modified for Project? (Yes/No)
MS022.6	Analysis of Per- and Polyfluorinated Alkyl Substances by LC/MS/MS and Isotope Dilution, 06/2023, Rev. 6	N/A	Definitive	Drinking Water/PFAS	HPLC – Agilent Technologies 1260 or 1290	SGS Laboratories	No	No
MS017.12	Analysis of Perfluorinated Alkyl Substances by LC/MS/MS, 06/2023, Rev. 12	N/A	Definitive	Drinking Water/PFAS	HPLC – Agilent Technologies 1260 or 1290	SGS Laboratories	No	No

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## Worksheet #24—Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for Corrective Action	SOP Reference
Agilent HPLC/MS/MS (Electrospray detector) by EPA Method 533	Mass Calibration verification; ICAL 5 points minimum; 7 points per SGS – Orlando routine procedure.	Major maintenance (per method) or second consecutive failure of opening CCV warrants recalibration.	Passing mass calibration within +/- 0.5 amu; ICAL must be forced through zero, $R > 0.995$ and each calibration point $\%D < 30\%$ . Low point $\%D < 50\%$ ; ICV and mid- and high-level CCV $\%D < 30\%$ ; low-level CCV $\%D < 50\%$ CCV ISTD $\%D < 30\%$ .	Instrument maintenance, mass re-tuning; standard inspection, recalibration.	Laboratory Analyst	MS022.6
Agilent HPLC/MS/MS (Electrospray detector) by EPA Method 537.1 Version 2	Mass Calibration verification; ICAL 5 points minimum per method; 8 points SGS – Orlando's routine procedure	Major maintenance (per method) or second consecutive failure of opening CCV warrants recalibration. Mass calibration verification recommended frequency -- weekly	Passing mass calibration within +/- 0.5 amu; ICAL must be forced through zero, $R \geq 0.995$ and each calibration point $\%D < 30\%$ . Low point $\%D < 50\%$ ; CCV and ICV $\%D < 30\%$ ;	Instrument maintenance, mass re-tuning, standard inspection, recalibration	Laboratory Analyst	MS017.12

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Worksheet #25—Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/position responsible for corrective action	Reference
Agilent HPLC/MS/MS	Spray chamber, Clean capillary	EPA 533 Perfluorinated compounds	Check Tune Leak checks Pressure check Mobile phase filters Needle inspection	Need for maintenance determined by passing calibration– see MS022	Passing calibration	Check LC column Run Autotune Check calculations Re-run affected samples	Laboratory Analyst	MS022
Agilent HPLC/MS/MS	Spray chamber, Clean capillary	EPA 537.1 Perfluorinated compounds	Check Tune Leak checks Pressure check Mobile phase filters Needle inspection	Need for maintenance determined by passing calibration– see MS017	Passing calibration	Check LC column Run Autotune Check calculations Re-run affected samples	Laboratory Analyst	MS017

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## Worksheet #27—Sample Custody Requirements Table

### Sample Labeling

Sample labels will include, at a minimum, client name, site, sample location identification (ID) as described in **Worksheet #18**, date and time collected, analysis method, preservative, and sampler's initials. Labels will be applied to the sample bottles to ensure that they do not separate. The sample bottles the laboratory will provide will have color-coded stickers on each bottle to differentiate the different preservatives used for each method.

### Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory)

Samples will be collected by field team members under the supervision of the FTL. As samples are collected, they will be placed into the appropriate containers required for each method and labeled as outlined above. Samples will be cushioned with packaging material and placed into coolers containing enough ice to keep the samples  $\leq 10^{\circ}\text{C}$ , samples must be shipped on ice and must not exceed  $10^{\circ}\text{C}$  within the first 48 hours after collection. Samples are valid if any ice remains in the cooler when it is received at the laboratory or bottles are received within 2 days and must be confirmed to be at or below  $10^{\circ}\text{C}$  when the samples are received at the laboratory. Storage in the laboratory will be  $\leq 6^{\circ}\text{C}$ , but not frozen. The chain-of-custody will also be placed into the cooler. Coolers will be shipped directly to the laboratory via methods described in **Worksheet #21**, with the air bill number indicated on the chain-of-custody (to relinquish custody). Upon delivery, the laboratory will log in each cooler and report the status of the samples.

### Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):

Samples are received as is, temperature and preservation checked and recorded. Samples outside of acceptance criteria are flagged and brought to Town's attention. Town then forwards it to Navy RPM for approval. Samples are placed in controlled temperature storage according to analyses ordered. After analyses are completed and reports are submitted to the clients, samples are archived for 60 days and later disposed according to matrix and whether or not they are considered hazardous waste.

### Sample Identification Procedures:

Upon opening the cooler, the receiving clerk signs the chain-of-custody and then takes the temperature using the temperature blank (if absent, then a sample container or infrared thermometer is used). The sample containers in the cooler are unpacked and checked against the client's chain-of-custody and any discrepancies or breakage are noted on the chain-of-custody. Next, if any water samples require preservative, the clerk will check the pH values to determine if they are in the acceptable pH range. The clerk will deliver the chain-of-custody (and other paperwork; e.g., temperature or pH QA notice) to the PM for LIMS entry and client contact, if needed. The field logbook will identify the sample ID with the location, depth, date and time collected, and the parameters requested. The laboratory will assign each field sample a laboratory sample ID based on information in the chain-of-custody. The laboratory will send sample log-in forms to the Project Chemist to check sample IDs and parameters are correct.

### Chain-of-custody Procedures:

Chain-of-custody will include, at a minimum, laboratory contact information, client contact information, sample information, and relinquished by and received by information. Sample information will include sample ID, date and time collected, number and type of containers, preservative information, analysis method, and comments. The chain-of-custody will also have the sampler's name and signature. The chain-of-custody will link location of the sample from the field logbook to the laboratory receipt of the sample. The laboratory will use the sample information to populate the LIMS database for each sample. SGS - Orlando is designated a secure facility.

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## Worksheet #28-1—Laboratory QC Samples Table

<b>Matrix:</b> Drinking Water <b>Analytical Group:</b> PFAS <b>Analytical Method/SOP Reference:</b> USEPA Method 533/MS022.6					
QC Sample <sup>a</sup>	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicators
Laboratory Reagent Blank	One per prep. batch of up to 20 samples.	For the determination of native PFAS, the levels measured in the method blank of all method analytes must be below 1/3 the LOQ.	Verify instrument clean (evaluate calibration blank and samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re - prepare and reanalyze the method blank and all samples processed with the contaminated blank.	Analyst/ Supervisor	Bias/ Contamination
Isotope Performance Standards	All standards and sample extracts.	Peak area counts for each isotope performance standard must be within 50–150% of the average peak area in the initial calibration.	If an isotope performance standard area for a sample does not meet these criteria, reanalyze the extract in a subsequent Analysis Batch. If the isotope performance standard area 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.	Analyst/ Supervisor	Accuracy
Isotope Dilution Analogues	All samples prior to extraction.	50%–200% recovery for each analog.	If an isotope dilution analog fails to meet the recovery criterion, evaluate the area of the isotope performance standard to which the analog is referenced and the recovery of the analogues in the CCCs. If necessary, recalibrate and service the LC/MS/MS system. Take CA, then analyze the failed extract in a subsequent Analysis Batch. If the repeat analysis meets the 50–200% recovery criterion, report only data for the reanalyzed extract. If the repeat analysis fails the recovery criterion after CA, extraction of the sample must be repeated provided a sample is available and still within the holding time.	Analyst/ Supervisor	Accuracy/ Precision
Laboratory Fortified Blank (LFB)	One LFB is required for each extraction batch of up to 20 field samples. Rotate the fortified concentrations between low, medium, and high amounts.	For analytes fortified at concentrations $\leq 2$ x the LOQ, the result must be within 50–150% of the true value; 70–130% of the true value if fortified at concentrations greater than 2 x the LOQ.	Reanalyze LFB once. If acceptable, report. Evaluate samples for detections, and LFB for high bias. If LFB has high bias, and samples non - detect, report with case narrative comment. If LFB has low bias, or if there are detections for critical chemicals of concern, evaluate and re-prepare and reanalyze the LFB and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Analyst/ Supervisor	Accuracy/ Bias

<sup>a</sup> EPA Method 533 (USEPA, 2019a) is the basis for method performance criteria in this table.

## Worksheet #28-1—Laboratory QC Samples Table (Continued)

QC Sample <sup>a</sup>	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicators
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).	For analytes fortified at concentrations $\leq 2$ x the LOQ, the result must be within 50–150% of the true value; 70–130% of the true value if fortified at concentrations greater than 2 x the LOQ.	Evaluate the data, and re-prepare/ reanalyze the native sample and LFSM/LFSMD pair if laboratory error is indicated.	Analyst/ Supervisor	Accuracy/Bias
LFSMD or FD	Include at least one LFSMD or FD with each extraction batch	For analytes fortified at concentrations $\leq 2$ x the LOQ, the result must be within 50–150% of the true value; 70–130% of the true value if fortified at concentrations greater than 2 x the LOQ. For LFSMDs or FDs, relative percent differences must be $\leq 30\%$ ( $\leq 50\%$ if analyte concentration $\leq 2$ x the LOQ).	Evaluate the data, and re-prepare/ reanalyze the native sample and LFSM/LFSMD pair if laboratory error is indicated.	Analyst/ Supervisor	Accuracy/Bias/ Precision
LOD Verification	Quarterly for every analyte.	Spike a quality system matrix at concentration 2-4 x the DL. Must meet 3:1 signal-to-noise ratio, or for data systems that do not measure noise, results must be at least three standard deviations greater than the mean method blank concentration.	If verification fails, the DL determination must be repeated and a LOD verification. Alternatively pass two consecutive LOD verification at a higher spike and set the LOD at the higher concentration.	Analyst/ Supervisor	Accuracy
LOQ Verification	Quarterly for every analyte.	Spike a quality system matrix at a concentration equal to or greater than the low point of the calibration curve.	Must meet laboratory - specified precision and bias limits. If LOQ fails, repeat at a higher level until limits are met.	Analyst/ Supervisor	Precision/Bias

<sup>a</sup> EPA Method 533 (USEPA, 2019a) is the basis for method performance criteria in this table.

## Worksheet #28-2—Laboratory QC Samples Table

<b>Matrix:</b> Drinking Water <b>Analytical Group:</b> PFAS <b>Analytical Method/SOP Reference:</b> USEPA Method 537.1/MS017.12					
QC Sample <sup>a</sup>	Frequency/ Number	Method/ SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicators
Laboratory Reagent Blank	One per prep. batch of up to 20 samples.	For the determination of native PFAS, the levels measured in the method blank of all method analytes must be below 1/3 the LOQ.	Correct problem. Re-prep and reanalyze method blank and all samples processed with the contaminated blank. If reanalysis cannot be performed, the data must be qualified and explained in the case narrative.	Analyst/ Supervisor	Bias/ Contamination
LFB	One LFB is required for each extraction batch of up to 20 field samples. Rotate the fortified concentrations between low, medium, and high amounts.	Results of LFB analyses must be 70-130% of the true value for each method analyte for all fortified concentrations except the lowest calibration point. Results of the LFBs corresponding to the lowest calibration point for each method analyte must be 50-150% of the true value.	Correct problem, re-prep, and reanalyze LFB and all samples in associated batch for failed analytes. If reanalysis cannot be performed, the data must be qualified and explained in the case narrative.	Analyst/ Supervisor	Accuracy/Bias
LFSM	Analyze one LFSM per extraction batch (20 samples or less) fortified with method analytes at a concentration close to but greater than the native concentration, if known.	Recoveries at mid and high levels must be within 70-130% and within 50-150% at the low-level fortified amount (near the LOQ).	Evaluate the data to determine if the failed criteria are due to sample matrix or laboratory error. Re-prep if sufficient sample is available when lab error is suspected; otherwise, qualify data with narrative.	Analyst/ Supervisor	Accuracy/Bias
LFSMD	Analyze one LFSMD per extraction batch (20 samples or less) fortified with method analytes at a concentration close to but greater than the native concentration, if know.	Recoveries at mid and high levels must be within 70-130% and within 50-150% at the low-level fortified amount (near the LOQ). Method analyte RPDs for the LFSMD or FD must be ≤30% at mid and high levels of fortification and ≤50% near the LOQ.	Evaluate the data to determine if the failed criteria are due to sample matrix or laboratory error. Re-prep if sufficient sample is available when lab error is suspected; otherwise, qualify data with narrative.	Analyst/ Supervisor	Precision/ Accuracy/Bias

<sup>a</sup> EPA Method 537.1 Version 2 (USEPA, 2020a) is the basis for method performance criteria in this table

## Worksheet #28-2—Laboratory QC Samples Table (Continued)

QC Sample <sup>a</sup>	Frequency/ Number	Method/ SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicators
Surrogates Standards	Every field sample, standard, blank, and QC sample.	Within 70 to 130% of true value	Identify and correct the problem. Re-prepare and reanalyze all samples with failed surrogates in the associated preparatory batch. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Qualify all applicable data if acceptance criteria are not met and explain in case narrative.	Analyst/ Supervisor	Accuracy/ Precision
Internal Standard (IS)	Every field sample, standard, blank, and QC sample.	Peak area counts for all ISs in all injections must be within $\pm 50\%$ of the average peak area calculated during the initial calibration and 70 - 140% from the most recent CCC. If ISs do not meet this criterion, corresponding target results are invalid	If peak areas are unacceptable, analyze a second aliquot of the extract or sample if enough extract remains. If there is not enough extract, reanalyze the first aliquot. If second analysis meets acceptance criteria, report the second analysis. If it fails, either analysis may be reported with the appropriate flags	Analyst/ Supervisor	Accuracy
LOD Verification	Quarterly for every analyte.	Spike a quality system matrix at concentration 2-4x the DL. Must meet 3:1 signal-to-noise ratio, or for data systems that do not measure noise, results must be at least three standard deviations greater than the mean method blank concentration.	If verification fails, the DL determination must be repeated and a LOD verification. Alternatively pass two consecutive LOD verification at a higher spike and set the LOD at the higher concentration	Analyst/ Supervisor	Accuracy
LOQ Verification	Quarterly for every analyte.	Spike a quality system matrix at a concentration equal to or greater than the low point of the calibration curve.	Must meet laboratory-specified precision and bias limits. If LOQ fails, repeat at a higher level until limits are met.	Analyst/ Supervisor	Precision/Bias

<sup>a</sup> EPA Method 537.1 Version 2 (USEPA, 2020a) is the basis for method performance criteria in this table



## Worksheet #29—Project Documents and Records Table

Document	Where Maintained
Field Notebooks	Electronic .pdf copies in the project file. Hardcopy (bound notebook) in the project file. Archived at project closeout.
Chain-of-Custody Records	Electronic .pdf copies in the contractor's project file. Hardcopy in the data validation report. Archived at project closeout.
Shipping Air Bills	Hardcopy in the project file. Archived at project closeout.
Telephone Logs	Hardcopy in the project file. Archived at project closeout.
Corrective Action (CA) Forms	Electronic .pdf copies in the project file. Hardcopy in the project file. Archived at project closeout.
Various field measurements	Recorded in Field Notebook.
Pertinent telephone conversations	Recorded in Field Notebook.
Field equipment maintenance records	Inspected by FTL. Not maintained.
Sample Receipt, Chain-of-Custody, and Tracking Records	Electronic .pdf copies in the project file. Hardcopy in the full data package.
Standard Traceability Logs	Hardcopy in the full data package. Archived at project closeout.
Equipment Calibration Logs	Hardcopy in the full data package. Archived at project closeout.
Sample Preparation Logs	Hardcopy in the full data package. Archived at project closeout.
Run Logs	Hardcopy in the full data package. Archived at project closeout.
Equipment Maintenance, Testing, and Inspection Logs	Kept on file at the laboratory. Not maintained.
Reported Field Sample Results	Electronic .pdf copies in the project file. Hardcopy in the data package. Archived at project closeout.
Reported Result for Standards, QC Checks, and QC Samples	Hardcopy in the full data package. Archived at project closeout.
Instrument printouts (raw data) for Field Samples, Standards, QC Checks, and QC Samples	Hardcopy in the full data package. Archived at project closeout.
Data Package Completeness Checklists	Hardcopy in the data validation report. Archived at project closeout.
Sample Disposal Records	Maintained by the laboratory.
Extraction/Clean-up Records	Maintained by the laboratory.
Raw Data	Hardcopy in the full data package. Archived at project closeout.
Field Sampling Audit Checklists	Hardcopy in the project file. Archived at project closeout.
Fixed Laboratory Audit Checklists	If completed, hardcopy in the project file. Archived at project closeout.
Laboratory SOPs	Electronic .pdf copies in the project file. Hardcopy stored with the data package. Archived at project closeout

Data Validation Reports	Electronic .pdf copies in the project file. Hardcopy stored with the data package. Archived at project closeout.
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In general, documents are stored at the Town of Coupeville FCWTP until they are archived.

**Town of Coupeville FCWTP**  
4 NE 7<sup>th</sup> Coupeville, 98239  
(360) 678-4461

Following project completion, hard copy deliverables including chains-of-custody and raw data will be archived at the NAVFAC NW Administrative Record Repository:

NAVFAC NW  
1101 Tautog Circle  
Silverdale, WA 98315-1101  
(360) 556-0621

Worksheet #30—Analytical Services Table

Matrix	Analytical Group	Sample Locations/ID	Analytical Method	Data Package Turnaround Time	Laboratory/Organization	Backup Laboratory/Organization
Drinking Water	PFAS	Refer to <b>Worksheets #18</b> and <b>#20</b>	USEPA Methods 533 & 537.1	14-calendar days	SGS North America, Inc. – Orlando 4405 Vineland Rd., Ste C-15, Orlando, FL 32811 Heather Wandrey (407) 425-6700	A DoD laboratory accredited for the listed analytical methods

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## Worksheet #33—QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, and so forth)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Lab Accreditation Performance Testing	One during 12-month monitoring period	May 2024	Town PM	Included in project files

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## Worksheet #34-36—Data Verification and Validation (Steps I and IIa/IIb) Process Table

<b>Data Review Input</b>	<b>Description<sup>a</sup></b>	<b>Responsible for Verification or Validation</b>	<b>Step I/IIa/IIb<sup>b</sup></b>	<b>Internal/External<sup>c</sup></b>
Field Notebooks	Field notebooks will be reviewed internally and placed into the project file for archival at project closeout.	FTL/Town	Step I	Internal
Chains of Custody and Shipping Forms	Chain of custody forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the chain of custody forms will be initialed by the reviewer, a copy of the chains of custody forms retained in the site file, and the original and remaining copies taped inside the cooler for shipment. Chain of custody forms will also be reviewed for adherence to the SAP.	FTL/Town PM/Town	Step I	Internal & External
Sample Condition upon Receipt	Any discrepancies, missing, or broken containers will be communicated to the FTL in the form of laboratory logs.	FTL/Town	Step I	External
Documentation of Laboratory Method Deviations	Laboratory Method Deviations will be discussed and approved by the PM. Documentation will be incorporated into the case narrative, which becomes part of the final hard copy data package.	PM/Town	Step I	External
EDDs	EDDs will be compared against hard copy laboratory results (10 percent check). If errors are found during the 10-percent check, an additional 25 percent of the EDD to hardcopy will be checked to ensure that the discrepancy was an anomaly.	Data Validator	Step I	External
Case Narrative	Case narratives will be reviewed by the data validator during the DV process. This is verification that they were generated and applicable to the data packages.	Data Validator	Step I	External
Laboratory Data	All laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. All CoAs must be in the lab data reports to all full data validation.	Laboratory QAO	Step I	Internal
Laboratory Data	The data will be verified for completeness. To ensure completeness, EDDs will be compared to the SAP. This is a verification that all samples were included in the laboratory data and that correct analyte lists were reported. All CoAs must be in the lab data reports to all full data validation.	FTL/Town STC/DCG	Step I	External
Lab Accreditation Performance Testing	Upon completion, a copy of testing results will be placed in the site file.	PM/Town	Step I	Internal

Data Review Input	Description <sup>a</sup>	Responsible for Verification or Validation	Step I/IIa/IIb <sup>b</sup>	Internal/External <sup>c</sup>
CA Reports	CA reports will be reviewed by the PM and placed into the project file for archival at project closeout.	PM/Town	Step I	Internal
Laboratory Methods	During the pre-validation check, ensure that the laboratory analyzed samples using the correct methods specified in the SAP. If methods other than those specified in the SAP were used, the reason will be determined and documented.	PM/Town	Step IIa	External
Target Analyte List	During the pre-validation check, ensure that the laboratory reported all analytes from each analysis group as per <b>Worksheet #15-1 and Worksheet #15-2</b> . If the target analyte list is not correct, then it must be corrected prior to sending the data for validation.	FTL/Town STC/DCG	Step IIa	External
Laboratory Limits (DL/LOD/LOQs)	During the pre-validation check, the laboratory limits (DL, LOD, LOQs) will be compared to those listed in the project SAP. If limits were not met, the laboratory will be contacted and asked to provide an explanation, which will then be discussed in the associated project report. Often times the cause for minor laboratory limit deviation from those presented in the SAP is due to the quarterly update of laboratory LOD.	FTL/Town STC/DCG	Step IIb	External
Laboratory SOPs	Ensure that approved analytical laboratory SOPs were followed. Any such discrepancies will be discussed first in the data validation narrative and will be included in the associated project report.	Laboratory QAO	Step IIa	Internal
Sample Chronology	Holding times from collection to extraction or analysis and from extraction to analysis will be considered during the DV process.	Data Validator	Step IIa and IIb	External
Stage of Validation	Ten percent of raw data will undergo Stage 4 validation to confirm laboratory calculations. The remaining 90 percent will undergo Stage 2B validation. For a recalculated result, the data validator attempts to re-create the reported numerical value. The laboratory is asked for clarification if a discrepancy is identified, which cannot reasonably be attributed to rounding. In general, this is outside 5 percent difference.	Data Validator	Step IIa	External
Onsite Screening	All non-analytical field data will be reviewed against SAP requirements for completeness and accuracy based on the field calibration records.	FTL/Town	Step IIb	Internal
Documentation of Method QC Results	Establish that all required QC samples were run and met limits.	Data Validator	Step IIa	External
Documentation of Field QC Sample Results	Establish that all required QC samples were run, met limits, and will be discussed in the associated project report.	PM/Town	Step IIa	Internal



Data Review Input	Description <sup>a</sup>	Responsible for Verification or Validation	Step I/IIa/IIb <sup>b</sup>	Internal/External <sup>c</sup>
DoD ELAP Evaluation	Ensure that each laboratory is DoD ELAP certified for the analyses they are to perform. Ensure evaluation timeframe does not expire.	PM/Town	Step I	External
Analytical data for PFAS analyzed in drinking water	<p>Analytical methods as presented in this SAP and laboratory SOPs will be used to evaluate compliance against QA/QC criteria. Should adherence to QA/QC criteria yield deficiencies, data may be qualified.</p> <p>Guidelines and qualifiers from United States Department of Defense <i>General Data Validation Guidelines</i> (DoD, 2019) and EPA's <i>Data Review and Validation Guidelines for Perfluoroalkyl Substances (PFASs) Analyzed Using EPA Method 537</i> (EPA, 2018) will be applied as appropriate. <i>Per- and Polyfluoroalkyl Substances (PFAS): Reviewing Analytical Methods Data for Environmental Samples</i> (EPA, 2019a) may also be referenced. As specific modules for the analytical methods in this project are published, the data validators will refer to those modules for guidance.</p>	Data Validator	Step IIa and IIb	External

Notes:

- <sup>a</sup> Verification (Step I) is a completeness check that is performed before the data review process continues to determine whether the required information (complete data package) is available for further review. Validation (Step IIa) is a review that the data generated is in compliance with analytical methods, procedures, and contracts. Validation (Step IIb) is a comparison of generated data against measurement performance criteria in the SAP (both sampling and analytical).
- <sup>b</sup> Internal or external is in relation to the data generator.
- <sup>c</sup> Should the FTL or PM find discrepancies during the verification or validation procedures above, an email documenting the issue will be circulated to the Navy RPM.

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## Worksheet #37—Usability Assessment

### **Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:**

Non-detected site contaminants will be evaluated to ensure that LOQs, LODs, and DLs in Worksheet #15 were achieved. If LOQs, LODs, and DLs were achieved and the verification and validation steps yielded acceptable data, then the data are considered usable.

During verification and validation steps, data may be qualified as estimated with the following qualifiers:

- U = The analyte was not detected and was reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
- 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.
- N = The analysis indicates the presence of an analyte for which there was presumptive evidence to make a "tentative identification."
- NJ = The analyte has been "tentatively identified" or "presumptively" as present and the associated numerical value was the estimated concentration in the sample.
- 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.
- 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.

Analytical data will be checked to ensure the values and any qualifiers are appropriately transferred to the electronic database. The checks include comparison of hard copy data and qualifiers to the EDD. Once the data has been uploaded into the electronic database, another check will be performed to ensure all results were loaded accurately.

Field and laboratory precision will be compared as RPD between the two results. The project team must consider the greater of results between field duplicates.

Deviations from the SAP will be reviewed to assess whether CA is warranted and to assess impacts on the achievement of project objectives.

If significant biases are detected with laboratory QA/QC samples, they will be evaluated to assess impacts on decision making. Low biases will be described in greater detail as they represent a possible inability to detect compounds that may be present at the site.

If significant deviations are noted between laboratory and field precision, the cause will be further evaluated to assess impact on decision making.

## Data Quality Indicators

Quantifiable criteria, known as MPC, are presented in **Worksheet #12**. The precision, accuracy, representativeness, completeness, comparability and sensitivity (PARCCS) criteria will be the qualitative and quantitative indicators of data quality. The PARCCS criteria are defined and discussed as follows.

### Precision

Precision is a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision will be measured by using field duplicates, LFSM/LFSMD and matrix spike/matrix spike duplicate samples. It will be expressed in terms of the RPD as follows:

$$RPD = \frac{|C_1 - C_2|}{(C_1 + C_2)/2} \times 100$$

Where:

- RPD = relative percent difference
- C<sub>1</sub> = concentration of sample or LFSM
- C<sub>2</sub> = concentration of sample or LFSMD

### Precision

Accuracy is the degree of agreement of an observed measurement (or an average of the same measurement type) with an accepted reference or true value. Accuracy of analytical determinations will be measured using laboratory QC analyses such as laboratory control samples and surrogate spikes. Accuracy will be measured by evaluating the actual result against the known concentration added to a spiked sample and will be expressed as %R as shown below:

$$\%R = \frac{S - U}{C_{sa}} \times 100$$

Where:

- %R = percent recovery
- S = measured concentration of spiked aliquot
- U = measured concentration of unspiked aliquot
- C<sub>sa</sub> = concentration of spike added

### Representativeness

Representativeness is the reliability with which a measurement or measurement system reflects the true conditions under investigation. Representativeness is influenced by the number and location of the sampling points, sampling timing and frequency of monitoring efforts, and the field and laboratory procedures. The representativeness of data will be maintained by the use of established field and laboratory procedures and their consistent application.

### Comparability

Comparability expresses the confidence with which one dataset can be compared to another based on using EPA defined procedures, where available. If EPA procedures are not available, the procedures have been defined or referenced in the SAP.

The comparability of data will be established through well documented methods and procedures, standard reference materials, QC samples, performance evaluation study results, and by reporting each data type in consistent units.

### **Completeness**

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions. Analytical data validation and data quality assessment will determine which data will be valid and which data will be rejected. Percent completeness will be defined as follows:

$$\text{Percent Completeness} = \frac{V}{T} \times 100$$

Where:

V = Number of valid (not rejected) measurements over a given time  
S = Total number of planned measurements

The completeness goal for this project will be 100 percent for valid, usable data. If the completeness goal of the project is not achieved, any rejected or failed samples will be recollected.

### **Sensitivity**

Sensitivity is the measure of a concentration at which an analytical method can positively identify and report analytical results. The sensitivity of an analytical method will be indicated by the project - required LOQs, LODs, and DLs, as compared to the PALs.

**Describe the documentation that will be generated during the usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:**

A data table will be produced to reflect detected and non - detected analytes. Data qualifiers will be reflected in the tables and discussed in the data quality evaluation.

**Identify the personnel responsible for performing the usability assessment.**

The PM, Project Chemist, and other team members will be responsible for compiling the data. The data will then be presented to the Navy and stakeholders who will evaluate the data usability according to project objectives.

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# References

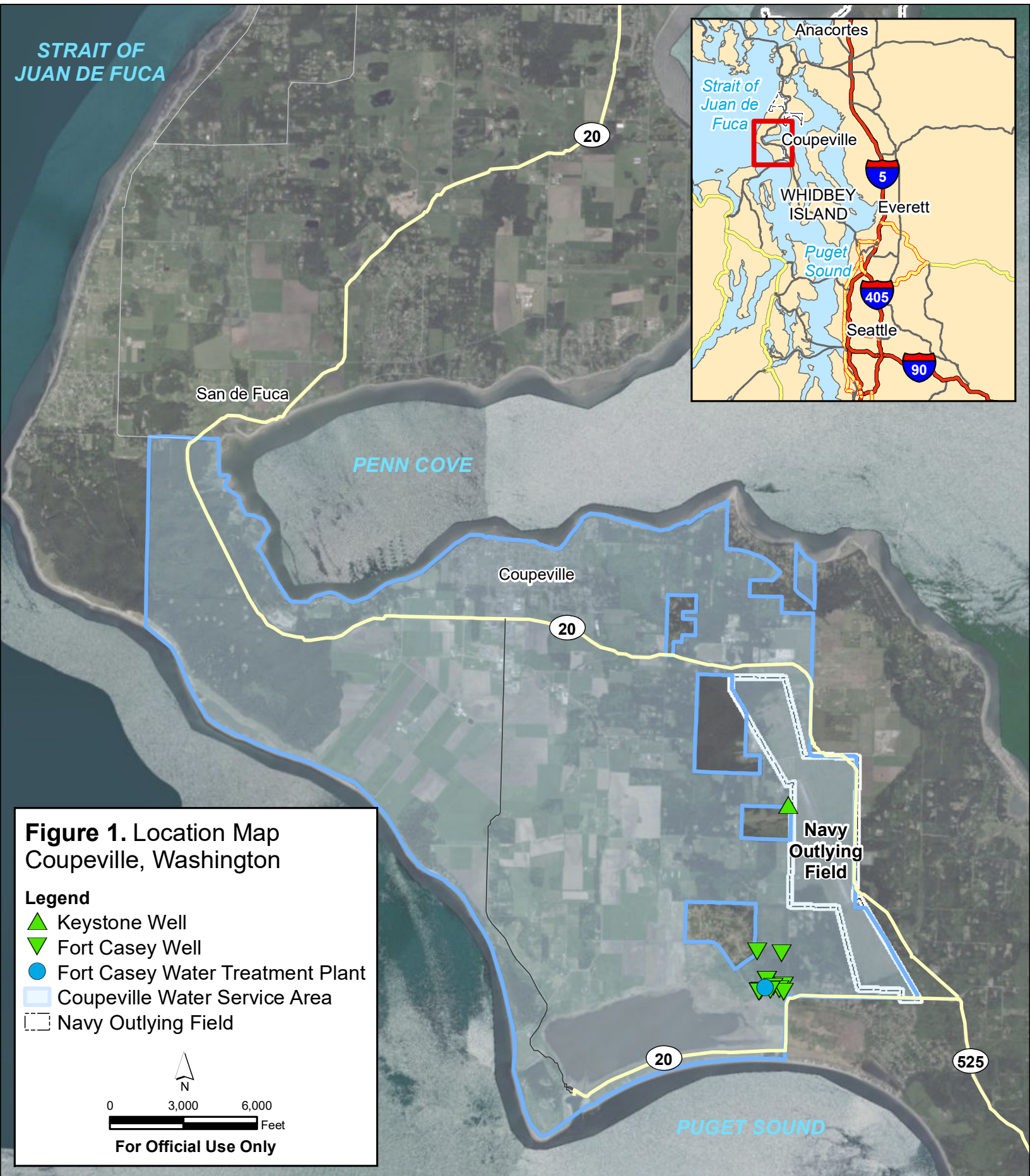
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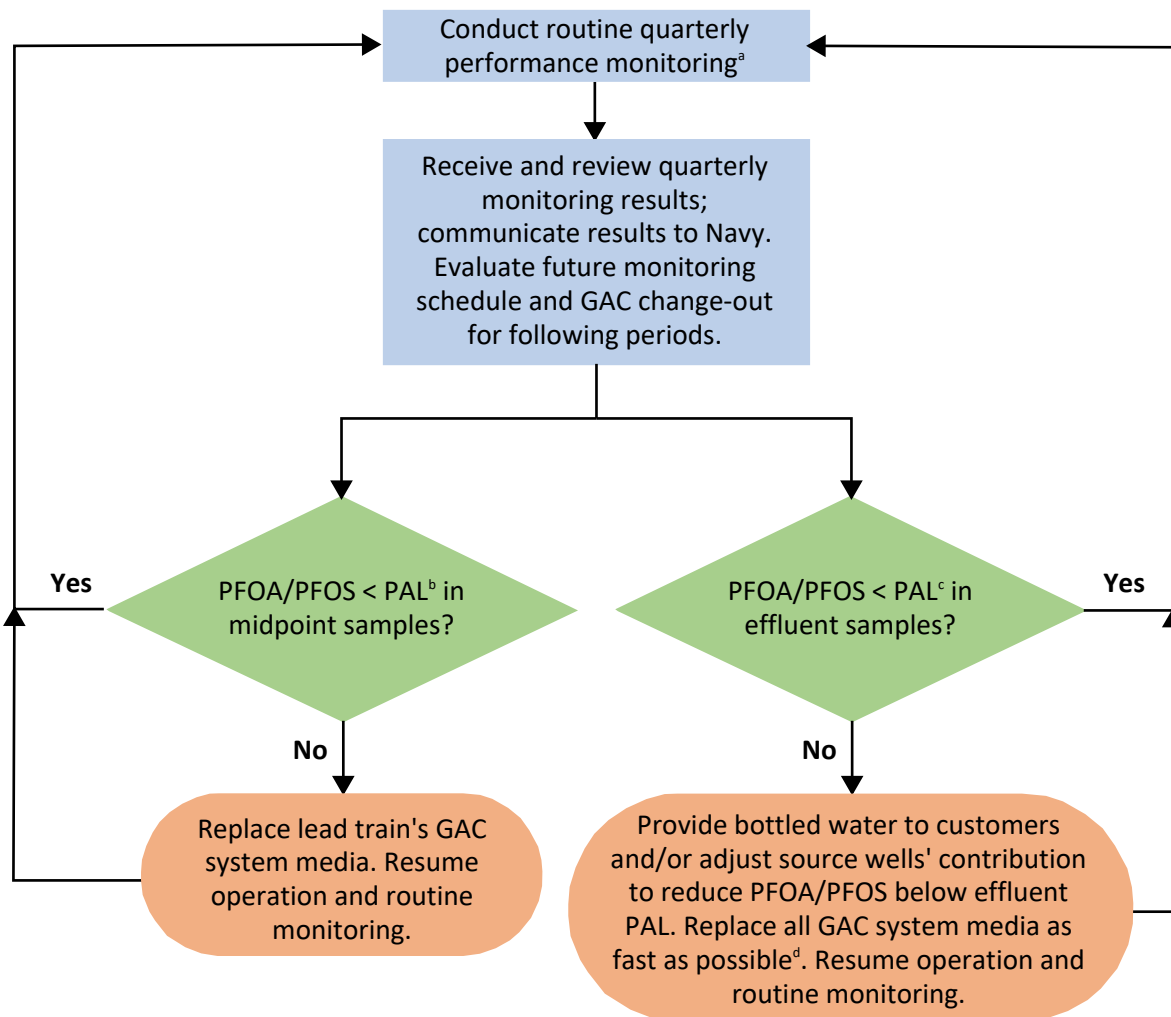
Figures

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**Figure 2. Proposed FCWTP Performance Monitoring Schedule and Decision Flowchart**



Notes:

GAC = granular activated carbon  
 PFAS = per- and polyfluoroalkyl substances  
 PFOA = perfluorooctanoic acid  
 PFOS = perfluorooctane sulfonate  
 PAL = project action limit (see Worksheet #11)

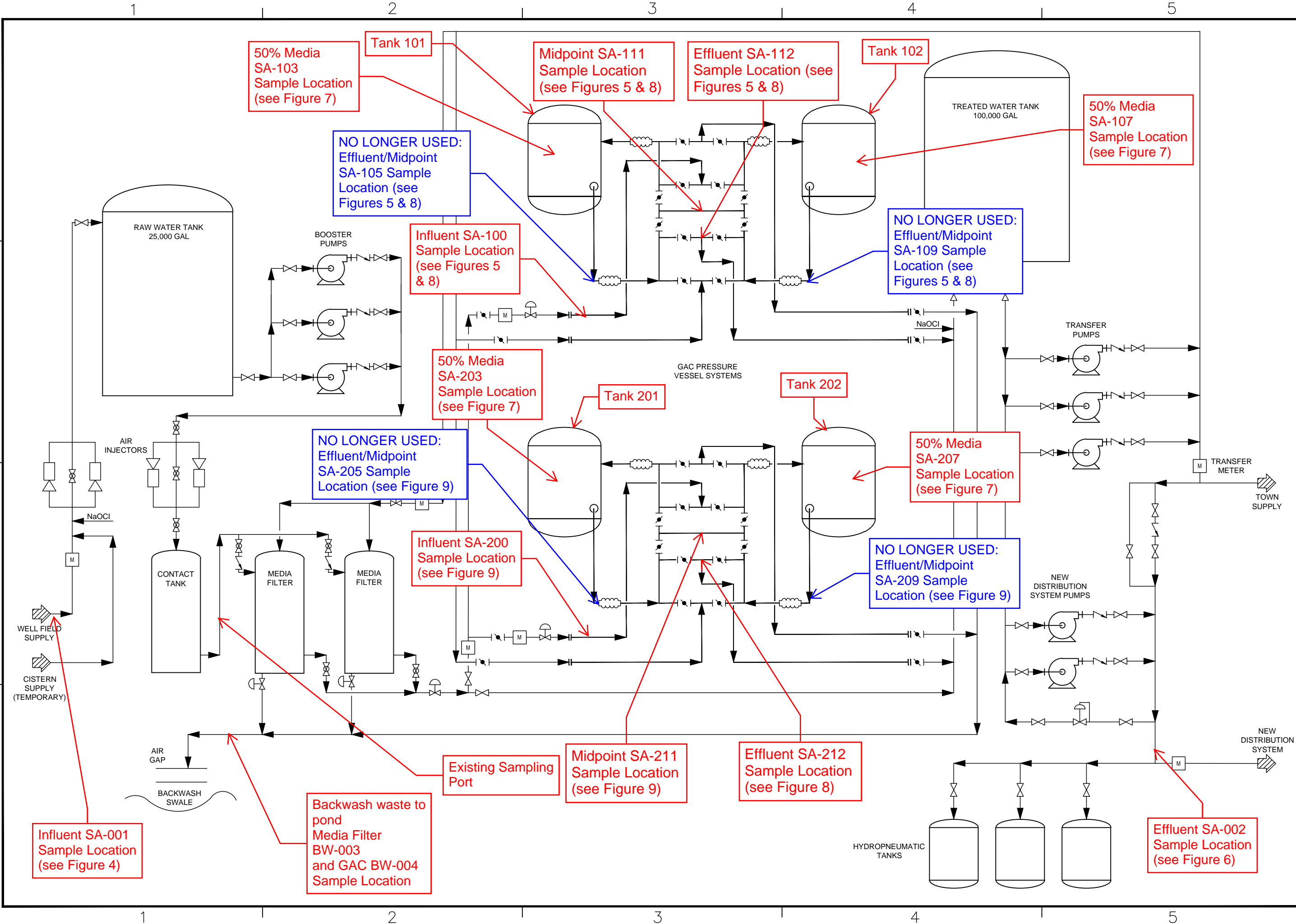
<sup>a</sup> Request 2-week turn around time. Each monitoring event consists of sampling and analysis for PFAS at twelve locations: three influent, two midpoints, four 50% media, and three effluent. Influent samples will be compared to the project action level (for PFOA/PFOS) and used for mass loading calculations. Midpoint and effluent samples will be used as specified in the flow diagram.

<sup>b</sup> The midpoint PAL of 35 micrograms per liter (µg/L) (which is half the effluent PAL) for the combined concentration of PFOS/PFOA will be used to trigger treatment media change out for this project because there will be at least a 10-day lag time between sample collection, laboratory result receipt, and GAC unit onsite maintenance. Using the midpoint PAL as the trigger will be conservative to keep the Town of Coupeville's municipal water supply below the target goal of less than the effluent PAL for PFOS/PFOA. Exceeding the midpoint PAL is not expected and would indicate that the GAC media capacity is < 18 months or that there is a possible hydraulic problem.

<sup>c</sup> The effluent PAL is 70 micrograms per liter (µg/L) for the combined concentration of PFOS/PFOA. It is extremely unlikely that the effluent will exceed this effluent PAL given the current influent PFOA/PFOS concentration and quarterly midpoint monitoring.

<sup>d</sup> The emergency bottled water would be provided under one of the Navy's periodic drinking water well sampling and bottled water contract task orders until GAC media replacement is complete. Emergency changeout for all GAC system media is expected to take 2 weeks but will be expedited if possible.

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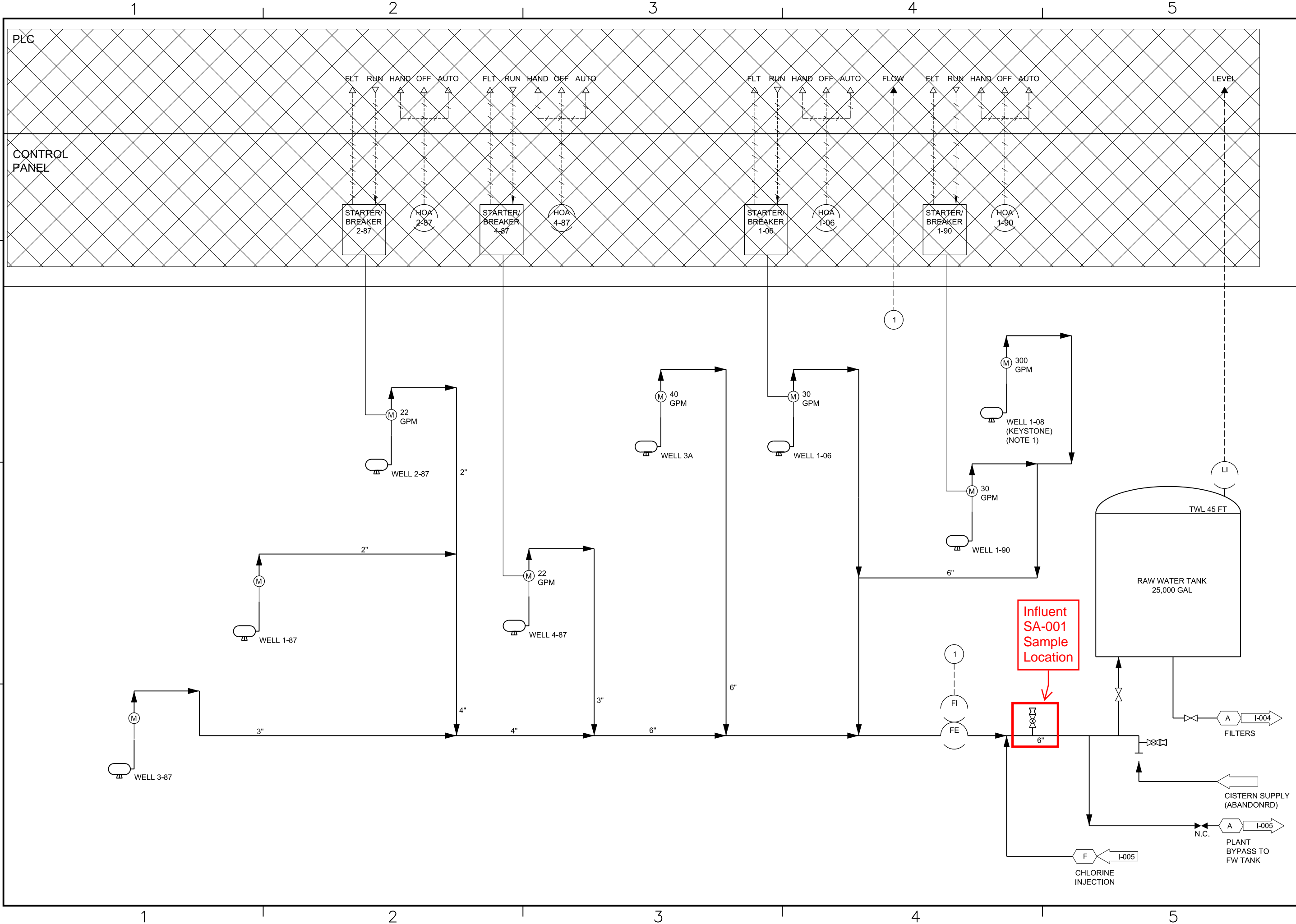
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FOR COMMANDER NWFAC		
ACTIVITY		
SATISFACTORY TO DATE		
DESIGNER: AR	DRAWN: DS	CHECKED: GH
PROJECT NO.: 695610		
CONSTR. CONTR. NO.: N62470-16-D-9004		
TASK ORDER NO.: N6247018F4160		
SHEET 56 OF 117		

DEPARTMENT OF THE NAVY  
 NAVAL FACILITIES ENGINEERING COMMAND NORTHWEST  
 WATER TREATMENT PLANT  
 WHIDBEY ISLAND, WA  
**TOWN OF COUPEVILLE**  
**WATER SUPPLY IMPROVEMENTS**  
 PROCESS MECHANICAL  
 PROCESS FLOW DIAGRAM

SCALE: AS NOTED  
 PROJECT NO.: 695610  
 CONSTR. CONTR. NO.: N62470-16-D-9004  
 TASK ORDER NO.: N6247018F4160  
 SHEET 56 OF 117  
**Figure 3**  
 DATE: MARCH 2019







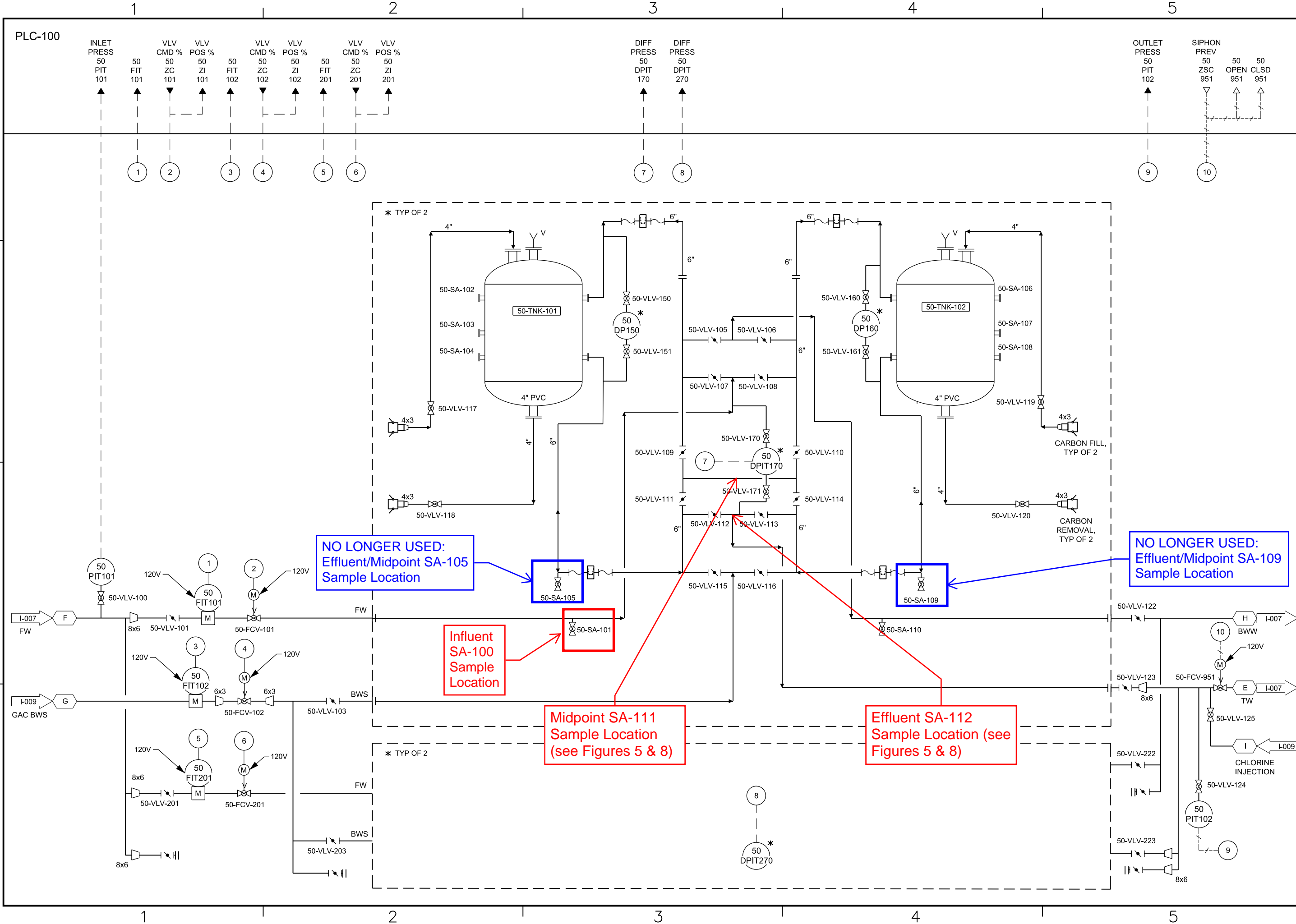
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ACTIVITY		
SATISFACTORY TO	DATE	
DES	JT	DRW
VM	CHK	SB
PA/DM		
BRANCH MANAGER		
CHIEF ENG/ARCH		
FIRE PROTECTION		
DEPARTMENT OF THE NAVY NAVAL FACILITIES ENGINEERING COMMAND NORTHWEST WATER TREATMENT PLANT <b>TOWN OF COUPEVILLE</b> <b>WATER SUPPLY IMPROVEMENTS</b> INSTRUMENTATION AND CONTROL DEMO WELLS SYSTEM P&ID		
SCALE:	NONE	
EPROJECT NO.:	695610	
CONSTR. CONTR. NO.:	N62470-16-D-9004	
TASK ORDER NO.:	N6247018F4160	
SHEET	88	OF 117
<b>Figure 4</b> DATE: MARCH 2019		



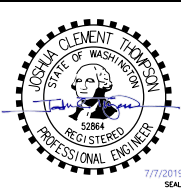
A/E INFO

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ACTIVITY		
SATISFACTORY TO	DATE	
DES	JT	DRW
VM	CHK	SB
PA/DM		
BRANCH MANAGER		
CHIEF ENG/ARCH		
FIRE PROTECTION		





APPR	DATE
SYN	DESCRIPTION



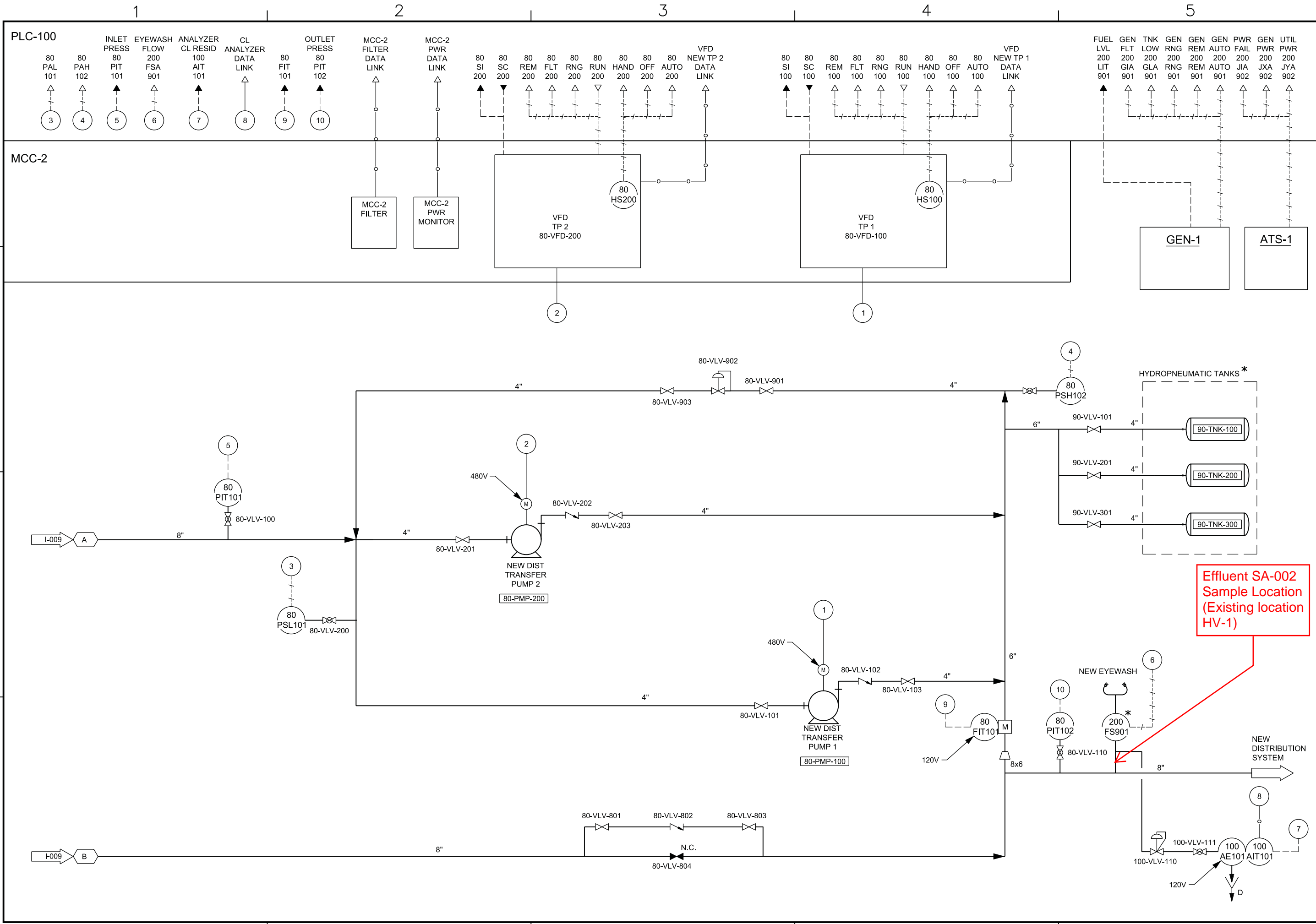
APPROVED	DATE				
FOR COMMANDER NAVFAC	ACTIVITY				
SATISFACTORY TO	DATE				
DES	JT	DRW	VM	CHK	SB
PM/TM	BRANCH MANAGER	CHIEF ENG/ARCH	FIRE PROTECTION		

DEPARTMENT OF THE NAVY  
 NAVAL FACILITIES ENGINEERING COMMAND NORTHWEST  
 WATER TREATMENT PLANT  
 WHIDBEY ISLAND, WA  
**TOWN OF COUPEVILLE**  
 WATER SUPPLY IMPROVEMENTS  
 INSTRUMENTATION AND CONTROL  
 NEW GAC SYSTEM P&ID

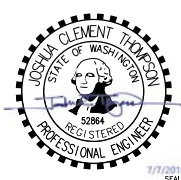
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CONSTR. CONTR. NO.:	N62470-16-D-9004
TASK ORDER NO.:	N6247018F4160
SHEET	93 OF 117

**Figure 5**





APPROVED	DATE	APPR
FOR COMMANDER NMFAC	ACTIVITY	DESCRIPTION
SATISFACTORY TO	DATE	DATE
DES	JT	DRW
VM	CHK	SB
PW/DM		
BRANCH MANAGER		
CHIEF ENG/ARCH		
FIRE PROTECTION		
DEPARTMENT OF THE NAVY NAVAL FACILITIES ENGINEERING COMMAND NAVAL FACILITIES ENGINEERING COMMAND NORTHWEST WATER TREATMENT PLANT <b>TOWN OF COUPEVILLE</b> <b>WATER SUPPLY IMPROVEMENTS</b> INSTRUMENTATION AND CONTROL NEW FW TANK AND TRANSFER PUMP SYSTEM P&ID 2		
SCALE:	NONE	
PROJECT NO.:	695610	
CONSTR. CONTR. NO.:	N62470-16-D-9004	
TASK ORDER NO.:	N6247018F4160	
SHEET	95	OF 117
<b>Figure 6</b> DATE: MARCH 2019		



A/E INFO

FOR COMMANDER NMFAC

ACTIVITY

SATISFACTORY TO DATE

DES JT DRW VM CHK SB

PW/DM

BRANCH MANAGER

CHIEF ENG/ARCH

FIRE PROTECTION

DEPARTMENT OF THE NAVY

NAVAL FACILITIES ENGINEERING COMMAND

NAVAL FACILITIES ENGINEERING COMMAND NORTHWEST

WATER TREATMENT PLANT

WHIDBEY ISLAND, WA

**TOWN OF COUPEVILLE**

**WATER SUPPLY IMPROVEMENTS**

INSTRUMENTATION AND CONTROL

NEW FW TANK AND TRANSFER PUMP SYSTEM P&ID 2

SCALE: NONE

PROJECT NO.: 695610

CONSTR. CONTR. NO. N62470-16-D-9004

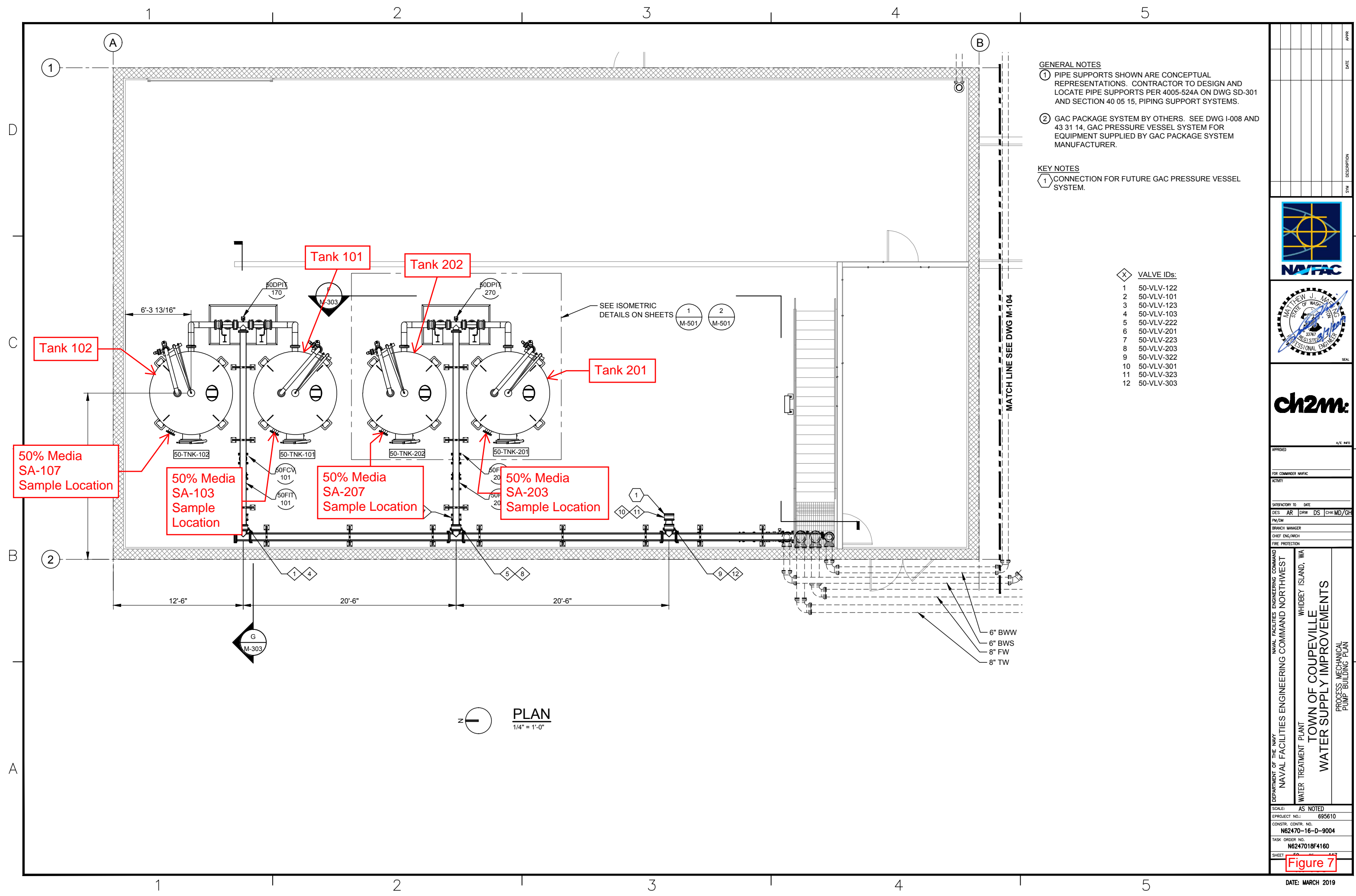
TASK ORDER NO. N6247018F4160

SHEET 95 OF 117

DATE: MARCH 2019



FILE NAME: C:\pwworkdir\pwworkdir\ch2mhill\_russell\0250335\TDC-M-103\_695610.dwg LAYOUT NAME: Layout1 PLOTTED: Wednesday, March 06, 2019 - 9:27am USER: russellr



**GENERAL NOTES**

- PIPE SUPPORTS SHOWN ARE CONCEPTUAL REPRESENTATIONS. CONTRACTOR TO DESIGN AND LOCATE PIPE SUPPORTS PER 4005-524A ON DWG SD-301 AND SECTION 40 05 15, PIPING SUPPORT SYSTEMS.
- GAC PACKAGE SYSTEM BY OTHERS. SEE DWG I-008 AND 43 31 14, GAC PRESSURE VESSEL SYSTEM FOR EQUIPMENT SUPPLIED BY GAC PACKAGE SYSTEM MANUFACTURER.

**KEY NOTES**

- CONNECTION FOR FUTURE GAC PRESSURE VESSEL SYSTEM.

**VALVE IDs:**

1	50-VLV-122
2	50-VLV-101
3	50-VLV-123
4	50-VLV-103
5	50-VLV-222
6	50-VLV-201
7	50-VLV-223
8	50-VLV-203
9	50-VLV-322
10	50-VLV-301
11	50-VLV-323
12	50-VLV-303

APPROVED	DATE	APP'R
FOR COMMANDER NWAFAC		
ACTIVITY		
SATISFACTORY TO DATE		
DES AR	DRW DS	CHK MD/CH
PM/DM		
BRANCH MANAGER		
CHIEF ENG/ARCH		
FIRE PROTECTION		
DEPARTMENT OF THE NAVY NAVAL FACILITIES ENGINEERING COMMAND NORTHWEST WATER TREATMENT PLANT TOWN OF COUPEVILLE WATER SUPPLY IMPROVEMENTS PROCESS MECHANICAL PUMP BUILDING PLAN		
SCALE:	AS NOTED	
PROJECT NO.:	695610	
CONSTR. CONTR. NO.	N62470-16-D-9004	
TASK ORDER NO.	N6247018F4160	
SHEET	447	
<b>Figure 7</b>		
DATE: MARCH 2019		





# GAC 100 Series

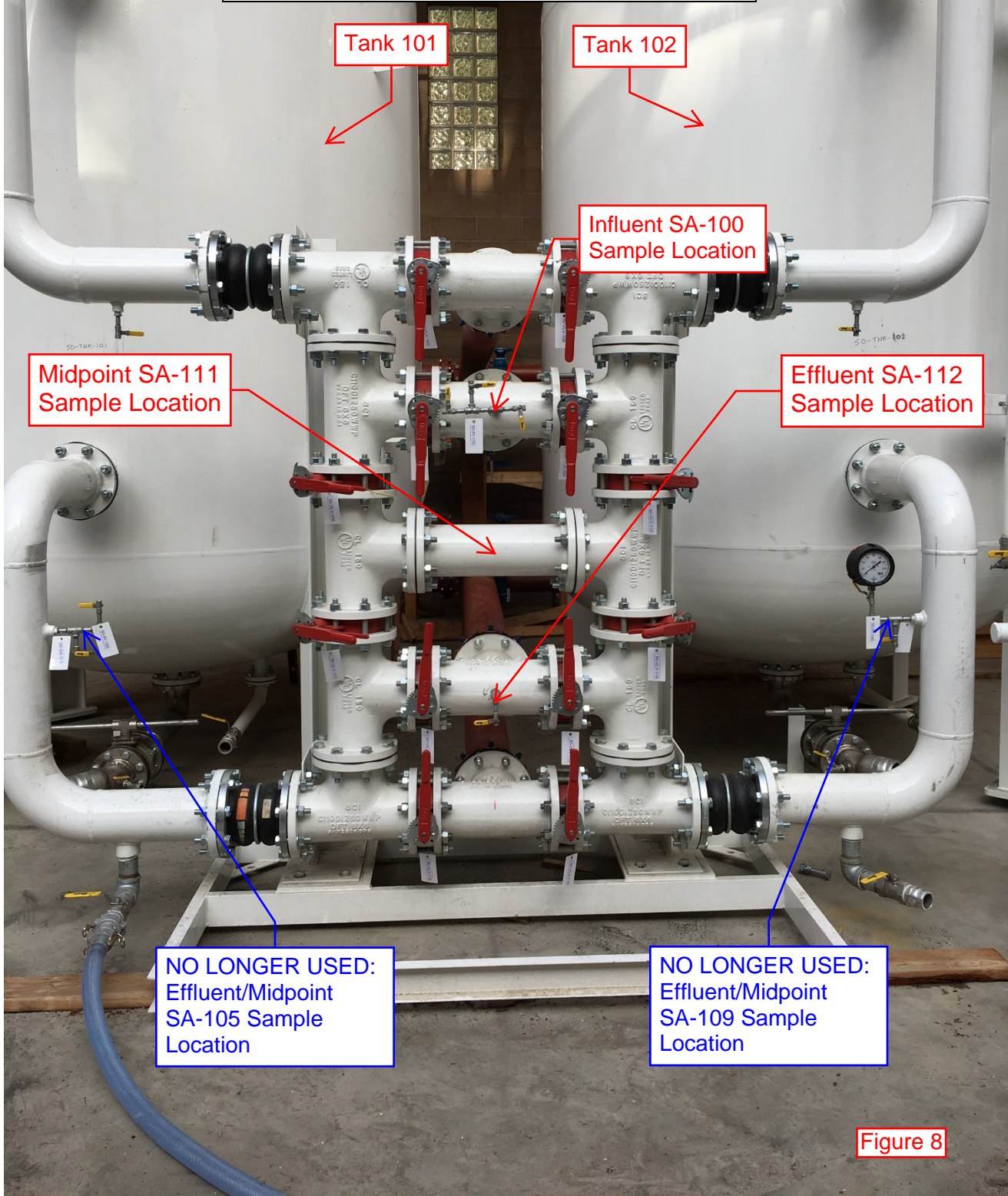


Figure 8

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# GAC 200 Series

Tank 201

Tank 202

Influent SA-200  
Sample Location

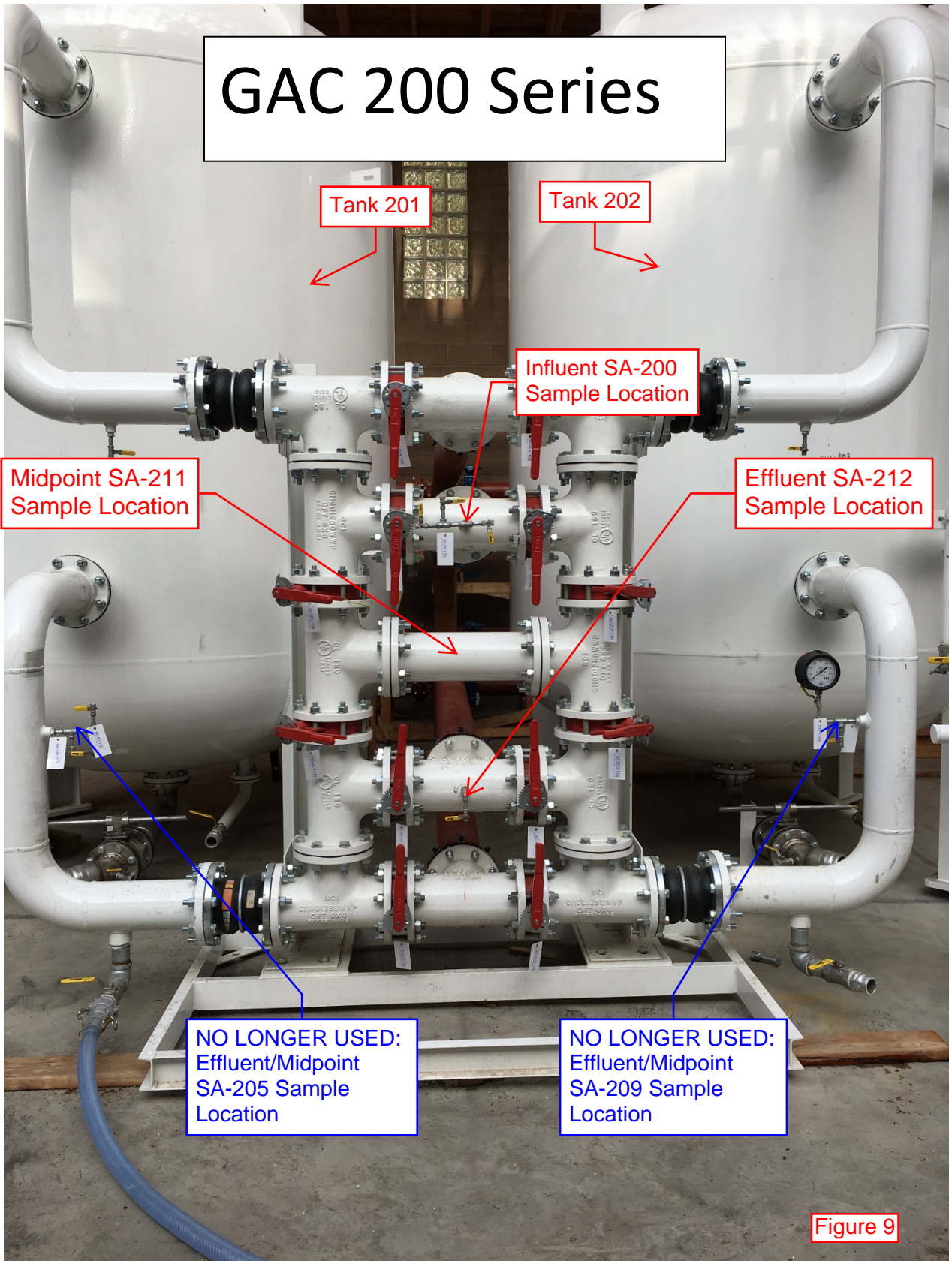
Midpoint SA-211  
Sample Location

Effluent SA-212  
Sample Location

NO LONGER USED:  
Effluent/Midpoint  
SA-205 Sample  
Location

NO LONGER USED:  
Effluent/Midpoint  
SA-209 Sample  
Location

Figure 9



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Appendix A  
APTIM Field Standard Operating  
Procedures

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# WORK INSTRUCTION

Work Instruction Title:	Shipping and Packaging of Non Hazardous Samples	AMS Number:	CFS-830-19-WI-30001
Work Instruction Owner:	Field Sampling Discipline Lead	Issuing Authority:	APTIM Quality Management

## SHIPPING AND PACKAGING OF NON HAZARDOUS SAMPLES

INT	Issued for Interim Use	M. Hadacek & S. Lachney	7/30/2017
<b>Rev</b>	<b>Changes</b>	<b>Approved</b>	<b>Date</b>



# Shipping and Packaging of Non Hazardous Samples

<b>AMS Number:</b>	<b>Revision:</b>	<b>Approval Date:</b>
<b>CFS-830-19-WI-30001</b>	<b>INT</b>	<b>7/30/2017</b>

## 1.0 PURPOSE

This document provides general instructions in the packaging and shipping of non-hazardous samples. The primary use of this procedure is for the transportation of samples collected on site to be sent off site for physical, chemical, and/or radiological analysis.

## 2.0 APPLICATION

This instruction applies to the shipping and packaging of all non-hazardous samples. Non-hazardous samples are those that do not meet any hazard class definitions found in 49 CFR 107-178, including materials designated as class 9 materials and materials that represent reportable quantities (hazardous substances) and/or materials that are not classified as Dangerous Goods under current IATA regulations. Such materials must be packaged and shipped per AMS-830-19-GL-30001.

In general most soil, air, and aqueous samples, including those that are acid or caustic preserved do not qualify as hazardous materials or Dangerous Goods. An exception is methanolic soil VOC vials: these containers are flammable in any quantity and must be packaged, shipped, and declared as Dangerous Goods whenever transported by air.

The class 9 “environmentally hazardous” designation should only be applied to samples if they are known or suspected (via screening) to contain a sufficient concentration of contaminant to pose a health and/ or environmental risk if spilled in transport. Samples for which screening has shown a potential hazard (i.e., Flammability) or those that are derived from a known hazard, including a site/facility with confirmed contamination by an infectious substance must also be shipped in accordance with the applicable DOT/IATA requirements

Improper shipment of hazardous materials, especially wilful misrepresentation and shipment as non-hazardous materials, is a violation of federal law and is punishable by fines and possible imprisonment of the guilty parties. It is also a violation of APTIM policy and can result in disciplinary action up to and including termination of employment.

## 3.0 REQUIREMENTS

1. Double-walled insulated cooler. Samples should not be packaged into Playmate™ type single-walled coolers as these will rarely maintain temperature during overnight transport
2. Packing/shipping tape
3. Custody seals or the materials to make them
4. Plastic zip bags 1-gallon and 1-quart size-bubble type if available
5. Ice-at least one large bag per cooler-separated into 1-gallon zip bags
6. Bubble wrap or other packing material.
7. Large plastic trash bag

Project Managers and Technical Leads should provide the project sampling plans to those packing any coolers to ensure that any project-specific requirements are known and complied with. Individuals performing this task are required to read and understand this instruction and any project-specific requirements.

## 4.0 REFERENCES

AMS-830-19-PR-30001	Field Sampling and Analysis in Support of Investigation and Remediation
AMS-710-04-PR-01106	Chain of Custody
AMS-830-19-GL-30001	Guidelines For Packaging and Shipping Of DOT/IATA Hazardous Samples
AMS-830-19-FM-30002	Sample Shipment Checklist





# Shipping and Packaging of Non Hazardous Samples

AMS Number:	Revision:	Approval Date:
CFS-830-19-WI-30001	INT	7/30/2017

U.S. Army Corps of Engineers, 2001, *Requirements for the Preparation of Sampling and Analysis Plans*, EM200-1-3, Washington, D.C.

U.S. Department of Transportation Regulations, 49 CFR Parts 108-178

International Air Transport Association (IATA), *Dangerous Goods Regulations*, current edition.

## 5.0 WORK INSTRUCTION

### 5.1 Packaging

- Use tape and seal off the cooler drain on the inside and outside to prevent leakage.
- Place packing material on the bottom of the shipping container (cooler) to provide a soft impact surface.
- Place a large (30-55 gallon or equivalent) plastic bag into the cooler (to minimize possibility of leakage during transit).
- Each cooler should have a dedicated Chain of Custody, use continuation forms if necessary to document all samples in the cooler.
- Cross-check the sample labels against the Chain of Custody as the cooler is packed.
- Starting with the largest glass containers, wrap each container with sufficient bubble wrap to ensure the best chance to prevent breakage of the container.
- Pack the largest glass containers in the bottom of the cooler, placing packing material between each of the containers to avoid breakage from bumping.
- Double-bag the ice (chips or cubes) in gallon- or quart-sized resealable plastic freezer bags and wedge the ice bags between the sample bottles.
- Add bagged ice across the top of the samples.
- When sufficiently full, seal the inner protective plastic bag, and place additional packing material on top of the bag to minimize shifting of containers during shipment.
- Tape a gallon-sized resealable plastic bag to the inside of the cooler lid, place the completed chain of custody document inside, and seal the bag shut.
- Tape the shipping container (cooler) shut using packing tape, duct tape, or other tear-resistant adhesive strips. Taping should be performed to ensure the lid cannot open during transport.
- Place a custody seal on two separate portions of the cooler, to provide evidence that the lid has not been opened prior to receipt by the intended recipient.

### 5.2 Labelling

- A "This Side Up" arrow should be adhered to all sides of the cooler, especially ones without obvious handles.
- The name and address of the receiver and the shipper must be on the top of the cooler.
- The air-bill must be attached to the top of the cooler.

### 5.3 Shipping Documentation

- A Sample Shipment Checklist (AMS-830-19-FM-30002) should be completed and kept in the project files.

## 6.0 EXHIBITS

Exhibit 6.1 AMS-720-01-FM-00020 Business Glossary

Exhibit 6.2 AMS-720-01-FM-00021 Technical Glossary



# WORK INSTRUCTION

Work Instruction Title:	<b>Decontamination of Contact Sampling Equipment in Support of Environmental Investigation and Remediation</b>	AMS Number:	<b>CFS-830-19-WI-30003</b>
Work Instruction Owner:	<b>Field Sampling Discipline Lead</b>	Issuing Authority:	<b>APTIM Quality Management</b>

## DECONTAMINATION OF CONTACT SAMPLING EQUIPMENT IN SUPPORT OF ENVIRONMENTAL INVESTIGATION AND REMEDIATION

INT	Issued for Interim Use	M. Hadacek & S. Lachney	7/30/2017
<b>Rev</b>	<b>Changes</b>	<b>Approved</b>	<b>Date</b>



# Decontamination of Contact Sampling Equipment in Support of Environmental Investigation and Remediation

AMS Number:	Revision:	Approval Date:
CFS-830-19-WI-30003	INT	7/30/2017

## 1.0 PURPOSE

The purpose of this document is to provide the methods and techniques to be utilized when decontaminating non-disposable contact sampling equipment either before/after use or in between sample collection efforts. Contact sampling equipment is equipment that comes in direct contact with the sample or the portion of a sample that will undergo analyses or physical testing.

## 2.0 APPLICATION

This work instruction applies to all APTIM efforts where environmental samples are being collected in the field in support of environmental investigation or remediation projects primarily for third parties. It is not applicable to general APTIM construction projects or facilities unless specifically referenced.

It is applicable when collecting samples for chemical, biological, and non-survey radiological testing as well as for field screening analysis using non-disposable direct contact sampling equipment. It is not intended to address decontamination of peristaltic or other sampling pumps and tubing or drill rig and DPT decontamination methods which are covered by other documents in AMS. It also does not apply to unexploded ordnance and radiological survey operations which have separate governing documents.

Attachment 1 has been modified from the U.S. Army Corps of Engineers Engineering Manual (EM-200-1-3), February 2001.

## 3.0 REQUIREMENTS

Project Managers and Project Technical Leads are responsible for making sure that sampling personnel have been provided and understand the necessary project plans, procedures, and quality control requirements before they commence with any sampling efforts.

## 4.0 REFERENCES

- AMS-830-19-PR-30001      Field Sampling and Analysis in Support of Environmental Investigation and Remediation
- US Army Corp of Engineers, Washington, D.C., 2001, Requirements for the Preparation of Sampling and Analysis Plans (EM-200-1-3), February.*

## 5.0 WORK INSTRUCTION

### 5.1 Planning

When planning a sample collection effort attention should be paid to the necessary decontamination of any non-disposable equipment utilized to collect, manipulate, or containerize the sample material. The following variables should be reviewed:

- Project specific requirements: The project plans will reflect any applicable client and/or regulator directed aspects such as whether or not an isolated and totally separate from the sampling area Decontamination Area is required. They may also provide an indication as to the "trace-nature" of the planned data usage or the data quality objectives which can influence decontamination approaches.
- Amount of non-disposal sampling equipment necessary to complete the task: Especially in remote project locations the best approach may be to have sufficient numbers of non-disposable sampling items available to collect all daily required samples such that the decontamination is only required on all used implements once per day. Ideally, sufficient sample collection, mixing, and containerization implements should be available to allow for sampling to proceed without the need to decontaminate after each individual effort as much as possible.



# Decontamination of Contact Sampling Equipment in Support of Environmental Investigation and Remediation

AMS Number:	Revision:	Approval Date:
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- Containerization, nature, and disposal of decontamination wastes: Decontamination wastes are considered to be Investigation Derived Waste (IDW) and must be managed, containerized, labelled, and disposed of in accordance with applicable requirements.

## 5.2 Decontamination Equipment and Materials

The following equipment and materials are recommended for decontamination of non-disposable sampling implements:

- Clean plastic liner or a large plastic tub to capture spillage
- Long handled brushes-to remove gross adhered materials
- Buckets-one for each required decontamination liquid type and rinse step. Alternatively, use spray bottles of appropriate materials, one per required decontamination liquid and capture the waste into a single bucket
- Tap Water or if unavailable store bought water
- Non-phosphate soap/detergent, mixed with tap or store bought water per instructions
- Absorbing agent solutions applicable to the analytes/parameters of interest see the project plan or Attachment 1. Acids/bases should be trace grade and all organic solvents should be pesticide grade or better.
- Final rinse solution; Analyte free Type II Reagent Grade or distilled water, per project plan.
- Paper towels; no print or perfume/scent, or lint free wipers
- Sample gloves
- Drums for waste and excess sample (investigation-derived waste)

## 5.3 Equipment Decontamination

At a minimum gross decontamination (physical removal of visible adhered material) should be completed each time implements are used; such as between grabs comprising a composite. More complete decontamination is performed between uses that represent the sample to be analysed. As an example, if a field composite is being created decontamination is not required between the distinct grabs each, but between the actual composites.

1. Complete gross decontamination with a brush or paper towel to remove visible adhered material-this is done in all instances
2. Don a pair of clean gloves.
3. Clean the implement with soap and water; dip and brush into a bucket or use a spray bottle to coat the implement while brushing/wiping any loosened material away
4. Rinse with tap or store bought water also by bucket or spray bottle
5. As necessary, rinse in the absorbing agent solutions. If a water insoluble organic solvent is used be sure to also rinse in alcohol so that the final rinse water will not bead up
6. Final rinse three times in the Reagent Grade or distilled water. If a Rinseate Blank is required, collect the residual of the last rinse as the sample, preserve as required for the parameters of interest
7. Shake off the excess liquid and either air dry on a clean surface (preferred) or wipe clean with paper towels/wipers (if needed quickly). If an Equipment Blank (clean sand sample) is required collect it with the freshly decontaminated implement(s)
8. Reuse or store for later use.



# Decontamination of Contact Sampling Equipment in Support of Environmental Investigation and Remediation

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## 6.0 EXHIBITS

- Exhibit 6.1           AMS-720-01-FM-00020 Business Glossary
- Exhibit 6.2           AMS-720-01-FM-00021 Technical Glossary

## 7.0 ATTACHMENTS

- Attachment 1- Recommended Decontamination Solutions



## Decontamination of Contact Sampling Equipment in Support of Environmental Investigation and Remediation

<b>AMS Number:</b>	<b>Revision:</b>	<b>Approval Date:</b>
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### ATTACHMENT 1 - RECOMMENDED DECONTAMINATION SOLUTIONS

Compound	Detergent Wash	Tap Water	Inorganic Desorbing Agent	Tap Water	Organic Desorbing Agent <sup>1</sup>	Final Water Rinse <sup>4</sup>	Air Dry
<b>Organic Constituents</b>							
Volatile Organic Compounds	✓	✓			Methanol Purge & Trap grade	✓	✓
Base Neutrals/Acid Extractable/PCBs/Pesticides	✓	✓			Hexane followed by Isopropyl Alcohol	✓	✓
Organic Bases <sup>2</sup>	✓	✓	1% nitric acid	✓	Isopropyl Alcohol	✓	✓
Organic Acids <sup>3</sup>	✓	✓	1% nitric acid		Isopropyl Alcohol	✓	✓
<b>Inorganic Constituents</b>							
Trace Metals and Radio Isotopes	✓	✓	10% Nitric acid -Trace metals grade	✓		✓	✓
Cations/Anions	✓	✓				✓	✓
Acidic Compounds	✓	✓				✓	✓
Basic Compounds (caustic)	✓	✓	1% nitric acid	✓		✓	✓
<b>Waste Profiling</b>							
All analytes-gross decontamination	✓	✓				✓	✓


1 – All organic solvents must be Pesticide Grade or better. The selection of appropriate solvent rinses should first consider if a *known or suspected* contaminant requires removal from sampling equipment. Secondly, identify whether the subsequent analytical protocol would be impacted by the proposed solvent or an impurity thereof (e.g., residual acetone present in isopropyl alcohol would be measured with certain volatile organics analysis).

2 - Organic bases include amines, hydrazines.

3 - Organic acids include phenols, thiols, nitro and sulfonic compounds.

4 - Use a grade of water appropriate to the application. For trace level analysis this must be Analyte Free Water. For non-trace applications store-bought distilled water is sufficient

Adapted from EM-200-1-3

	Document Type:	Level: 3
	<h1>Discipline-Specific Procedure</h1>	Owner: Applied Science & Engineering Origination Date: 6/5/2003 Revision Date: 8/25/2011
Group: <b>E&amp;I</b>	Title: <b>Field Logbook</b>	No: EID-FS-001 Revision No.: 2 Page 1 of 5

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## 1. PURPOSE

This procedure is intended to communicate the requirements for selection, use, and maintenance of all field logbooks. Field logbooks are often used to document observations, sampling information, and other pertinent information on project sites. They are considered legal documents and should be maintained and documented accordingly as part of the project file.

## 2. SCOPE

This procedure is applicable to all Shaw E & I site operations where field logbooks are utilized to document all site activities and pertinent information.

## 3. REFERENCES

- Nielsen Environmental Field School, 1997, *Field Notebook Guidelines*

## 4. DEFINITIONS

- **Significant detail**—Any piece and/or pieces of information or an observation that can be considered pertinent to the legal reconstruction of events, description of conditions, or documentation of samples and/or sampling procedures.
- **Significant event**—Any event or events that could influence or be considered pertinent to a specific task or function and therefore require documentation in the Field Logbook.
- **Field Logbook**—Logbooks used at field sites that contain detailed information regarding site activities that must include dates, times, personnel names, activities conducted, equipment used, weather conditions, etc. Field logbooks can be used by a variety of different field personnel and are part of the project file.

## 5. RESPONSIBILITIES

### 5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

### 5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

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## **6. PROCEDURE**

### **6.1 General**

Each site or operation, as applicable, will have one current Logbook, which will serve as an index of all activities performed at the site or in the task performance. The Logbook is initiated at the start of the first applicable activity. Summary entries are made for every day that covered activities take place. Multiple field logbooks may be used depending upon the number of different types of field personnel conducting work and the various activities at the site. These field logbooks and the site logbooks shall be made part of the project files.

Information recorded in field logbooks includes observations (significant events and details), data, calculations, time, weather, and descriptions of the data collection activity, methods, instruments, and results. Additionally, the field logbook may contain descriptions of wastes, biota, geologic material, and site features including sketches, maps, or drawings as appropriate.

### **6.2 Equipment and Materials**

- Logbook(s), bound with numbered pages, hard-covered, waterproof preferred. One per project or separate significant task (example-treatment residual composite collection).
- Indelible black or dark blue ink pen
- Other items needed to perform required tasks: compass, ruler, calculator, etc.

### **6.3 Preparation**

Site personnel responsible for maintaining field logbooks must be familiar with the SOPs for all tasks to be performed.

Field logbooks are project files and should remain with project documentation when not in use. *Personnel should not keep Field logbooks in their possession when not in use. Field logbooks should only leave the project site for limited periods, and they should always be returned to the site files or the designated on-site location (Sampler's Trailer, etc.).*

Field logbooks shall be bound with lined, consecutively numbered pages. All pages must be numbered prior to initial use of the field logbook.

The front cover shall include the following information:

- Project Number
- Project Name and Task(s) included in logbook
- Dates covered by logbook—the starting date must be entered on the first day of use
- Logbook number—if more than one logbook will be needed to cover project/task(s)

The inside front cover shall contain a listing and sign-off of each person authorized to make entries and/or review the logbook. All persons who make entries or review/approve such entries must signify their authority to enter into the logbook via their signature and the date of their signing on the inside front cover. If initials are used for entries instead of full names, the initials must be entered beside the full name on the inside cover.

### **6.4 Operation**

The following requirements must be met when using a field logbook:

- Record significant details and/or events, work, observations, material quantities, calculations, drawings, and related information directly in the field logbook. If data-collection forms are in



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use, the information on the form need not be duplicated in the field logbook. However, any forms used to record site information *must be referenced* in the field logbook.

- Information must be factual and unbiased.
- Do not start a new page until the previous one is full or has been marked with a single diagonal line so that additional entries cannot be made. Use both sides of each page.
- Write in black or dark blue indelible ink.
- Do not erase, scribble over, or blot out any entry. Do not use White-Out or like correction items. Before an entry has been signed and dated, changes may be made; however, care must be taken not to obliterate what was written originally. Indicate any deletion by a single line through the material to be deleted. Any change shall be initialed and dated. Error codes (Attachment 1) should be added to the end of the deleted entry. All error codes should be circled.
- Do not remove any pages from the book.
- Do not use loose paper and copy into the field logbook later.
- Record sufficient information to completely document field activities and all significant details/events applicable to the project/task(s) covered by the logbook.
- All entries should be neat and legible.

Specific requirements for field logbook entries include the following:

- Initial and date each page.
- Sign and date the final page of entries for each day.
- Initial, date, and if used, code all changes properly.
- Draw a diagonal line through the remainder of the final page at the end of the day.
- Record the following information on a daily basis:
  - a) Date and time
  - b) Name of individual making entry
  - c) Detailed description of activity being conducted including well, boring, sampling, location number as appropriate
  - d) Unusual site conditions
  - e) Weather conditions (i.e., temperature, cloud cover, precipitation, wind direction and speed) and other pertinent data
  - f) Sample pickup (chain-of-custody form numbers, carrier, time)
  - g) Sampling activities/sample log sheet numbers
  - h) Start and completion of borehole/trench/monitoring well installation or sampling activity
  - i) Health and Safety issues, such as PPE upgrades, monitoring results, near-misses, and incidents associated with the logbook areas
  - j) Instrumentation calibration details

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Entries into the field logbook shall be preceded with the time of the observation. The time should be recorded frequently and at the point of events or measurements that are critical to the activity being logged. All measurements made and samples collected must be recorded unless they are documented by automatic methods (e.g., data logger) or on a separate form required by an operating procedure. In such cases, the field logbook must reference the automatic data record or form.

While sampling, make sure to record observations such as color and odor. Indicate the locations from which samples are being taken, sample identification numbers, the order of filling bottles, sample volumes, and parameters to be analyzed. If field duplicate samples are being collected, note the duplicate pair sample identification numbers. If samples are collected that will be used for matrix spike and/or matrix spike/matrix spike duplicate analysis, record that information in the field logbook.

A sketch of the station location may be warranted. All maps or sketches made in the field logbook should have descriptions of the features shown and a direction indicator. There must be at least one fixed point with measurements on any map drawn. Maps and sketches should be oriented so that north is towards the top of the page.

Other events and observations that should be recorded include (but are not limited to) the following:

- Changes in weather that impact field activities
- Visitors to the site associated with the covered task(s). Note their time of arrival and departure and provide a brief summary of their purpose on site.
- Subcontractor activities applicable to the covered task(s)
- Deviations from procedures outlined in any governing documents, including the reason for the deviation. Deviations from procedures must be accompanied with the proper authorization.
- Significant events that may influence data, such as vehicles in the vicinity of VOC sampling efforts
- Problems, downtime, or delays
- Upgrade or downgrade of personal protective equipment

## **6.5 Post-Operation**

To guard against loss of data due to damage or disappearance of field logbooks, all original completed logbooks shall be securely stored by the project. All field logbooks will be copied at the end of each work shift and attached to the daily reports.

At the conclusion of each activity or phase of site work, the individual responsible for the field logbook will ensure that all entries have been appropriately signed and dated and that corrections were made properly (single lines drawn through incorrect information, initialed, coded, and dated). The completed field logbook shall be submitted to the project records file.

## **6.6 Restrictions/Limitations**

Field logbooks constitute the official record of on-site technical work, investigations, and data collection activities. Their use, control, and ownership are restricted to activities pertaining to specific field operations carried out by Shaw personnel and their subcontractors. They are documents that may be used in court to indicate and defend dates, personnel, procedures, and techniques employed during site activities. Entries made in these notebooks should be factual,

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clear, precise, and as non-subjective as possible. Field logbooks, and entries within, are not to be utilized for personal use.

**7. ATTACHMENTS**

- Attachment 1, Common Data Error Codes

**8. FORMS**

None

**9. RECORDS**

- Field Logbook

**10. REVISION HISTORY AND APPROVAL**

Revision Level	Revision Description	Responsible Manager
Revision Date		
00	Initial Issue	N/A
6/5/2003		
01	New template, new numbering of procedure, Section 1 Purpose- content added, Section 2 edited, Section 4-Definitions edited. Sections 6.2, 6.3, 6.4, 6.5 and 6.6 were all edited.	Guy Gallelo
9/8/2006		
02	Modified format only to align with Governance Management framework	Scott Logan
8/25/2011		




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**Attachment 1  
Common Data Error Codes**

COMMON DATA ERROR CODES

- RE Recording Error
- CE Calculation Error
- TE Transcription Error
- SE Spelling Error
- CL Changed for Clarity
- DC Original Sample Description Changed After Further Evaluation
- WO Write Over
- NI Not Initialed and Dated at Time of Entry
- OB Not Recorded at the Time of Initial Observation

All Error Codes should be circled.

	Document Type: <h1>Discipline-Specific Procedure</h1>	Level: 3 Owner: Applied Science & Engineering Origination Date: 7/2/2003 Revision Date: 8/25/2011
Group: <b>E&amp;I</b>	Title: <b>Chain of Custody Documentation - Paper</b>	No: EID-FS-003 Revision No.: 2 Page 1 of 4

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## 1. PURPOSE

The purpose of this procedure is to provide the requirements for completion of written Chain of Custody (COC) documentation and to provide a suggested Chain of Custody Form for project use.

## 2. SCOPE

This procedure is applicable to all Shaw E & I efforts where samples are transferred among parties, including to off-site testing facilities. Adherence to this procedure is not required whenever the same individual/team is performing the sampling and testing within the same workday, and transfer to the testing process is being documented by other means, e.g. sampling and then field-screening in a mobile laboratory.

## 3. REFERENCES

- U.S. Environmental Protection Agency, 1986, *Test Methods for Evaluating Solid Waste; Physical/Chemical Methods, SW-846*, Third Edition.
- U.S. Army Corps of Engineers, *Requirements for the Preparation of Sampling and Analysis Plans*, EM200-1-3.
- Shaw E & I, 2002, *Sampler's Training Course Handout*.

## 4. DEFINITIONS

- **Custody**—The legal term used to define the control and evidence traceability of an environmental sample. A sample is considered to be in an individual's custody when it is in actual physical possession of the person, is in view of the person, is locked in a container controlled by the person, or has been placed into a designated secure area by the person.
- **Chain of Custody Form**—A form used to document and track the custody and transfers of a sample from collection to analysis or placement in a designated secure area within the testing facility.
- **COC Continuation Page**—Additional page(s) that may be included with a Chain of Custody form. The continuation page(s) contain the information on additional samples contained within the *same* cooler/shipping container associated with the cooler/shipping container Chain of Custody form.

## 5. RESPONSIBILITIES

### 5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

### 5.2 Project Responsibility

Shaw E & I employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

Group: <b>E&amp;I</b>	Title: <b>Chain of Custody Documentation - Paper</b>	No: EID-FS-003 Revision No.: 2 Page 2 of 4
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For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

## **6. PROCEDURE**

### **6.1 Documentation**

All Chain of Custody documentation must be completed in indelible ink. All corrections must be performed using standard single-line cross-out methods, and the initials of the individual making the change must be included beside the corrected entry.

### **6.2 Continuation Pages**

Continuation pages may be utilized for shipping containers/coolers with sufficient samples/sample containers that all of the lines of the Chain of Custody form are used before the documentation of the cooler/shipping container is complete. The number of pages in total must be filled out. *All samples entered onto a Continuation Page must be included in the same cooler/shipping container as those on the Chain of Custody form itself.*

### **6.3 Header Information**

- Each Chain of Custody form must be assigned a unique Reference Document Number—use the Project/proposal number followed by a unique numeric sequence or current date (if only one cooler sent per day). Continuation Pages should contain the same Document Reference Number as the Chain of Custody form that they are associated with. The project team should maintain a log of Chain of Custody Reference Document Numbers.
- The page identifier and total page count section must be completed. Total pages include the Chain of Custody form and any attached Continuation Pages.
- Project number, name, and location information must be completed for all forms.
- If available, the laboratory Purchase Order Number should be included on the appropriate line.
- The name and phone number of the *Project Contact* should be included; the Project Contact should be a responsible individual that the laboratory may access to address analytical issues. This person is usually the analytical lead for the project.
- The *Shipment Date* should be provided on the applicable lines.
- If shipping by carrier, the *Waybill/Airbill Number* must be included. Note: couriers will not sign custody documents. Therefore, inclusion of the waybill/airbill number on the Chain of Custody is the *only* means of documenting the transfer to the carrier.
- Laboratory Destination and Contact information should be provided.
- The Sampler(s) names should be provided on the appropriate line. This line should include all persons whose initials appear on any of the sample containers, to provide the laboratory a means of cross-referencing containers.
- The “Send Report To” information should be completed. If multiple reports/locations are needed, the information should be provided on a separate page included with the Chain of Custody documents.

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#### **6.4 Sample Information Section—Including on Continuation Page(s)**

During actual sampling, each sample must be entered on the COC form at the time of collection in order to document possession. The sampler must not wait until sampling is completed before entering samples on the COC.

- Complete the *Sample ID Number* for each line. If there are multiple container types for a sample, use additional lines to indicate the needed information.
- Ensure that the *Sample Description* matches the description on the sample label—the laboratory will use this information for cross-referencing.
- Provide the *Collection Date* and *Time*. These must match those on the sample label and Field Logbook/Logsheets.
- Indicate whether the sample is a Grab or Composite sample.
- Indicate the *Matrix* of the sample. Use the Matrix Codes listed on the Chain of Custody form.
- Indicate the *Number of Containers* and the *Container Type*. If a sample has multiple container types, use multiple lines and cross-out the information spaces to the left of the container blocks. *Failure to do this may cause the laboratory to log-in each container type as a separate sample/lab-ID, resulting in a confused report and invoice.*
  - Alternatively, if each sample has the same number/type container types, use “various” in the *Container Type* block and provide detail in the *Special Instructions* section, e.g., “Each sample consists of one 16-oz jar, two pre-weighed VOC w/DI water, and one pre-weighed VOC w/Methanol.”
- Check the appropriate *Preservative* box for each line/container type.
- Write in and check the *Analyses Requested* boxes for each line/container type. The appropriate method number (e.g., EPA Method 8260C) must be written as well as the method name.
- Indicate the *Turn-around Time Requested* for each sample.
- Use the *Special Instructions* section to provide important information to the laboratory, e.g., samples that may require dilution or samples that will need to be composited by the laboratory. This section may also be used to inform the laboratory of additional information contained in attachments to the Chain of Custody package.
- Circle the appropriate *QC/Data Package Level* requested.

#### **6.5 Custody Transfer Section**

- The first *Relinquished By* space must be completed by the individual who will either transfer the samples or seal the shipping container.
- If the samples will be transferred to a courier, write the courier/carrier company in the *Received By* box and enter the Date and Time that the shipping container was closed.
- All other transfers must be performed in person, and the Relinquisher must witness the signing by the Receiver.
- A copy of the Chain of Custody form and all associated Continuation Pages should be maintained in the project files.

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**7. ATTACHMENTS**

None

**8. FORMS**

- EID-FS-003.01, Shaw E & I Chain of Custody Form
- EID-FS-003.02, Shaw E & I COC Continuation Page


**9. RECORDS**

- EID-FS-003.01, Chain of Custody Form
- EID-FS-003.02, Chain of Custody Continuation Page(s)

**10. REVISION HISTORY AND APPROVAL**

Revision Level	Revision Description	Responsible Manager
Revision Date		
00	Initial Issue	N/A
07/22/2003		
01	New template, new numbering of procedure, Section 6.3 was edited, content was added in Section 6.4	Guy Gallelo
09/08/2006		
02	Modified format only to align with Governance Management framework	Scott Logan
08/25/2011		



	Document Type: <h1>Discipline-Specific Procedure</h1>	Level: 3 Owner: Applied Science & Engineering Origination Date: 8/14/2003 Revision Date: 8/25/2011
Group: <b>E&amp;I</b>	Title: <b>Custody Seals</b>	No: EID-FS-005 Revision No.: 2 Page 1 of 3

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## 1. PURPOSE

The purpose of this procedure is to provide the requirements for completion and attachment of Custody Seals on environmental samples and shipping containers.

## 2. SCOPE

This procedure is applicable to all Shaw E & I efforts where sample legal defensibility and custody integrity is desired. Adherence to this procedure is not required whenever the same individual/team is performing the sampling and testing within the same workday, and transfer to the testing process is being documented by other means, i.e. sampling and then field-screening in a mobile laboratory.

## 3. REFERENCES

- U.S. Environmental Protection Agency, 1986, *Test Methods for Evaluating Solid Waste; Physical/Chemical Methods, SW-846*, Third Edition.
- U.S. Army Corps of Engineers, *Requirements for the Preparation of Sampling and Analysis Plans, EM200-1-3*
- Shaw E & I, 2002, Sampler's Training Course Handout.

## 4. DEFINITIONS

- **Custody**—The legal term used to define the control and evidence traceability of an environmental sample. A sample is considered to be in one's custody if it is in actual physical possession of the person, is in view of the person, has been locked in a container controlled by the person, or has been placed into a designated secure area by the person.
- **Custody Seal**—Commercially available thin strips of adhesive paper with write-in lines for the date/time and identification of the using party. Custody seals are placed over the caps of sample containers and along the cover seals of shipping containers as a means to detect tampering before arrival at the testing facility. All Shaw E & I strategic alliance laboratories provide Custody Seals in their sample container supply kits.

## 5. RESPONSIBILITIES

### 5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

### 5.2 Project Responsibility

Shaw E & I employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw E & I employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting

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information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

## 6. PROCEDURE

### 6.1 Completing the Custody Seal Information

- All Custody Seals must be completed in indelible ink. All corrections must be made using standard single-line cross-out methods, and the initials of the individual making the change must be included beside the corrected entry.
- Each Custody Seal attached must be completed by writing the *Date*, at a minimum, and signing with *full signature* by the person responsible for the sealing of the sample.
- If a space is provided, the *Time* should also be added.

### 6.2 Attaching the Custody Seals

Whenever possible, custody seals should be attached over the sample container lids during actual sampling and not when the samples are packaged for shipment. This will provide confidence in legal custody and will demonstrate non-tampering during the sample collection process.

Do not attach custody seals to VOC sample containers, as contamination may occur. For these samples, the custody seal should be used to seal the folded plastic zip bag that holds the sample containers.

- For sample jars, the completed Custody Seal should be placed across the top of the lid with the edges below the lid/jar interface and attached to the jar material. This will require the visible breaking of the seal in order to open the container.
- Sample coolers and shipping containers should have Custody Seals attached in such a manner that the seal extends lengthwise from the top edge of the lid to the side of the cooler/container.

## 7. ATTACHMENTS

None

## 8. FORMS

None

## 9. RECORDS

None


## 10. REVISION HISTORY AND APPROVAL

Revision Level	Revision Description	Responsible Manager
Revision Date		
00	Initial Issue	N/A
08/14/2003		
01	New template, new numbering of procedure, no content changes	Guy Gallelo
09/08/2006		

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Revision Level	Revision Description	Responsible Manager
Revision Date		
02	Modified format only to align with Governance Management framework	Scott Logan
08/25/2011		

	Document Type: <h1>Discipline-Specific Procedure</h1>	Level: 3 Owner: Applied Science & Engineering Origination Date: 8/17/2003 Revision Date: 8/25/2011
Group: <b>E&amp;I</b>	Title: <b>Sample Labeling</b>	No: EID-FS-006 Revision No.: 2 Page 1 of 2

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## 1. PURPOSE

The purpose of this procedure is to provide the requirements for completion and attachment of sample labels on environmental sample containers.

## 2. SCOPE

This procedure is applicable to all Shaw E & I projects/proposals where samples will be collected.

## 3. REFERENCES

- U.S. Environmental Protection Agency, 1986, *Test Methods for Evaluating Solid Waste; Physical/Chemical Methods*, SW-846, Third Edition.
- U.S. Army Corps of Engineers, *Requirements for the Preparation of Sampling and Analysis Plans*, EM200-1-3
- Shaw E & I, 2002, Sampler's Training Course Handout.

## 4. DEFINITIONS

- **Sample Label**—Any writing surface with an adhesive backing that can be used to document sample identification information. The sample label is attached to the sample container as a means of identification and, in some commercially available or laboratory-supplied containers, may be pre-attached. All Shaw E & I strategic alliance laboratories provide sample labels or pre-labeled containers in their sample container supply kits.

## 5. RESPONSIBILITIES

### 5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

### 5.2 Project Responsibility

Shaw E & I employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw E & I employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager, or designee, is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (i.e. checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

## 6. PROCEDURE

- All sample labels must be completed in indelible ink. All corrections must be performed using standard single-line cross-out methods, and the initials of the individual making the change must be included beside the corrected entry.

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- Sample labels should be completed and attached as samples are collected. Do not wait until final packaging to attach and/or complete the sample labels.
- Sample labels must be attached to the non-sealing portion of the container. Do not place labels on or across sample container caps.
- If the laboratory has provided pre-labeled containers, make sure to fill one for each parameter set needed. Laboratory pre-labeled containers are often bar-coded and it is important to provide a complete container set for each sample.
- The following information must be recorded on the Sample Label:
  - Sample Identification Number
  - Date and Time collected
  - Initials of person(s) responsible for collection
- If a space is provided, the *Analysis Requested* should also be added.
- If a *Description* is provided, remember it must match that on the Chain of Custody form for cross-referencing purposes.
- Cover the completed and attached label with clear plastic tape to prevent bleeding of the ink if it becomes wetted. *Do not perform this step for pre-weighed VOC vials, as the final weight values will be influenced by the mass of the tape. Protect these containers by enclosing the rack/holder in a plastic bag within the cooler.*

**7. ATTACHMENTS**

None

**8. FORMS**


None

**9. RECORDS**

None

**10. REVISION HISTORY AND APPROVAL**

Revision Level	Revision Description	Responsible Manager
Revision Date		
00	Initial issue	N/A
09/08/2006		
01	Updated template, procedure numbering change, updated Section 2- Scope, Edited content in section 6.	Guy Gallelo
09/08/2006		
02	Modified format only to align with Governance Management framework	Scott Logan
08/28/2011		

	Document Type:	Level: 3
	<h1>Discipline-Specific Procedure</h1>	Owner: Applied Science & Engineering Origination Date: 1/19/2004 Revision Date: 8/25/2011
Group: <b>E&amp;I</b>	Title: <b>Water Quality Meter Use</b>	No: EID-FS-204 Revision No.: 2 Page 1 of 4

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## 1. PURPOSE

This procedure is intended to provide general guidance and methods for using a field meter to measure water quality parameters from groundwater or surface water that is being purged, sampled, or monitored.

## 2. SCOPE

This procedure is applicable to all Shaw E & I projects where water quality monitoring is required using a water quality meter. The water quality meter may be a stand-alone meter or it may be a combined multi-probe unit used to measure temperature, pH, specific conductance, and/or other water quality parameters. The most common methods used for measuring water quality are instruments that measure in-situ parameters in one of the following two ways:

- Water is extracted from its source using a pump and measured in a flow-through cell or in some instances captured and then measured in individual aliquots. This method is preferred when monitoring wells are sampled for laboratory analysis of chemical parameters, and groundwater purging is required.
- The meter is submerged directly into the sample source, such as a monitoring well or surface water body, to collect in-situ monitoring parameters.

## 3. REFERENCES

- U.S. Army Corps of Engineers, 2001, *Requirements for the Preparation of Sampling and Analysis Plans, Appendix C*, EM-200-1-3, Washington, D.C.
- American Society of Testing and Materials, *Standard Guide for Selection of Purging and Sampling Devices for Ground-Water Monitoring Wells*, D6634-01, West Conshohocken, PA.
- American Society of Testing and Materials, *Standard Guide for Sampling Ground-Water Monitoring Wells*, D4448-01, West Conshohocken, PA.

## 4. DEFINITIONS

- **Water Quality Meter**—A device used to measure specific field parameters indicative of water quality, such as temperature, pH, specific conductance, and/or other parameters. The meter may be stand-alone or it may be a combined multi-probe unit.
- **Pump**—An electric, compressed air, or inert gas-driven device that raises liquids by means of pressure or suction. The types of pumps that should be used for water quality monitoring should be chosen based on the well size and depth, the type of contaminants, and the specific factors affecting the overall performance of the sampling or monitoring effort. The types of pumps that may be used include centrifugal, peristaltic, centrifugal submersible, gas displacement, and bladder pumps.
- **pH**—The negative log of the hydrogen ion concentration ( $-\log_{10} [H^+]$ ); a measure of the acidity or alkalinity of a solution, numerically equal to 7 for neutral solutions, increasing with increasing alkalinity and decreasing with increasing acidity. The scale is 0 to 14.
- **Turbidity**—A measure of overall water clarity determined by measuring the degree to which light traveling through a water column is scattered by the suspended organic (including algae)

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and inorganic particles. Turbidity is commonly measured in Nephelometric Turbidity Units (NTU), but may also be measured in Jackson Turbidity Units (JTU).

- **Specific Conductance (SC)**—A measure of how well water can conduct an electrical current. Conductivity increases with increasing amount and mobility of ions such as chloride, nitrate, sulfate, phosphate, sodium, magnesium, calcium, and iron, and can be used as an indicator of water pollution. The unit of conductance is expressed as microsiemens (1/1,000,000 siemen) per centimeter, or  $\mu\text{S}/\text{cm}$ .
- **Oxidation-Reduction (Redox) Potential**—A measure in volts of the affinity of a substance for electrons compared with hydrogen. Liquids that are more strongly electronegative than hydrogen (i.e. capable of oxidizing) have positive redox potentials. Liquids less electronegative than hydrogen (i.e. capable of reducing) have negative redox potentials.
- **Dissolved Oxygen (DO)**—Refers to the amount of oxygen expressed as mg/L that is contained in particular water. The amount of oxygen that can be held by the water depends on the water temperature, salinity, purity, and pressure.
- **Salinity**—The amount of dissolved salts in water, generally expressed in parts per thousand (ppt).

## 5. RESPONSIBILITIES

### 5.1 Procedure Responsibility

The Field Sampling Discipline Lead is responsible for maintenance, management, and revision of this procedure. Questions, comments, or suggestions regarding this technical SOP should be directed to the Field Sampling Discipline Lead.

### 5.2 Project Responsibility

Shaw employees performing this task, or any portion thereof, are responsible for meeting the requirements of this procedure. Shaw employees conducting technical review of task performance are also responsible for following appropriate portions of this SOP.

For those projects where the activities of this SOP are conducted, the Project Manager or designee is responsible for ensuring that those activities are conducted in accordance with this and other appropriate procedures. Project participants are responsible for documenting information in sufficient detail to provide objective documentation (checkprints, calculations, reports, etc.) that the requirements of this SOP have been met. Such documentation shall be retained as project records.

## 6. PROCEDURE

### 6.1 Equipment

The following equipment is recommended for use in performing water quality measurements:

- Water Quality Meter(s)
- Spare parts such as alkaline batteries (if used) and sensor probes
- Pump and discharge hose/line for use with a flow-through cell
- Paper towels or lint-free wipes
- De-ionized water
- Sample gloves

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- Calibration solutions for all parameters being measured; within expiration dates
- Plastic sheeting
- Logbook or log sheets

## 6.2 General Instructions

- Ensure that the measuring range of the instrument encompasses the expected sample concentration or units.
- Before going to the field, locate all necessary field supplies such as deionized water, calibration solutions, decontamination supplies, and spare parts.
- Consult the instrument's operation manual as well as the project-specific sampling plan to verify that you have prepared the proper equipment and supplies to successfully complete the work.

## 6.3 Calibration

*Calibration **must** be performed **at least once per day** during operation. Calibrate the meter according to the instrument's operating manual. If sampling and monitoring is being performed for long periods of time, periodically check the instrument calibration using the operating manual's recommended frequency.*

In order to avoid limiting the field personnel to one particular model, only general calibration instructions are presented in this procedure.

- Locate a clean, protected area in which to set up and calibrate the instrument. Ensure that sufficient supplies of de-ionized water, clean paper towels, buffer solutions, and standard solutions are available.
- Inspect the meter and probes for damage. Some of the probes are very delicate or have a thin membrane installed over the probe. Be careful when handling the meter/probes so as not to damage them. If damaged, replace probes in accordance with the instrument's operating manual or obtain a different meter.
- Turn on the meter and allow it to "warm-up" for the manufacturer-specified time (usually 15 to 30 minutes). Check the battery power to determine if the meter has sufficient power to operate for the monitoring period. Replace the batteries, if necessary.
- Calibrate the meter according to the instrument's operating manual. In general, calibration is performed by immersing the probe(s) in aliquots of calibration standard solution(s) and following certain meter keystrokes to set the calibration for each parameter. *Do not immerse the probe into the stock container of the solution. Always transfer a small amount of the solution into a separate container to calibrate the probe(s).* If calibrating for multiple parameters using more than one solution, be sure to wipe off and rinse the probe with de-ionized water between solutions.
- Recheck each parameter after calibration by immersing the probe into the calibration solution and reading it like a sample reading. If the agreement is not within 25% of the solution's known concentration, repeat the calibration process with a new solution aliquot.
- Discard the used calibration solution aliquots when finished into an appropriate waste container.
- Record the calibration data in the field logbook or log sheet.



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#### 6.4 Operation of the Instrument

- If using a flow-through cell system, attach the extraction pump and lines in accordance with the pump and meter manufacturer's instructions. Allow the lines to fill and the probes to become immersed before switching the instrument to its measurement mode.
- If using a down-hole system, allow a few minutes for the probe to stabilize before taking a reading.
- Operate the meter in accordance with the instrument's operating manual.
- Collect the field parameter reading(s) per the project requirements, and record them in a field logbook or on log sheets.
- Decontaminate the meter before collecting data from the next sample source. For a flow-through system, flush the lines with three line volumes of de-ionized water or replace with new ones between samples.

#### 7. ATTACHMENTS

None

#### 8. FORMS

None

#### 9. RECORDS

- Logbook or Logsheet

#### 10. REVISION HISTORY AND APPROVAL

Revision Level	Revision Description	Responsible Manager
Revision Date		
00	Initial issue.	N/A
01/19/2004		
01	Updated template and numbering of procedure.	Guy Gallelo
09/22/2006		
02	Modified format only to align with Governance Management framework.	Scott Logan
08/25/2011		

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Appendix B  
Department of Defense Environmental  
Laboratory Accreditation Program  
Accreditation Letters

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# CERTIFICATE OF ACCREDITATION

**The ANSI National Accreditation Board**

Hereby attests that

**SGS North America Inc. - Orlando**  
**4405 Vineland Road, Suite C-15**  
**Orlando, FL 32811**

Fulfills the requirements of

**ISO/IEC 17025:2017**

and

**U.S. Department of Defense (DoD) Quality Systems Manual**  
**for Environmental Laboratories (DoD QSM V 5.4)**

In the field of

**TESTING**

This certificate is valid only when accompanied by a current scope of accreditation document.  
The current scope of accreditation can be verified at [www.anab.org](http://www.anab.org).

Jason Stine, Vice President

Expiry Date: 15 December 2024

Certificate Number: L2229



This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2017.  
This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory  
quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).



**SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017**

**AND**

**U.S. Department of Defense (DoD) Quality Systems Manual for  
Environmental Laboratories (DoD QSM V 5.4)**

**SGS North America Inc. - Orlando**

4405 Vineland Road, Suite C-15  
Orlando, FL 32811  
Svetlana Izosimova, Ph. D., QA Officer  
407-425-6700

**TESTING**

Valid to: **December 15, 2024**

Certificate Number: **L2229**

**Environmental**

<b>Drinking Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
LC/MS/MS	EPA 537 rev. 1.1	Perfluorohexanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluoroheptanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorooctanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorononanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorodecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluoroundecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorododecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorotridecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorotetradecanoic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorobutanesulfonic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorohexanesulfonic Acid
LC/MS/MS	EPA 537 rev. 1.1	Perfluorooctanesulfonic Acid
LC/MS/MS	EPA 537 rev. 1.1	N-Methyl perfluorooctanesulfonamidoacetic acid
LC/MS/MS	EPA 537 rev. 1.1	N-Ethyl perfluorooctanesulfonamidoacetic acid

<b>Drinking Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
LC/MS/MS	EPA 537.1	Perfluorohexanoic Acid
LC/MS/MS	EPA 537.1	Perfluoroheptanoic Acid
LC/MS/MS	EPA 537.1	Perfluorooctanoic Acid
LC/MS/MS	EPA 537.1	Perfluorononanoic Acid
LC/MS/MS	EPA 537.1	Perfluorodecanoic Acid
LC/MS/MS	EPA 537.1	Perfluoroundecanoic Acid
LC/MS/MS	EPA 537.1	Perfluorododecanoic Acid
LC/MS/MS	EPA 537.1	Perfluorotridecanoic Acid
LC/MS/MS	EPA 537.1	Perfluorotetradecanoic Acid
LC/MS/MS	EPA 537.1	Perfluorobutanesulfonic Acid
LC/MS/MS	EPA 537.1	Perfluorohexanesulfonic Acid
LC/MS/MS	EPA 537.1	Perfluorooctanesulfonic Acid
LC/MS/MS	EPA 537.1	N-Methyl perfluorooctanesulfonamidoacetic acid
LC/MS/MS	EPA 537.1	N-Ethyl perfluorooctanesulfonamidoacetic acid
LC/MS/MS	EPA 537.1	ADONA
LC/MS/MS	EPA 537.1	2,3,3,3-Tetrafluoro-2-(heptafluoropropoxy)propanoic acid (HFPO-DA; GenX)
LC/MS/MS	EPA 537.1	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS; F53B minor)
LC/MS/MS	EPA 537.1	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS; F53B major)
LC/MS/MS	EPA 533	Perfluorobutanoic acid
LC/MS/MS	EPA 533	Perfluoropentanoic acid
LC/MS/MS	EPA 533	Perfluorohexanoic acid
LC/MS/MS	EPA 533	Perfluoroheptanoic acid
LC/MS/MS	EPA 533	Perfluorooctanoic acid
LC/MS/MS	EPA 533	Perfluorononanoic acid

<b>Drinking Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
LC/MS/MS	EPA 533	Perfluorodecanoic acid
LC/MS/MS	EPA 533	Perfluoroundecanoic acid
LC/MS/MS	EPA 533	Perfluorododecanoic acid
LC/MS/MS	EPA 533	Perfluorobutanesulfonic acid
LC/MS/MS	EPA 533	Perfluoropentanesulfonic acid
LC/MS/MS	EPA 533	Perfluorohexanesulfonic acid
LC/MS/MS	EPA 533	Perfluoroheptanesulfonic acid
LC/MS/MS	EPA 533	Perfluorooctanesulfonic acid
LC/MS/MS	EPA 533	4:2 Fluorotelomer sulfonate
LC/MS/MS	EPA 533	6:2 Fluorotelomer sulfonate
LC/MS/MS	EPA 533	8:2 Fluorotelomer sulfonate
LC/MS/MS	EPA 533	Perfluoro-3-methoxypropanoic acid
LC/MS/MS	EPA 533	Perfluoro-4-methoxybutanoic acid
LC/MS/MS	EPA 533	Nonafluoro-3,6-dioxahexanoic acid
LC/MS/MS	EPA 533	Perfluoro(2-ethoxyethane)sulfonic acid
LC/MS/MS	EPA 533	Hexafluoropropylene oxide dimer acid
LC/MS/MS	EPA 533	4,8-Dioxa-3H-perfluorononanoic acid
LC/MS/MS	EPA 533	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid
LC/MS/MS	EPA 533	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
GC/ECD	EPA 8011	1,2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1,2-Dibromo-3-Chloropropane (DBCP)
GC/ECD	EPA 504.1	1,2-Dibromoethane (EDB)
GC/ECD	EPA 504.1	1,2-Dibromo-3-Chloropropane (DBCP)
GC/ECD	EPA 504.1	1,2,3-Trichloropropane (1,2,3-TCP)
GC/FID	EPA 8015C/D	Diesel range organics (DRO)



Non-Potable Water		
Technology	Method	Analyte
GC/FID	EPA 8015C/D	Oil Range Organics (ORO)
GC/FID	EPA 8015C/D	Gasoline range organics (GRO)
GC/ECD	EPA 608.3; EPA 8081B	4,4`-DDD
GC/ECD	EPA 608.3; EPA 8081B	4,4`-DDE
GC/ECD	EPA 608.3; EPA 8081B	4,4`-DDT
GC/ECD	EPA 608.3; EPA 8081B	Aldrin
GC/ECD	EPA 608.3; EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 608.3; EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 608.3; EPA 8081B	delta-BHC
GC/ECD	EPA 608.3; EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 608.3; EPA 8081B	Chlordane (tech.)
GC/ECD	EPA 608.3; EPA 8081B	alpha-Chlordane
GC/ECD	EPA 608.3; EPA 8081B	gamma-Chlordane
GC/ECD	EPA 608.3; EPA 8081B	Dieldrin
GC/ECD	EPA 608.3; EPA 8081B	Endosulfan I
GC/ECD	EPA 608.3; EPA 8081B	Endosulfan II
GC/ECD	EPA 608.3; EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 608.3; EPA 8081B	Endrin
GC/ECD	EPA 608.3; EPA 8081B	Endrin aldehyde
GC/ECD	EPA 608.3; EPA 8081B	Endrin ketone
GC/ECD	EPA 608.3; EPA 8081B	Heptachlor
GC/ECD	EPA 608.3; EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 608.3; EPA 8081B	Methoxychlor
GC/ECD	EPA 608.3; EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 608.3; EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 608.3; EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 608.3; EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 608.3; EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 608.3; EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 608.3; EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 608.3; EPA 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	Total PCB



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Non-Potable Water		
Technology	Method	Analyte
GC/FPD	EPA 8141B	Azinphos-methyl (Guthion)
GC/FPD	EPA 8141B	Bolstar (Sulprofos)
GC/FPD	EPA 8141B	Carbophenothion
GC/FPD	EPA 8141B	Chlorpyrifos
GC/FPD	EPA 8141B	Coumaphos
GC/FPD	EPA 8141B	Demeton-o
GC/FPD	EPA 8141B	Demeton-s
GC/FPD	EPA 8141B	Demeton
GC/FPD	EPA 8141B	Diazinon
GC/FPD	EPA 8141B	Dichlorovos (DDVP Dichlorvos)
GC/FPD	EPA 8141B	Dimethoate
GC/FPD	EPA 8141B	Disulfoton
GC/FPD	EPA 8141B	EPN
GC/FPD	EPA 8141B	Ethion
GC/FPD	EPA 8141B	Ethoprop
GC/FPD	EPA 8141B	Famphur
GC/FPD	EPA 8141B	Fensulfothion
GC/FPD	EPA 8141B	Fenthion
GC/FPD	EPA 8141B	Malathion
GC/FPD	EPA 8141B	Merphos
GC/FPD	EPA 8141B	Methyl parathion (Parathion methyl)
GC/FPD	EPA 8141B	Mevinphos
GC/FPD	EPA 8141B	Monocrotophos
GC/FPD	EPA 8141B	Naled
GC/FPD	EPA 8141B	Parathion ethyl
GC/FPD	EPA 8141B	Phorate
GC/FPD	EPA 8141B	Ronnel
GC/FPD	EPA 8141B	Stirofos
GC/FPD	EPA 8141B	Sulfotepp
GC/FPD	EPA 8141B	Tetraethyl pyrophosphate (TEPP)
GC/FPD	EPA 8141B	Thionazin (Zinophos)
GC/FPD	EPA 8141B	Tokuthion (Prothiophos)
GC/FPD	EPA 8141B	Trichloronate
GC/FPD	EPA 8141B	O,O,O-Triethyl phosphorothioate
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4-D



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Non-Potable Water		
Technology	Method	Analyte
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop (Dichloroprop)
GC/ECD	EPA 8151A	Dinoseb (2-sec-butyl-4,6-dinitrophenol DNBP)
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2,4,5-TP)
GC/FID	RSK-175	Acetylene
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/FID	RSK-175	Propane
GC/FID	FL-PRO	Total Petroleum Hydrocarbons (TPH)
GC/FID	MA-VPH	Volatile petroleum range organics (VPH)
GC/FID	MA-EPH	Extractable petroleum range organics (EPH)
GC/FID	IA-OA1	Gasoline range organics (GRO)
GC/FID	IA-OA2	Diesel range organics (DRO)
GC/FID	TN-GRO	Gasoline range organics (GRO)
GC/FID	TN-EPH	Extractable petroleum range organics (EPH)
GC/FID	WI-DRO	Diesel range organics (DRO)
GC/FID	KS LRH	Low-Range Hydrocarbons (LRH)
GC/FID	KS MRH	Mid-Range Hydrocarbons (MRH)
GC/FID	KS HRH	High-Range Hydrocarbons (HRH)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,1,1,2-Tetrachloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,1,1-Trichloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,1,2,2-Tetrachloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,1,2-Trichloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,1-Dichloroethane

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,1-Dichloroethylene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,1-Dichloropropene
GC/MS	EPA 624.1; EPA 8260C/D	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,2,3-Trichlorobenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,2,3-Trichloropropane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,2,4-Trichlorobenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,2,4-Trimethylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,2-Dibromoethane (EDB Ethylene dibromide)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,2-Dichlorobenzene (o-Dichlorobenzene)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,2-Dichloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,2-Dichloroethene (total)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,2-Dichloropropane
GC/MS	EPA 8260C/D	1,2-Dichlorotrifluoroethane (Freon 123)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,3,5-Trimethylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,3-Dichlorobenzene (m-Dichlorobenzene)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,3-Dichloropropane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	1,4-Dichlorobenzene (p-Dichlorobenzene)
GC/MS	EPA 8260C	1-Chlorohexane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	2,2-Dichloropropane



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Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	2-Butanone (Methyl ethyl ketone MEK)
GC/MS	EPA 624.1; EPA 8260C/D	2-Chloroethyl vinyl ether
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	2-Chlorotoluene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	2-Hexanone
GC/MS	EPA 8260C	2-Nitropropane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	4-Chlorotoluene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Acetone
GC/MS	EPA 8260C/D	Acetonitrile
GC/MS	EPA 624.1; EPA 8260C/D	Acrolein (Propenal)
GC/MS	EPA 624.1; EPA 8260C/D	Acrylonitrile
GC/MS	EPA 8260C/D	Allyl chloride (3-Chloropropene)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Benzene
GC/MS	EPA 8260C/D	Benzyl Chloride
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Bromobenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Bromochloromethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Bromodichloromethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Bromoform
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	n-Butylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	sec-Butylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	tert-Butylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Carbon disulfide
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Carbon tetrachloride

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Chlorobenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Chloroethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Chloroform
GC/MS	EPA 8260C/D	Chloroprene
GC/MS	EPA 624.1; EPA 8260C/D	Cyclohexane
GC/MS	EPA 8260C/D	Cyclohexanone
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	cis-1,2-Dichloroethylene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	trans-1,2-Dichloroethylene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	cis-1,3-Dichloropropene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	trans-1,3-Dichloropropylene
GC/MS	EPA 8260C/D	cis-1,4-Dichloro-2-butene
GC/MS	EPA 8260C/D	trans-1,4-Dichloro-2-butene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Di-isopropylether (DIPE)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Dibromochloromethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Dibromomethane (Methylene Bromide)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Dichlorodifluoromethane
GC/MS	EPA 8260C/D	Diethyl ether
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D; EPA 8260C/D SIM	p-Dioxane (1,4-Dioxane)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Ethanol (Ethyl Alcohol)
GC/MS	EPA 8260C/D	Ethyl acetate
GC/MS	EPA 8260C/D	Ethyl methacrylate
GC/MS	EPA 8260C	Ethyl tert-butyl alcohol (ETBA)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Ethyl tert-butyl ether (ETBE)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Ethylbenzene

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Hexachlorobutadiene
GC/MS	EPA 8260C/D	Hexane
GC/MS	EPA 8260C/D	Iodomethane (Methyl iodide)
GC/MS	EPA 8260C/D	Isobutyl alcohol (2-Methyl-1-propanol)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	p-Isopropyltoluene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Isopropylbenzene
GC/MS	EPA 8260C/D	Methacrylonitrile
GC/MS	EPA 624.1; EPA 8260C/D	Methyl Acetate
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Methyl bromide (Bromomethane)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Methyl chloride (Chloromethane)
GC/MS	EPA 624.1; EPA 8260C/D	Methylcyclohexane
GC/MS	EPA 8260C/D	Methyl methacrylate
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Methylene chloride
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Naphthalene
GC/MS	EPA 8260C/D	Pentachloroethane
GC/MS	EPA 8260C/D	Propionitrile (Ethyl cyanide)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	n-Propylbenzene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Styrene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	tert-Amyl alcohol (TAA)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	tert-Amyl methyl ether (TAME)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	tert-Butyl alcohol (TBA)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	tert-Butyl formate (TBF)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Tetrachloroethylene (Perchloroethylene)

Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8260C/D	Tetrahydrofuran
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Toluene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Trichloroethene (Trichloroethylene)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Trichlorofluoromethane
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Vinyl acetate
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Vinyl chloride
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	Xylene (total)
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	m,p-Xylene
GC/MS	EPA 624.1; SM 6200B-11; EPA 8260C/D	o-Xylene
GC/MS	EPA 625.1; EPA 8270D/E	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 625.1; EPA 8270D/E	1,2,4-Trichlorobenzene
GC/MS	EPA 625.1; EPA 8270D/E	1,2-Dichlorobenzene (o-Dichlorobenzene)
GC/MS	EPA 625.1; EPA 8270D/E	1,2-Diphenylhydrazine
GC/MS	EPA 8270D/E	1,3,5-Trinitrobenzene (1,3,5-TNB)
GC/MS	EPA 625.1; EPA 8270D/E	1,3-Dichlorobenzene (m-Dichlorobenzene)
GC/MS	EPA 8270D/E	1,3-Dinitrobenzene (1,3-DNB)
GC/MS	EPA 625.1; EPA 8270D/E	1,4-Dichlorobenzene (p-Dichlorobenzene)
GC/MS	EPA 8270D/E	1,4-Naphthoquinone
GC/MS	EPA 8270D/E	1,4-Phenylenediamine
GC/MS	EPA 8270D/E	1-Chloronaphthalene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	1-Methylnaphthalene
GC/MS	EPA 8270D/E	1-Naphthylamine
GC/MS	EPA 625.1; EPA 8270D/E	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 625.1; EPA 8270D/E	2,4,5-Trichlorophenol
GC/MS	EPA 625.1; EPA 8270D/E	2,4,6-Trichlorophenol
GC/MS	EPA 625.1; EPA 8270D/E	2,4-Dichlorophenol
GC/MS	EPA 625.1; EPA 8270D/E	2,4-Dimethylphenol
GC/MS	EPA 625.1; EPA 8270D/E	2,4-Dinitrophenol
GC/MS	EPA 625.1; EPA 8270D/E	2,4-Dinitrotoluene (2,4-DNT)



Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270D/E	2,6-Dichlorophenol
GC/MS	EPA 625.1; EPA 8270D/E	2,6-Dinitrotoluene (2,6-DNT)
GC/MS	EPA 8270D/E	2-Acetylaminofluorene
GC/MS	EPA 625.1; EPA 8270D/E	2-Chloronaphthalene
GC/MS	EPA 625.1; EPA 8270D/E	2-Chlorophenol
GC/MS	EPA 625.1; EPA 8270D/E	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-o-cresol)
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	2-Methylnaphthalene
GC/MS	EPA 625.1; EPA 8270D/E	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270D/E	2-Naphthylamine
GC/MS	EPA 625.1; EPA 8270D/E	2-Nitroaniline
GC/MS	EPA 625.1; EPA 8270D/E	2-Nitrophenol
GC/MS	EPA 8270D/E	2-Picoline (2-Methylpyridine)
GC/MS	EPA 625.1; EPA 8270D/E	3,3`-Dichlorobenzidine
GC/MS	EPA 8270D/E	3,3`-Dimethylbenzidine
GC/MS	EPA 8270D/E	3-Methylcholanthrene
GC/MS	EPA 625.1; EPA 8270D/E	3&4-Methylphenol (m,p-Cresol)
GC/MS	EPA 625.1; EPA 8270D/E	3-Nitroaniline
GC/MS	EPA 8270D/E	4-Aminobiphenyl
GC/MS	EPA 625.1; EPA 8270D/E	4-Bromophenyl phenyl ether
GC/MS	EPA 625.1; EPA 8270D/E	4-Chloro-3-methylphenol
GC/MS	EPA 625.1; EPA 8270D/E	4-Chloroaniline
GC/MS	EPA 625.1; EPA 8270D/E	4-Chlorophenyl phenylether
GC/MS	EPA 8270D/E	4-Dimethyl aminoazobenzene
GC/MS	EPA 625.1; EPA 8270D/E	4-Nitroaniline
GC/MS	EPA 625.1; EPA 8270D/E	4-Nitrophenol
GC/MS	EPA 8270D/E	5-Nitro-o-toluidine
GC/MS	EPA 8270D/E	7,12-Dimethylbenz(a) anthracene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Acenaphthene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Acenaphthylene
GC/MS	EPA 625.1; EPA 8270D/E	Acetophenone
GC/MS	EPA 625.1; EPA 8270D/E	Aniline
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Anthracene



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Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270D/E	Aramite
GC/MS	EPA 625.1; EPA 8270D/E	Atrazine
GC/MS	EPA 625.1; EPA 8270D/E	Benzaldehyde
GC/MS	EPA 625.1; EPA 8270D/E	Benzydine
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Benzo(a)anthracene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Benzo(a)pyrene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Benzo(b)fluoranthene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Benzo(g,h,i)perylene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Benzo(k)fluoranthene
GC/MS	EPA 625.1; EPA 8270D/E	Benzoic acid
GC/MS	EPA 625.1; EPA 8270D/E	Benzyl alcohol
GC/MS	EPA 625.1; EPA 8270D/E	Biphenyl(1,1'-Biphenyl)
GC/MS	EPA 625.1; EPA 8270D/E	bis(2-Chloroethoxy)methane
GC/MS	EPA 625.1; EPA 8270D/E	bis(2-Chloroethyl) ether
GC/MS	EPA 625.1; EPA 8270D/E	bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))
GC/MS	EPA 625.1; EPA 8270D/E	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 625.1; EPA 8270D/E	Butyl benzyl phthalate
GC/MS	EPA 625.1; EPA 8270D/E	Carbazole
GC/MS	EPA 625.1; EPA 8270D/E	Caprolactam
GC/MS	EPA 8270D/E	Chlorobenzilate
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Chrysene
GC/MS	EPA 8270D/E	Diallate
GC/MS	EPA 625.1; EPA 8270D/E	Di-n-butyl phthalate
GC/MS	EPA 625.1; EPA 8270D/E	Di-n-octyl phthalate
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Dibenz(a,h)anthracene
GC/MS	EPA 8270D/E	Dibenz(a,j)acridine
GC/MS	EPA 625.1; EPA 8270D/E	Dibenzofuran
GC/MS	EPA 625.1; EPA 8270D/E	Diethyl phthalate
GC/MS	EPA 625.1; EPA 8270D/E	Dimethyl phthalate



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Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270D/E	a,a-Dimethylphenethylamine
GC/MS	EPA 8270D/E	Diphenyl Ether
GC/MS	EPA 8270D/E EPA 8270D/E SIM	p-Dioxane (1,4-Dioxane)
GC/MS	EPA 8270D/E	Ethyl methanesulfonate
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Fluoranthene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Fluorene
GC/MS	EPA 625.1; EPA 8270D/E	Hexachlorobenzene
GC/MS	EPA 625.1; EPA 8270D/E	Hexachlorobutadiene
GC/MS	EPA 625.1; EPA 8270D/E	Hexachlorocyclopentadiene
GC/MS	EPA 625.1; EPA 8270D/E	Hexachloroethane
GC/MS	EPA 8270D/E	Hexachlorophene
GC/MS	EPA 8270D/E	Hexachloropropene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270D	Isodrin
GC/MS	EPA 625.1; EPA 8270D/E	Isophorone
GC/MS	EPA 8270D/E	Isosafrole
GC/MS	EPA 8270D/E	Kepone
GC/MS	EPA 8270D/E	Methapyrilene
GC/MS	EPA 8270D/E	Methyl methanesulfonate
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Naphthalene
GC/MS	EPA 625.1; EPA 8270D/E	Nitrobenzene
GC/MS	EPA 8270D/E	Nitroquinoline-1-oxide
GC/MS	EPA 8270D/E	n-Nitroso-di-n-butylamine
GC/MS	EPA 625.1; EPA 8270D/E	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270D/E	n-Nitrosodiethylamine
GC/MS	EPA 625.1; EPA 8270D/E	n-Nitrosodimethylamine
GC/MS	EPA 625.1; EPA 8270D/E	n-Nitrosodiphenylamine
GC/MS	EPA 8270D/E	n-Nitrosodiphenylamine/Diphenylamine (analyte pair)
GC/MS	EPA 8270D/E	n-Nitrosomethylethylamine
GC/MS	EPA 8270D/E	n-Nitrosomorpholine
GC/MS	EPA 8270D/E	n-Nitrosopiperidine



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Non-Potable Water		
Technology	Method	Analyte
GC/MS	EPA 8270D/E	n-Nitrosopyrrolidine
GC/MS	EPA 8270D/E	Pentachlorobenzene
GC/MS	EPA 8270D/E	Pentachloroethane
GC/MS	EPA 8270D/E	Pentachloronitrobenzene
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Pentachlorophenol
GC/MS	EPA 8270D/E	Phenacetin
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Phenanthrene
GC/MS	EPA 625.1; EPA 8270D/E	Phenol
GC/MS	EPA 8270D/E	Pronamide (Kerb)
GC/MS	EPA 625.1; EPA 8270D/E; EPA 8270D/E SIM	Pyrene
GC/MS	EPA 625.1; EPA 8270D/E	Pyridine
GC/MS	EPA 8270D/E	Safrole
GC/MS	EPA 8270D/E	Simazine
GC/MS	EPA 8270D/E	Thionazin (Zinophos)
GC/MS	EPA 8270D/E	o-Toluidine
GC/MS	EPA 8270D/E	Dimethoate
GC/MS	EPA 8270D/E	Disulfoton
GC/MS	EPA 8270D/E	Famphur
GC/MS	EPA 8270D/E	Methyl parathion (Parathion methyl)
GC/MS	EPA 8270D/E	Parathion ethyl
GC/MS	EPA 8270D/E	Phorate
GC/MS	EPA 8270D/E	O,O,O-Triethyl phosphorothioate
HPLC	EPA 8330A/B	1,3,5-Trinitrobenzene (1,3,5-TNB)
HPLC	EPA 8330A/B	1,3-Dinitrobenzene (1,3-DNB)
HPLC	EPA 8330A/B	2,4,6-Trinitrotoluene (2,4,6-TNT)
HPLC	EPA 8330A/B	2,4-Dinitrotoluene (2,4-DNT)
HPLC	EPA 8330A/B	2,6-Dinitrotoluene (2,6-DNT)
HPLC	EPA 8330A/B	2-Amino-4,6-dinitrotoluene (2-am-dnt)
HPLC	EPA 8330A/B	2-Nitrotoluene
HPLC	EPA 8330A/B	3,5-Dinitroaniline
HPLC	EPA 8330A/B	3-Nitrotoluene
HPLC	EPA 8330A/B	4-Amino-2,6-dinitrotoluene (4-am-dnt)
HPLC	EPA 8330A/B	4-Nitrotoluene

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
HPLC	EPA 8330A/B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC	EPA 8330A/B	Nitrobenzene
HPLC	EPA 8330A/B	Nitroglycerin
HPLC	EPA 8330A/B	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)
HPLC	EPA 8330A/B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC	EPA 8330A/B	Pentaerythritoltetranitrate (PETN)
HPLC	EPA 8330A/B	2,4-diamino-6-Nitrotoluene
HPLC	EPA 8330A/B	2,6-diamino-4-Nitrotoluene
HPLC	EPA 8330A/B	DNX
HPLC	EPA 8330A/B	MNX
HPLC	EPA 8330A/B	TNX
LC/MS/MS	EPA 6850	Perchlorate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorobutanoic Acid (PFBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoropentanoic Acid (PFPeA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorohexanoic Acid (PFHxA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoroheptanoic Acid (PFHpA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctanoic Acid (PFOA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorononanoic Acid (PFNA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorodecanoic Acid (PFDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoroundecanoic Acid (PFUnA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorododecanoic Acid (PFDoA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorotridecanoic Acid (PFTrDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorotetradecanoic Acid (PFTA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorobutanesulfonic Acid (PFBS)

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorohexanesulfonic Acid (PFHxS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctanesulfonic Acid (PFOS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorononanesulfonic Acid (PFNS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorodecanesulfonic Acid (PFDS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoroheptanesulfonic Acid (PFHpS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoropentanesulfonic Acid (PFPeS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctane sulfonamide (PFOSA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Methyl perfluorooctanesulfonamidoacetic acid (MeFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Ethyl perfluorooctanesulfonamidoacetic acid (EtFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	4:2 Fluorotelomer Sulfonate (FTS 4:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	6:2 Fluorotelomer Sulfonate (FTS 6:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	8:2 Fluorotelomer Sulfonate (FTS 8:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	ADONA
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	2,3,3,3-Tetrafluoro-2- (heptafluoropropoxy)propanoic acid (HFPO-DA; GenX)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	11-Chloroeicosafluoro-3-oxaundecane-1- sulfonic acid (11Cl-PF3OUdS; F53B minor)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	9-Chlorohexadecafluoro-3-oxanone-1- sulfonic acid (9Cl-PF3ONS; F53B major)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	3:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	5:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	7:3 Fluorotelomer carboxylate

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	10:2 Fluorotelomer sulfonate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorododecanesulfonic acid
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoro-3-methoxypropanoic acid (PFMPA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoro-4-methoxybutanoic acid (PFMBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoro (2-ethoxyethane) sulfonic acid (PFEEESA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorohexadecanoic acid (PFHxDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctadecanoic acid (PFOcDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	4-PFecHS (Perfluoro-4-ethylcyclohexanesulfonate)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Methyl perfluorooctane sulfonamide
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Ethyl perfluorooctane sulfonamide
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Methyl perfluorooctane sulfonamidoethanol
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Ethyl perfluorooctane sulfonamidoethanol
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorobutanoic Acid (PFBA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoropentanoic Acid (PFPeA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorohexanoic Acid (PFHxA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoroheptanoic Acid (PFHpA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorooctanoic Acid (PFOA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorononanoic Acid (PFNA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorodecanoic Acid (PFDA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoroundecanoic Acid (PFUnA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorododecanoic Acid (PFDoA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorotridecanoic Acid (PFTrDA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorotetradecanoic Acid (PFTA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorobutanesulfonic Acid (PFBS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorohexanesulfonic Acid (PFHxS)

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorooctanesulfonic Acid (PFOS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorononanesulfonic Acid (PFNS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorodecanesulfonic Acid (PFDS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoroheptanesulfonic acid (PFHpS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoropentanesulfonic Acid (PFPeS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorododecanesulfonic Acid (PFDoS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (FTS 4:2)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (FTS 6:2)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (FTS 8:2)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	3-Perfluoropropyl propanoic acid (3:3 FTCA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	2H,2H,3H,3H-Perfluorooctanoic acid (5:3 FTCA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	3-Perfluoroheptyl propanoic acid (7:3 FTCA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorooctanesulfonamide (PFOSA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	N-Methyl perfluorooctanesulfonamide (NMeFOSA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	N-Ethyl perfluorooctanesulfonamide (NEtFOSA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	N-Methyl erfluorooctanesulfonamidoacetic acid (MeFOSAA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	N-Ethyl perfluorooctanesulfonamidoacetic acid (EtFOSAA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	N-Methyl perfluorooctane sulfonamidoethanol (NMeFOSE)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	N-Ethyl perfluorooctane sulfonamidoethanol (NEtFOSE)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	4,8-Dioxa-3H-perfluorononanoic acid (ADONA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Hexafluoropropylene oxide dimer acid (HFPO-DA)



Non-Potable Water		
Technology	Method	Analyte
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoro-3-methoxypropanoic acid (PFMPA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoro-4-methoxybutanoic acid (PFMBA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoro (2-ethoxyethane) sulfonic acid (PFEESA)
ICP	EPA 200.7; EPA 6010C/D	Aluminum
ICP	EPA 200.7; EPA 6010C/D	Antimony
ICP	EPA 200.7; EPA 6010C/D	Arsenic
ICP	EPA 200.7; EPA 6010C/D	Barium
ICP	EPA 200.7; EPA 6010C/D	Beryllium
ICP	EPA 200.7; EPA 6010C/D	Cadmium
ICP	EPA 200.7; EPA 6010C/D	Calcium
ICP	EPA 200.7; EPA 6010C/D	Chromium
ICP	EPA 200.7; EPA 6010C/D	Cobalt
ICP	EPA 200.7; EPA 6010C/D	Copper
ICP	EPA 200.7; EPA 6010C/D	Iron
ICP	EPA 200.7; EPA 6010C/D	Lead
ICP	EPA 200.7; EPA 6010C/D	Magnesium
ICP	EPA 200.7; EPA 6010C/D	Manganese
ICP	EPA 200.7; EPA 6010C/D	Molybdenum
ICP	EPA 200.7; EPA 6010C/D	Nickel
ICP	EPA 200.7; EPA 6010C/D	Potassium
ICP	EPA 200.7; EPA 6010C/D	Selenium
ICP	EPA 200.7; EPA 6010C/D	Silver
ICP	EPA 200.7; EPA 6010C/D	Sodium
ICP	EPA 200.7; EPA 6010C/D	Strontium
ICP	EPA 200.7; EPA 6010C/D	Thallium
ICP	EPA 200.7; EPA 6010C/D	Tin
ICP	EPA 200.7; EPA 6010C/D	Titanium
ICP	EPA 200.7; EPA 6010C/D	Vanadium
ICP	EPA 200.7; EPA 6010C/D	Zinc
ICP/MS	EPA 200.8; EPA 6020A/B	Aluminum
ICP/MS	EPA 200.8; EPA 6020A/B	Antimony
ICP/MS	EPA 200.8; EPA 6020A/B	Arsenic



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Non-Potable Water		
Technology	Method	Analyte
ICP/MS	EPA 200.8; EPA 6020A/B	Barium
ICP/MS	EPA 200.8; EPA 6020A/B	Beryllium
ICP/MS	EPA 200.8; EPA 6020A/B	Cadmium
ICP/MS	EPA 200.8; EPA 6020A/B	Calcium
ICP/MS	EPA 200.8; EPA 6020A/B	Chromium
ICP/MS	EPA 200.8; EPA 6020A/B	Cobalt
ICP/MS	EPA 200.8; EPA 6020A/B	Copper
ICP/MS	EPA 200.8; EPA 6020A/B	Iron
ICP/MS	EPA 200.8; EPA 6020A/B	Lead
ICP/MS	EPA 200.8; EPA 6020A/B	Magnesium
ICP/MS	EPA 200.8; EPA 6020A/B	Manganese
ICP/MS	EPA 200.8; EPA 6020A/B	Molybdenum
ICP/MS	EPA 200.8; EPA 6020A/B	Nickel
ICP/MS	EPA 200.8; EPA 6020A/B	Potassium
ICP/MS	EPA 200.8; EPA 6020A/B	Selenium
ICP/MS	EPA 200.8; EPA 6020A/B	Silver
ICP/MS	EPA 200.8; EPA 6020A/B	Sodium
ICP/MS	EPA 200.8; EPA 6020A/B	Strontium
ICP/MS	EPA 200.8; EPA 6020A/B	Thallium
ICP/MS	EPA 200.8; EPA 6020A/B	Tin
ICP/MS	EPA 200.8; EPA 6020A/B	Titanium
ICP/MS	EPA 200.8; EPA 6020A/B	Vanadium
ICP/MS	EPA 200.8; EPA 6020A/B	Zinc
CVAA	EPA 7470A	Mercury
CVAA	EPA 245.1	Mercury
UV/VIS	EPA 7196A	Hexavalent Chromium (Cr6+)
UV/VIS	EPA 9012B	Cyanide (Total)
IC	EPA 300; EPA 9056A	Bromide
IC	EPA 300; EPA 9056A	Chloride
IC	EPA 300; EPA 9056A	Fluoride
IC	EPA 300; EPA 9056A	Nitrate
IC	EPA 300; EPA 9056A	Nitrite
IC	EPA 300; EPA 9056A	Sulfate
IC	EPA 300; EPA 9056A	Total nitrate-nitrite
IC	EPA 300; EPA 9056A	Orthophosphate

<b>Non-Potable Water</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
Automated Colorimetry	EPA 350.1	Ammonia
Automated Colorimetry	EPA 350.1	Ammonia, Gas Diffusion Option
Automated Colorimetry	EPA 351.2	Total Kjeldahl Nitrogen
Automated Colorimetry	EPA 353.2	Nitrate
Automated Colorimetry	EPA 353.2	Nitrite
Automated Colorimetry	EPA 353.2	Nitrate + Nitrite
Manual Colorimetry	EPA 365.4	Orthophosphate
Automated Colorimetry	EPA 365.1	Orthophosphate
Automated Colorimetry	EPA 365.1	Total Phosphorus
Manual Colorimetry	EPA 365.4	Total Phosphorus
Titrimetric	SM 2320B-11	Alkalinity, Total
Titrimetric	SM 4500-S2 F-11	Sulfide, Iodometric
Gravimetric Methods	EPA 1664A; EPA 1664B; EPA 9070A	Oil and Grease
Gravimetric Methods	SM 2540B-11	Total Residue (Total Solids)
Gravimetric Methods	SM 2540C-11	Filterable Residue (Total Dissolved Solids)
Gravimetric Methods	SM 2540D-11	Non-Filterable Residue (Total Suspended Solids)
Electrometric Methods	SM 4500H+B-11; EPA 9040C	Hydrogen Ion (Ph)
Electrometric Methods	EPA 120.1	Specific conductivity
Combustion	EPA 9060A	Total Organic Carbon
Combustion	SM 5310B-11	Total Organic Carbon
Ignitability	EPA 1020B/ASTM D3278-78	Flash Point
Waste Characterization	EPA Ch.7	Reactive Cyanide and Reactive Sulfide
Waste Characterization	EPA Section 7.3	Reactive Cyanide
Waste Characterization	EPA Section 7.3	Reactive Sulfide
<b>Preparation</b>	<b>Method</b>	<b>Type</b>
Organic Preparation	EPA 3510C	Separatory Funnel Liquid-Liquid Extraction
Organic Preparation	EPA 3511	Micro-extraction
Organic Preparation	EPA 3535A; EPA 3535A MOD	Solid Phase Extraction
Organic Preparation	EPA 8151A	Chlorinated Herbicides, Liquid-Liquid Extraction
Organic Preparation	EPA 608; EPA 625	Separatory Funnel Liquid-Liquid Extraction
Volatile Organic Preparation	SW836 5030B	Closed System Purge and Trap



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Non-Potable Water		
Technology	Method	Analyte
Volatile Organic Preparation	EPA 624	Closed System Purge and Trap
Volatile Organic Preparation	SM 6200B-11	Closed System Purge and Trap
Lachat MicroDistillation	EPA 9012B	Cyanide MicroDistillation; proprietary method
Inorganic Preparation	EPA 3010A	Metals Acid Digestion by Hotblock
Inorganic Preparation	EPA 7470A	CVAA Digestion by Hotblock
Organics Cleanup	EPA 3660B	Sulfur Cleanup
Organics Cleanup	EPA 3665A	Sulfuric Acid Cleanup

Solid and Chemical Materials		
Technology	Method	Analyte
GC/ECD	EPA 8011	1,2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1,2-Dibromo-3-Chloropropane (DBCP)
GC/FID	EPA 8015C/D	Diesel range organics (DRO)
GC/FID	EPA 8015C/D	Oil Range Organics (ORO)
GC/FID	EPA 8015C/D	Gasoline range organics (GRO)
GC/ECD	EPA 8081B	4,4' -DDD
GC/ECD	EPA 8081B	4,4' -DDE
GC/ECD	EPA 8081B	4,4' -DDT
GC/ECD	EPA 8081B	Aldrin
GC/ECD	EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Chlordane (tech.)
GC/ECD	EPA 8081B	alpha-Chlordane
GC/ECD	EPA 8081B	gamma-Chlordane
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I
GC/ECD	EPA 8081B	Endosulfan II
GC/ECD	EPA 8081B	Endosulfan sulfate

Solid and Chemical Materials		
Technology	Method	Analyte
GC/ECD	EPA 8081B	Endrin
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin ketone
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	Total PCB
GC/FPD	EPA 8141B	Azinphos-methyl (Guthion)
GC/FPD	EPA 8141B	Bolstar (Sulprofos)
GC/FPD	EPA 8141B	Carbophenothion
GC/FPD	EPA 8141B	Chlorpyrifos
GC/FPD	EPA 8141B	Coumaphos
GC/FPD	EPA 8141B	Demeton-o
GC/FPD	EPA 8141B	Demeton-s
GC/FPD	EPA 8141B	Demeton
GC/FPD	EPA 8141B	Diazinon
GC/FPD	EPA 8141B	Dichlorvos (DDVP Dichlorvos)
GC/FPD	EPA 8141B	Dimethoate
GC/FPD	EPA 8141B	Disulfoton
GC/FPD	EPA 8141B	EPN
GC/FPD	EPA 8141B	Ethion
GC/FPD	EPA 8141B	Ethoprop
GC/FPD	EPA 8141B	Famphur
GC/FPD	EPA 8141B	Fensulfothion
GC/FPD	EPA 8141B	Fenthion
GC/FPD	EPA 8141B	Malathion



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Solid and Chemical Materials		
Technology	Method	Analyte
GC/FPD	EPA 8141B	Merphos
GC/FPD	EPA 8141B	Methyl parathion (Parathion methyl)
GC/FPD	EPA 8141B	Mevinphos
GC/FPD	EPA 8141B	Monocrotophos
GC/FPD	EPA 8141B	Naled
GC/FPD	EPA 8141B	Parathion ethyl
GC/FPD	EPA 8141B	Phorate
GC/FPD	EPA 8141B	Ronnel
GC/FPD	EPA 8141B	Stirofos
GC/FPD	EPA 8141B	Sulfotepp
GC/FPD	EPA 8141B	Tetraethyl pyrophosphate (TEPP)
GC/FPD	EPA 8141B	Thionazin (Zinophos)
GC/FPD	EPA 8141B	Tokuthion (Prothiophos)
GC/FPD	EPA 8141B	Trichloronate
GC/FPD	EPA 8141B	O,O,O-Triethyl phosphorothioate
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop (Dichlorprop)
GC/ECD	EPA 8151A	Dinoseb (2-sec-butyl-4,6-dinitrophenol DNBP)
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2,4,5-TP)
GC/FID	FL-PRO	Total Petroleum Hydrocarbons (TPH)
GC/FID	MA-VPH	Volatile petroleum range organics (VPH)
GC/FID	MA-EPH	Extractable petroleum range organics (EPH)
GC/FID	IA-OA1	Gasoline range organics (GRO)
GC/FID	IA-OA2	Diesel range organics (DRO)
GC/FID	TN-GRO	Gasoline range organics (GRO)
GC/FID	TN-EPH	Extractable petroleum range organics (EPH)



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Solid and Chemical Materials		
Technology	Method	Analyte
GC/FID	KS LRH	Low-range Hydrocarbons (LRH)
GC/FID	KS MRH	Mid-Range Hydrocarbons (MRH)
GC/FID	KS HRH	High-Range Hydrocarbons (HRH)
GC/MS	EPA 8260C/D	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260C/D	1,1,1-Trichloroethane
GC/MS	EPA 8260C/D	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260C/D	1,1,2-Trichloroethane
GC/MS	EPA 8260C/D	1,1-Dichloroethane
GC/MS	EPA 8260C/D	1,1-Dichloroethylene
GC/MS	EPA 8260C/D	1,1-Dichloropropene
GC/MS	EPA 8260C/D	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260C/D	1,2,3-Trichlorobenzene
GC/MS	EPA 8260C/D	1,2,3-Trichloropropane
GC/MS	EPA 8260C/D	1,2,4-Trichlorobenzene
GC/MS	EPA 8260C/D	1,2,4-Trimethylbenzene
GC/MS	EPA 8260C/D	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260C/D	1,2-Dibromoethane (EDB Ethylene dibromide)
GC/MS	EPA 8260C/D	1,2-Dichlorobenzene (o-Dichlorobenzene)
GC/MS	EPA 8260C/D	1,2-Dichloroethane
GC/MS	EPA 8260C/D	1,2-Dichloroethene (total)
GC/MS	EPA 8260C/D	1,2-Dichloropropane
GC/MS	EPA 8260C/D	1,2-Dichlorotrifluoroethane (Freon 123)
GC/MS	EPA 8260C/D	1,3,5-Trimethylbenzene
GC/MS	EPA 8260C/D	1,3-Dichlorobenzene (m-Dichlorobenzene)
GC/MS	EPA 8260C/D	1,3-Dichloropropane
GC/MS	EPA 8260C/D	1,4-Dichlorobenzene (p-Dichlorobenzene)
GC/MS	EPA 8260C/D	1-Chlorohexane
GC/MS	EPA 8260C/D	2,2-Dichloropropane
GC/MS	EPA 8260C/D	2-Butanone (Methyl ethyl ketone MEK)
GC/MS	EPA 8260C/D	2-Chloroethyl vinyl ether
GC/MS	EPA 8260C/D	2-Chlorotoluene
GC/MS	EPA 8260C/D	2-Hexanone
GC/MS	EPA 8260C/D	2-Nitropropane
GC/MS	EPA 8260C/D	4-Chlorotoluene



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Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260C/D	4-Methyl-2-pentanone (MBK)
GC/MS	EPA 8260C/D	Acetone
GC/MS	EPA 8260C/D	Acetonitrile
GC/MS	EPA 8260C/D	Acrolein (Propenal)
GC/MS	EPA 8260C/D	Acrylonitrile
GC/MS	EPA 8260C/D	Allyl chloride (3-Chloropropene)
GC/MS	EPA 8260C/D	Benzene
GC/MS	EPA 8260C/D	Benzyl Chloride
GC/MS	EPA 8260C/D	Bromobenzene
GC/MS	EPA 8260C/D	Bromochloromethane
GC/MS	EPA 8260C/D	Bromodichloromethane
GC/MS	EPA 8260C/D	Bromoform
GC/MS	EPA 8260C/D	n-Butylbenzene
GC/MS	EPA 8260C/D	sec-Butylbenzene
GC/MS	EPA 8260C/D	tert-Butylbenzene
GC/MS	EPA 8260C/D	Carbon disulfide
GC/MS	EPA 8260C/D	Carbon tetrachloride
GC/MS	EPA 8260C/D	Chlorobenzene
GC/MS	EPA 8260C/D	Chloroethane
GC/MS	EPA 8260C/D	Chloroform
GC/MS	EPA 8260C/D	Chloroprene
GC/MS	EPA 8260C/D	Cyclohexane
GC/MS	EPA 8260C/D	Cyclohexanone
GC/MS	EPA 8260C/D	cis-1,2-Dichloroethylene
GC/MS	EPA 8260C/D	trans-1,2-Dichloroethylene
GC/MS	EPA 8260C/D	cis-1,3-Dichloropropene
GC/MS	EPA 8260C/D	trans-1,3-Dichloropropylene
GC/MS	EPA 8260C/D	cis-1,4-Dichloro-2-butene
GC/MS	EPA 8260C/D	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260C/D	Di-isopropylether (DIPE)
GC/MS	EPA 8260C/D	Dibromochloromethane
GC/MS	EPA 8260C/D	Dibromomethane (Methylene Bromide)
GC/MS	EPA 8260C/D	Dichlorodifluoromethane
GC/MS	EPA 8260C/D	Diethyl ether
GC/MS	EPA 8260C/D; EPA 8260C/D SIM	p-Dioxane (1,4-Dioxane)
GC/MS	EPA 8260C/D	Ethanol (Ethyl Alcohol)





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Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260C/D	Ethyl acetate
GC/MS	EPA 8260C/D	Ethyl methacrylate
GC/MS	EPA 8260C/D	Ethyl tert-butyl alcohol (ETBA)
GC/MS	EPA 8260C/D	Ethyl tert-butyl ether (ETBE)
GC/MS	PA 8260C/D	Ethylbenzene
GC/MS	EPA 8260C/D	Ethylene Oxide
GC/MS	EPA 8260C/D	Hexachlorobutadiene
GC/MS	EPA 8260C/D	Hexane
GC/MS	EPA 8260C/D	Iodomethane (Methyl iodide)
GC/MS	EPA 8260C/D	Isobutyl alcohol (2-Methyl-1-propanol)
GC/MS	EPA 8260C/D	p-Isopropyltoluene
GC/MS	EPA 8260C/D	Isopropylbenzene
GC/MS	EPA 8260C/D	Methacrylonitrile
GC/MS	EPA 8260C/D	Methyl Acetate
GC/MS	EPA 8260C/D	Methyl bromide (Bromomethane)
GC/MS	EPA 8260C/D	Methyl chloride (Chloromethane)
GC/MS	EPA 8260C/D	Methylcyclohexane
GC/MS	EPA 8260C/D	Methyl methacrylate
GC/MS	EPA 8260C/D	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260C/D	Methylene chloride
GC/MS	EPA 8260C/D	Naphthalene
GC/MS	EPA 8260C/D	Pentachloroethane
GC/MS	EPA 8260C/D	Propionitrile (Ethyl cyanide)
GC/MS	EPA 8260C/D	n-Propylbenzene
GC/MS	EPA 8260C/D	Styrene
GC/MS	EPA 8260C/D	tert-Amyl alcohol (TAA)
GC/MS	EPA 8260C/D	tert-Amyl methyl ether (TAME)
GC/MS	EPA 8260C/D	tert-Butyl alcohol (TBA)
GC/MS	EPA 8260C/D	tert-Butyl formate (TBF)
GC/MS	EPA 8260C/D	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA 8260C/D	Tetrahydrofuran
GC/MS	EPA 8260C/D	Toluene
GC/MS	EPA 8260C/D	Trichloroethene (Trichloroethylene)
GC/MS	EPA 8260C/D	Trichlorofluoromethane
GC/MS	EPA 8260C/D	Vinyl acetate
GC/MS	EPA 8260C/D	Vinyl chloride



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Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8260C/D	Xylene (total)
GC/MS	EPA 8260C/D	m,p-Xylene
GC/MS	EPA 8260C/D	o-Xylene
GC/MS	EPA 8270D/E	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270D/E	1,2,4-Trichlorobenzene
GC/MS	EPA 8270D/E	1,2-Dichlorobenzene (o-Dichlorobenzene)
GC/MS	EPA 8270D/E	1,2-Diphenylhydrazine
GC/MS	EPA 8270D/E	1,3,5-Trinitrobenzene (1,3,5-TNB)
GC/MS	EPA 8270D/E	1,3-Dichlorobenzene (m-Dichlorobenzene)
GC/MS	EPA 8270D/E	1,3-Dinitrobenzene (1,3-DNB)
GC/MS	EPA 8270D/E	1,4-Dichlorobenzene (p-Dichlorobenzene)
GC/MS	EPA 8270D/E	1,4-Naphthoquinone
GC/MS	EPA 8270D/E	1,4-Phenylenediamine
GC/MS	EPA 8270D/E	1-Chloronaphthalene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	1-Methylnaphthalene
GC/MS	EPA 8270D/E	1-Naphthylamine
GC/MS	EPA 8270D/E	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270D/E	2,4,5-Trichlorophenol
GC/MS	EPA 8270D/E	2,4,6-Trichlorophenol
GC/MS	EPA 8270D/E	2,4-Dichlorophenol
GC/MS	EPA 8270D/E	2,4-Dimethylphenol
GC/MS	EPA 8270D/E	2,4-Dinitrophenol
GC/MS	EPA 8270D/E	2,4-Dinitrotoluene (2,4-DNT)
GC/MS	EPA 8270D/E	2,6-Dichlorophenol
GC/MS	EPA 8270D/E	2,6-Dinitrotoluene (2,6-DNT)
GC/MS	EPA 8270D/E	2-Acetylaminofluorene
GC/MS	EPA 8270D/E	2-Chloronaphthalene
GC/MS	EPA 8270D/E	2-Chlorophenol
GC/MS	EPA 8270D/E	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-o-cresol)
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	2-Methylnaphthalene
GC/MS	EPA 8270D/E	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270D/E	2-Naphthylamine
GC/MS	EPA 8270D/E	2-Nitroaniline
GC/MS	EPA 8270D/E	2-Nitrophenol
GC/MS	EPA 8270D/E	2-Picoline (2-Methylpyridine)



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Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270D/E	3,3'-Dichlorobenzidine
GC/MS	EPA 8270D/E	3,3'-Dimethylbenzidine
GC/MS	EPA 8270D/E	3-Methylcholanthrene
GC/MS	EPA 8270D/E	3&4-Methylphenol (m,p-Cresol)
GC/MS	EPA 8270D/E	3-Nitroaniline
GC/MS	EPA 8270D/E	4-Aminobiphenyl
GC/MS	EPA 8270D/E	4-Bromophenyl phenyl ether
GC/MS	EPA 8270D/E	4-Chloro-3-methylphenol
GC/MS	EPA 8270D/E	4-Chloroaniline
GC/MS	EPA 8270D/E	4-Chlorophenyl phenylether
GC/MS	EPA 8270D/E	4-Dimethyl aminoazobenzene
GC/MS	EPA 8270D/E	4-Nitroaniline
GC/MS	EPA 8270D/E	4-Nitrophenol
GC/MS	EPA 8270D/E	5-Nitro-o-toluidine
GC/MS	EPA 8270D/E	7,12-Dimethylbenz(a) anthracene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Acenaphthene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Acenaphthylene
GC/MS	EPA 8270D/E	Acetophenone
GC/MS	EPA 8270D/E	Aniline
GC/MS	EPA 8270D; EPA 8270D SIM	Anthracene
GC/MS	EPA 8270D/E	Aramite
GC/MS	EPA 8270D/E	Atrazine
GC/MS	EPA 8270D/E	Benzaldehyde
GC/MS	EPA 8270D/E	Benzidine
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Benzo(a)anthracene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Benzo(a)pyrene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270D/E	Benzoic acid
GC/MS	EPA 8270D/E	Benzyl alcohol
GC/MS	EPA 8270D/E	Biphenyl (1,1'-Biphenyl)
GC/MS	EPA 8270D/E	bis(2-Chloroethoxy) methane
GC/MS	EPA 8270D/E	bis(2-Chloroethyl) ether
GC/MS	EPA 8270D/E	bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))



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Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270D/E	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 8270D/E	Butyl benzyl phthalate
GC/MS	EPA 8270D/E	Carbazole
GC/MS	EPA 8270D/E	Caprolactam
GC/MS	EPA 8270D/E	Chlorobenzilate
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Chrysene
GC/MS	EPA 8270D/E	Diallate
GC/MS	EPA 8270D/E	Di-n-butyl phthalate
GC/MS	EPA 8270D/E	Di-n-octyl phthalate
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Dibenz(a,h)anthracene
GC/MS	EPA 8270D/E	Dibenz(a,j)acridine
GC/MS	EPA 8270D/E	Dibenzofuran
GC/MS	EPA 8270D/E	Diethyl phthalate
GC/MS	EPA 8270D/E	Dimethyl phthalate
GC/MS	EPA 8270D/E	a,a-Dimethylphenethylamine
GC/MS	EPA 8270D/E	Diphenyl Ether
GC/MS	EPA 8270D/E EPA 8270D/E SIM	p-Dioxane (1,4-Dioxane)
GC/MS	EPA 8270D/E	Ethyl methanesulfonate
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Fluoranthene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Fluorene
GC/MS	EPA 8270D/E	Hexachlorobenzene
GC/MS	EPA 8270D/E	Hexachlorobutadiene
GC/MS	EPA 8270D/E	Hexachlorocyclopentadiene
GC/MS	EPA 8270D/E	Hexachloroethane
GC/MS	EPA 8270D/E	Hexachlorophene
GC/MS	EPA 8270D/E	Hexachloropropene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270D/E	Isodrin
GC/MS	EPA 8270D/E	Isophorone
GC/MS	EPA 8270D/E	Isosafrole
GC/MS	EPA 8270D/E	Kepone
GC/MS	EPA 8270D/E	Methapyrilene
GC/MS	EPA 8270D/E	Methyl methanesulfonate
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Naphthalene
GC/MS	EPA 8270D/E	Nitrobenzene



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Solid and Chemical Materials		
Technology	Method	Analyte
GC/MS	EPA 8270D/E	Nitroquinoline-1-oxide
GC/MS	EPA 8270D/E	n-Nitroso-di-n-butylamine
GC/MS	EPA 8270D/E	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270D/E	n-Nitrosodiethylamine
GC/MS	EPA 8270D/E	n-Nitrosodimethylamine
GC/MS	EPA 8270D/E	n-Nitrosodiphenylamine
GC/MS	EPA 8270D/E	n-Nitrosodiphenylamine/Diphenylamine (analyte pair)
GC/MS	EPA 8270D/E	n-Nitrosomethylethylamine
GC/MS	EPA 8270D/E	n-Nitrosomorpholine
GC/MS	EPA 8270D/E	n-Nitrosopiperidine
GC/MS	EPA 8270D/E	n-Nitrosopyrrolidine
GC/MS	EPA 8270D/E	Pentachlorobenzene
GC/MS	EPA 8270D/E	Pentachloroethane
GC/MS	EPA 8270D/E	Pentachloronitrobenzene
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Pentachlorophenol
GC/MS	EPA 8270D/E	Phenacetin
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Phenanthrene
GC/MS	EPA 8270D/E	Phenol
GC/MS	EPA 8270D/E	Pronamide (Kerb)
GC/MS	EPA 8270D/E; EPA 8270D/E SIM	Pyrene
GC/MS	EPA 8270D/E	Pyridine
GC/MS	EPA 8270D/E	Safrole
GC/MS	EPA 8270D/E	Simazine
GC/MS	EPA 8270D/E	o-Toluidine
GC/MS	EPA 8270D/E	Dimethoate
GC/MS	EPA 8270D/E	Disulfoton
GC/MS	EPA 8270D/E	Famphur
GC/MS	EPA 8270D/E	Methyl parathion (Parathion methyl)
GC/MS	EPA 8270D/E	Parathion ethyl
GC/MS	EPA 8270D/E	Phorate
GC/MS	EPA 8270D/E	Sulfotepp
GC/MS	EPA 8270D/E	Thionazin (Zinophos)
GC/MS	EPA 8270D/E	O,O,O-Triethyl phosphorothioate
HPLC	EPA 8330A/B	1,3,5-Trinitrobenzene (1,3,5-TNB)
HPLC	EPA 8330A/B	1,3-Dinitrobenzene (1,3-DNB)

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
HPLC	EPA 8330A/B	2,4,6-Trinitrotoluene (2,4,6-TNT)
HPLC	EPA 8330A/B	2,4-Dinitrotoluene (2,4-DNT)
HPLC	EPA 8330A/B	2,6-Dinitrotoluene (2,6-DNT)
HPLC	EPA 8330A/B	2-Amino-4,6-dinitrotoluene (2-am-dnt)
HPLC	EPA 8330A/B	2-Nitrotoluene
HPLC	EPA 8330A/B	3,5-Dinitroaniline
HPLC	EPA 8330A/B	3-Nitrotoluene
HPLC	EPA 8330A/B	4-Amino-2,6-dinitrotoluene (4-am-dnt)
HPLC	EPA 8330A/B	4-Nitrotoluene
HPLC	EPA 8330A/B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC	EPA 8330A/B	Nitrobenzene
HPLC	EPA 8330A/B	Nitroglycerin
HPLC	EPA 8330A/B	Methyl-2,4,6-trinitrophenylnitramine (Tetryl)
HPLC	EPA 8330A/B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC	EPA 8330A/B	Pentaerythritoltetranitrate (PETN)
HPLC	EPA 8330A/B	DNX
HPLC	EPA 8330A/B	MNX
HPLC	EPA 8330A/B	TNX
LC/MS/MS	EPA 6850	Perchlorate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorobutanoic Acid (PFBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoropentanoic Acid (PFPeA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorohexanoic Acid (PFHxA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoroheptanoic Acid (PFHpA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctanoic Acid (PFOA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorononanoic Acid (PFNA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorodecanoic Acid (PFDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoroundecanoic Acid (PFUnA)

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorododecanoic Acid (PFDoA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorotridecanoic Acid (PFTrDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorotetradecanoic Acid (PFTA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorobutanesulfonic Acid (PFBS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorohexanesulfonic Acid (PFHxS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctanesulfonic Acid (PFOS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorononanesulfonic Acid (PFNS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorodecanesulfonic Acid (PFDS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoroheptanesulfonic Acid (PFHpS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoropentanesulfonic Acid (PFPeS)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctane sulfonamide (PFOSA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Methyl perfluorooctanesulfonamidoacetic acid (MeFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Ethyl perfluorooctanesulfonamidoacetic acid (EtFOSAA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	4:2 Fluorotelomer Sulfonate (FTS 4:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	6:2 Fluorotelomer Sulfonate (FTS 6:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	8:2 Fluorotelomer Sulfonate (FTS 8:2)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	ADONA
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	2,3,3,3-Tetrafluoro-2- (heptafluoropropoxy)propanoic acid (HFPO-DA; GenX)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	11-Chloroeicosafluoro-3-oxaundecane-1- sulfonic acid (11Cl-PF3OUdS; F53B minor)

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS; F53B major)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	3:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	5:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	7:3 Fluorotelomer carboxylate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	10:2 Fluorotelomer sulfonate
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorododecanesulfonic acid
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoro-3-methoxypropanoic acid (PFMPA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoro-4-methoxybutanoic acid (PFMBA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluoro (2-ethoxyethane) sulfonic acid (PFEEESA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorohexadecanoic acid (PFHxDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	Perfluorooctadecanoic acid (PFOcDA)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	4-PFecHS (Perfluoro-4-ethylcyclohexanesulfonate)
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Methyl perfluorooctane sulfonamide
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Ethyl perfluorooctane sulfonamide
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Methyl perfluorooctane sulfonamidoethanol
LC/MS/MS	PFAS by LCMSMS Compliant with QSM 5.4 Table B-15	N-Ethyl perfluorooctane sulfonamidoethanol
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorobutanoic Acid (PFBA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoropentanoic Acid (PFPeA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorohexanoic Acid (PFHxA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoroheptanoic Acid (PFHpA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorooctanoic Acid (PFOA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorononanoic Acid (PFNA)



<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorodecanoic Acid (PFDA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoroundecanoic Acid (PFUnA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorododecanoic Acid (PFDoA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorotridecanoic Acid (PFTrDA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorotetradecanoic Acid (PFTA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorobutanesulfonic Acid (PFBS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorohexanesulfonic Acid (PFHxS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorooctanesulfonic Acid (PFOS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorononanesulfonic Acid (PFNS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorodecanesulfonic Acid (PFDS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoroheptanesulfonic acid (PFHpS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoropentanesulfonic Acid (PFPeS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorododecanesulfonic Acid (PFDoS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (FTS 4:2)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (FTS 6:2)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (FTS 8:2)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	3-Perfluoropropyl propanoic acid (3:3 FTCA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	2H,2H,3H,3H-Perfluorooctanoic acid (5:3 FTCA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	3-Perfluoroheptyl propanoic acid (7:3 FTCA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluorooctanesulfonamide (PFOSA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	N-Methyl perfluorooctanesulfonamide (NMeFOSA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	N-Ethyl perfluorooctanesulfonamide (NEtFOSA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	N-Methyl erfluorooctanesulfonamidoacetic acid (MeFOSAA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	N-Ethyl perfluorooctanesulfonamidoacetic acid (EtFOSAA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	N-Methyl perfluorooctane sulfonamidoethanol (NMeFOSE)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	N-Ethyl perfluorooctane sulfonamidoethanol (NEtFOSE)

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
LC/MS/MS	EPA Draft Method 1633 Rev. 2	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	4,8-Dioxa-3H-perfluorononanoic acid (ADONA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Hexafluoropropylene oxide dimer acid (HFPO-DA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoro-3-methoxypropanoic acid (PFMPA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoro-4-methoxybutanoic acid (PFMBA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Nonafluoro-3,6-dioxaheptanoic acid (NFDHA)
LC/MS/MS	EPA Draft Method 1633 Rev. 2	Perfluoro (2-ethoxyethane) sulfonic acid (PFEEESA)
ICP	EPA 6010C/D	Aluminum
ICP	EPA 6010C/D	Antimony
ICP	EPA 6010C/D	Arsenic
ICP	EPA 6010C/D	Barium
ICP	EPA 6010C/D	Beryllium
ICP	EPA 6010C/D	Cadmium
ICP	EPA 6010C/D	Calcium
ICP	EPA 6010C/D	Chromium
ICP	EPA 6010C/D	Cobalt
ICP	EPA 6010C/D	Copper
ICP	EPA 6010C/D	Iron
ICP	EPA 6010C/D	Lead
ICP	EPA 6010C/D	Magnesium
ICP	EPA 6010C/D	Manganese
ICP	EPA 6010C/D	Molybdenum
ICP	EPA 6010C/D	Nickel
ICP	EPA 6010C/D	Potassium
ICP	EPA 6010C/D	Selenium
ICP	EPA 6010C/D	Silver
ICP	EPA 6010C/D	Sodium
ICP	EPA 6010C/D	Strontium
ICP	EPA 6010C/D	Thallium



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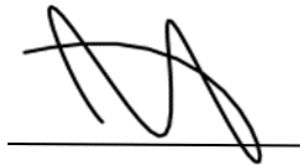
Solid and Chemical Materials		
Technology	Method	Analyte
ICP	EPA 6010C/D	Tin
ICP	EPA 6010C/D	Titanium
ICP	EPA 6010C/D	Vanadium
ICP	EPA 6010C/D	Zinc
ICP/MS	EPA 6020A/B	Aluminum
ICP/MS	EPA 6020A/B	Antimony
ICP/MS	EPA 6020A/B	Arsenic
ICP/MS	EPA 6020A/B	Barium
ICP/MS	EPA 6020A/B	Beryllium
ICP/MS	EPA 6020A/B	Cadmium
ICP/MS	EPA 6020A/B	Calcium
ICP/MS	EPA 6020A/B	Chromium
ICP/MS	EPA 6020A/B	Cobalt
ICP/MS	EPA 6020A/B	Copper
ICP/MS	EPA 6020A/B	Iron
ICP/MS	EPA 6020A/B	Lead
ICP/MS	EPA 6020A/B	Magnesium
ICP/MS	EPA 6020A/B	Manganese
ICP/MS	EPA 6020A/B	Molybdenum
ICP/MS	EPA 6020A/B	Nickel
ICP/MS	EPA 6020A/B	Potassium
ICP/MS	EPA 6020A/B	Selenium
ICP/MS	EPA 6020A/B	Silver
ICP/MS	EPA 6020A/B	Sodium
ICP/MS	EPA 6020A/B	Strontium
ICP/MS	EPA 6020A/B	Thallium
ICP/MS	EPA 6020A/B	Tin
ICP/MS	EPA 6020A/B	Titanium
ICP/MS	EPA 6020A/B	Vanadium
ICP/MS	EPA 6020A/B	Zinc
CVAA	EPA 7471B	Mercury
UV/VIS	EPA 7196A	Hexavalent Chromium (Cr6+)
UV/VIS	EPA 9012B	Cyanide (Total)
IC	EPA 9056A	Bromide

<b>Solid and Chemical Materials</b>		
<b>Technology</b>	<b>Method</b>	<b>Analyte</b>
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate
IC	EPA 9056A	Nitrite
IC	EPA 9056A	Sulfate
IC	EPA 9056A	Total nitrate-nitrite
Gravimetric Methods	SM 2540G	% solids
Electrometric Methods	EPA 9045D	Hydrogen Ion (pH)
Ignitability	EPA 1020B MOD	Flash Point
Waste Characterization	EPA Ch.7	Reactive Cyanide and Reactive Sulfide
Waste Characterization	EPA Section 7.3	Reactive Cyanide
Waste Characterization	EPA Section 7.3	Reactive Sulfide
<b>Preparation</b>	<b>Method</b>	<b>Type</b>
Organics Preparation	EPA 3510C	Separatory Funnel Liquid-Liquid Extraction; Leachates
TCLP Preparation	EPA 1311	Toxicity Characteristic Leaching Procedure
SPLP Preparation	EPA 1312	Synthetic Precipitation Leaching Procedure
Organics Preparation	EPA 8011	Microextraction
Organics Preparation	EPA 3546	Microwave Extraction
Organics Preparation	EPA 3550C	Ultrasonic Extraction
Organics Preparation	EPA 3580A	Waste Dilution for Extractable Organics
Organics Preparation	EPA 8330A; EPA 8332	Ultrasonic Extraction
Organics Preparation	EPA 8330B	Shaker Table Extraction
Volatile Organics Preparation	EPA 3585	Waste Dilution for Volatile Organics
Volatile Organics Preparation	EPA 5030A	Closed System Purge and Trap; Bulk Soils
Volatile Organics Preparation	EPA 5030B	Closed System Purge and Trap; Leachates and Methanol Extracts
Volatile Organics Preparation	EPA 5035; EPA 5035A	Closed System Purge and Trap
Organics Cleanup	EPA 3660B	Sulfur Cleanup
Organics Cleanup	EPA 3665A	Sulfuric Acid Cleanup
Lachat MicroDistillation	EPA 9012B	Cyanide MicroDistillation; proprietary method
Inorganic Preparation	EPA 3010A	Metals Acid Digestion by Hotblock; Leachates

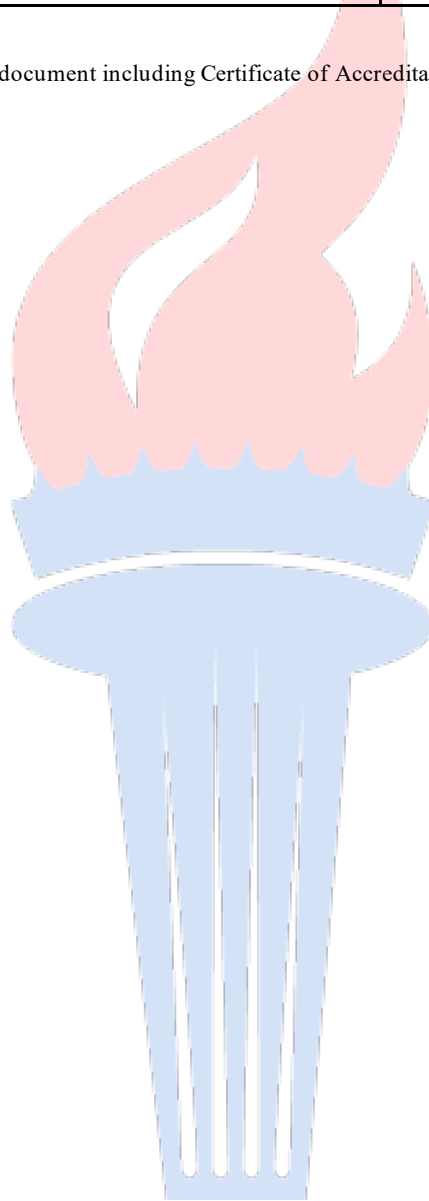
Solid and Chemical Materials		
Technology	Method	Analyte
Inorganic Preparation	EPA 3050B	Metals Acid Digestion by Hotblock
Inorganic Preparation	EPA 3060A	Alkaline Digestion, Cr6+
Inorganic Preparation	EPA 7470A	CVAA Digestion by Hotblock; Leachates
Inorganic Preparation	EPA 7471B	CVAA Digestion by Hotblock

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2229.



Jason Stine, Vice President



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Appendix C  
SGS Laboratory Standard Operating  
Procedures

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## ANALYSIS OF PER- and POLYFLUORINATED ALKYL SUBSTANCES BY LC/MS/MS AND ISOTOPE DILUTION

Prepared by: Norm Farmer Date: 06/05/2023

Approved by: Natasha Gumtie Date: 06/12/2023

### Annual Review

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## TITLE: ANALYSIS OF PER- and POLYFLUORINATED ALKYL SUBSTANCES BY LC/MS/MS AND ISOTOPE DILUTION

### REFERENCES: EPA 533

#### 1.0 SCOPE AND APPLICATION, SUMMARY

##### 1.1 Scope and Application

1.1.1 This method is used to determine the concentrations of select Per- and Polyfluorinated Alkyl Substances (PFAS) in potable and finished drinking water utilizing an HPLC equipped with a tandem mass spectrometer (MS/MS).

1.1.2 The following compounds can be reported by this method:

ANALYTES	SYNONYM	CAS #
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUnDA	2058-94-8
Perfluorododecanoic acid	PFDoDA	307-55-1
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorooctanesulfonic acid	PFOS	1763-23-1
4:2 Fluorotelomer sulfonate	4:2 FTS	757124-72-4
6:2 Fluorotelomer sulfonate	6:2 FTS	27619-97-2
8:2 Fluorotelomer sulfonate	8:2 FTS	39108-34-4
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5
Nonafluoro-3,6-dioxahexanoic acid	NFDHA	151772-58-6
Perfluoro(2-ethoxyethane) sulfonic acid	PFEESA	113507-82-7
Hexafluoropropylene oxide dimer acid	HFPO-DA (GenX)	13252-13-6
4,8-Dioxa-3H-perfluorononanoic acid	ADONA	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

- 1.1.3 The reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. Reporting limits may vary depending on matrix complications and volumes. Reporting limits for this method are in the range of 0.002-0.004 ug/l or 2.0 to 4.0 ng/l.
- 1.1.4 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.
- 1.1.5 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a "J" or "I" qualifier. Some program or project specifications may require that no values below the RL be reported.

## 1.2 Summary

- 1.2.1 This method is adapted from EPA 533 for the analysis of short chained PFAS analytes in potable and finished drinking water. The method utilizes an isotope dilution standard technique.
- 1.2.2 Samples are received, stored, and extracted within the appropriate holding times.
- 1.2.3 Sample preparation is performed in accordance with SGS – Orlando SOP OP072.
- 1.2.4 Perfluorinated compounds are separated, detected, and quantitated using an LC/MS/MS. After HPLC separation and ionization, the specific Perfluorinated compound is isolated in the first mass spectrometer and transferred to a collision cell for fragmentation. The resulting fragments are introduced into the second mass spectrometer where they are detected and quantified.
- 1.2.5 Perfluorinated analytes may exist in branched and/or linear form. Fluorotelomer production results in linear isomers only but electrochemical fluorination results in branched and linear isomers. The branched isomers may account for up to 30% of the total analyte. The branched isomer will elute just before the linear isomer.
- 1.2.6 Manual integrations are performed in accordance with SOP QA029.

## 2.0 PRESERVATION AND HOLDING TIME

### 2.1 Preservation

- 2.1.1 Samples shall be collected in 250mL polypropylene (PP) or polyethylene (HDPE) bottles. Caps must not have Teflon liners. Various studies have shown that HDPE is preferable to PP for this analysis.

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- 2.1.2 1.0 g/l of Ammonium acetate must be added to each sample as a preservative. Ammonium Acetate will sequester free chlorine to form chloramine.
- 2.1.3 The samples must be chilled to  $\leq 10^{\circ}\text{C}$  from the time of collection until arrival at the laboratory. Samples must not exceed  $10^{\circ}\text{C}$  during the first 48 hours after collection. The samples must be refrigerated at  $\leq 6^{\circ}\text{C}$  from the time of receipt until extraction.
- 2.1.4 The extracts must be stored at room temperature to minimize analyte adsorption to the container walls. If extracts are stored  $\leq 6^{\circ}\text{C}$  they must be allowed to come to room temperature prior to analysis. All extracts must be vortexed just prior to transfer to the autosampler vials.

## 2.2 Holding Time

- 2.1.1 Aqueous samples must be extracted within 28 days of collection.
- 2.1.2 Extracts must be analyzed within 28 days of extraction.

## 3.0 INTERFERENCES

- 3.1 Data from all blanks, samples, and spikes must be evaluated for interferences. Method interferences may be caused by contaminants in solvents, reagents, or glassware. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) or Teflon products, HPLC solvent lines, methanol, aluminum foil, SPE transfer lines, bottle caps, etc. All these materials must be demonstrated to be free from interferences.
- 3.2 Contact with glass containers, pipettes, or syringes should be minimized since the Perfluorinated compounds can potentially adsorb to glass surfaces.
- 3.3 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 of the water. Humic and/or fulvic material can be co-extracted during SPE and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent. Total organic carbon (TOC) is a good indicator of the humic content of the sample.
- 3.4 SPE cartridges can be a source of interferences. The analysis of field and method blanks can provide important information regarding the presence or absence of such interferences. Brands and lots of SPE devices must be tested to ensure that contamination does not preclude analyte identification and quantitation.
- 3.5 The background from method analytes or other contaminants that interfere with the measurement of method analytes must be below 1/3 of the Reporting Limit.
- 3.6 Water and containers used for equipment blanks or field blanks must be tested prior to use. For smaller sampling events DI water will be provided in the same type of bottle

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used for sample collection. For larger sampling events four-liter HDPE containers should be used. Containers should be filled with DI water and allowed to sit for several hours before testing. If the bottles are from the same lot and filled with DI on the same day, then one analysis per 10 containers should suffice. The DI water and container blanks must be free of any analytes of interest or interferences at 1/3 the required reporting limit to be acceptable.

- 3.7 A field blank should be collected with each set of samples. Each field blank consists of 4 bottles. Two bottles are filled with DI water at the lab and the other two bottles are empty. If Ammonium Acetate is being used for the samples then the two bottles with DI water should also contain Ammonium Acetate. At the sampling site the sampler should open then two empty bottles and transfer the DI water from the full bottles into them. Cap the bottles, label as field blanks, and return them to the laboratory along with the samples for analysis.

#### **4.0 DEFINITIONS**

- 4.1 **Batch:** A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.
- 4.2 **Blank Spike (BS):** An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS) or Quality Control Sample (QCS).
- 4.3 **Continuing Calibration Verification (CCV):** A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.
- 4.4 **Field Blank (FB):** 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, preservation, and all analytical procedures. The purpose of the FB is to determine if method analytes or other interferences are present in the field environment.
- 4.5 **Holding Time:** The maximum times that samples may be held prior to preparation and/or analysis and still considered valid.
- 4.6 **Isotope Dilution Standards (Extracted Internal Standards):** A standard containing isotopically labelled versions of the native target analytes. These isotopes are usually labelled with C13 or O18 atoms. Isotope Dilution Standards are used to measure the extraction efficiency and to correct the concentrations of the native analytes based on the recovery of their isotopically labelled analogs. The terms isotope dilution standards and extracted internal standard are used interchangeably throughout this SOP. Technically if a direct mass labelled analog is used to quantitate the native analyte it is an isotope

dilution technique; however, if a direct mass labelled analog is not available for quantitation and a similar mass labelled analog is used, it is an extracted internal standard technique.

- 4.7 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point must be at a level equal to or below the LLOQ.
- 4.8 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor must be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard or Second Source standard.
- 4.9 Matrix Spike (MS): A sample spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.
- 4.10 Matrix Spike Duplicate (MSD): A replicate sample spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.
- 4.11 Method Blank (MB): 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 processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.
- 4.12 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.
- 4.13 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

## **5.0 REAGENTS**

- 5.1 Water – HPLC grade or equivalent
  - Water with 0.1% acetic acid: Add 1ml of acetic acid to 1.0 L of HPLC Water
- 5.2 Methanol – HPLC grade or equivalent
  - Methanol with 0.1% acetic acid: Add 1ml of acetic acid to 1.0 L of Methanol
- 5.3 Methanol:Water Solution – 80% methanol and 20% water volume:volume
- 5.4 Acetic Acid – HPLC grade or equivalent
- 5.5 Ammonium Acetate – HPLC grade or equivalent – free of interferences

- 5.6 Technical PFOA standard – Qualitative for branched isomer retention time determination.
- 5.7 Perfluorinated Alkyl Substances stock standards – Traceable to Certificate of Analysis.
- 5.8 Mass labeled – Injection Standards

Perfluoro-[2,3,4-<sup>13</sup>C<sub>2</sub>]butanoic acid (13C3-PFBA)  
 Perfluoro-[1,2-<sup>13</sup>C<sub>2</sub>]octanoic acid (13C2-PFOA)  
 Perfluoro-1-[1,2,3,4-<sup>13</sup>C<sub>4</sub>]octanesulfonic acid (13C4-PFOS)

- 5.9 Mass labeled – Isotope Dilution Standards – Extracted Internal Standards

13C4-PFBA	13C6-PFDA	13C2-4:2FTS
13C5-PFPeA	13C7-PFUnDA	13C2-6:2FTS
13C5-PFHxA	13C2-PFDoDA	13C2-8:2FTS
13C4-PFHpA	13C3-PFBS	13C3-HFPO-DA
13C8-PFOA	13C3-PFHxS	
13C9-PFNA	13C8-PFOS	

## 6.0 APPARATUS

- 6.1 HPLC – Agilent Technologies 1260 or 1290

Suitable HPLC equipped with an autosampler, pump, and column compartment. System may have a membrane degasser.

- 6.2 MS/MS – Agilent Technologies 6460A or 6470A/B

LC/MS/MS must be capable of negative ion electrospray ionization near the required flow rate of the HPLC Column. The system must be capable of performing MS/MS to produce unique precursor and product ions for the PFAS method analytes within the specified retention time segments. A minimum of 10 scans across each peak is required to ensure adequate precision.

- 6.3 Data System – Agilent Technologies MassHunter B.07.0x, B.08.0x, B10.0x.

6.3.1 A computer system interfaced to the HPLC/MS/MS that allows for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

6.3.2 The software must allow for the viewing of the specific MS/MS Spectra acquired over the analytical run. Comparisons can then be made between spectra from standards and samples.

6.3.3 Data is archived to a backup server for long term storage.

- 6.4 Columns: Agilent Poroshell 120 EC C18 2.7um, 100 x 2.1 mm ID or equivalent
- 6.5 Delay Columns: Agilent Poroshell or Eclipse C18 50 x 4.6 mm ID or equivalent
- 6.6 Disposable polyethylene transfer pipettes
- 6.7 15ml Centrifuge tubes - HDPE or PP
- 6.8 Polyethylene screw cap and autosampler vials
- 6.9 Volumetric Pipettors and volumetric “plasticware” for dilutions of standards and extracts.
- 6.10 4-liter LDPE or HDPE containers – For DI water for Equipment Blanks, shown to be PFAS free
- 6.11 250ml polyethylene or polypropylene bottles – shown to be PFAS free

## **7.0 PROCEDURE**

### **7.1 Standards Preparation**

Standards are prepared from commercially available certified neat or reference standards. All standards must be logged in the HPLC Standards Logbook. All standards shall be traceable to their original source. The standards must be stored at  $\leq 6^{\circ}\text{C}$ , or as recommended by the manufacturer. All standards must be allowed to come to room temperature and vortexed prior to use. Calibration levels, spike and surrogate concentrations, preparation information, and vendor part numbers can be found in the MS STD Summary in the Active SOP directory. A summary of the calibration concentrations can be found in Table 2.

#### **7.1.1 Stock Standard Solutions**

Stock standards are available from some commercial vendors. All vendors must supply a “Certificate of Analysis” with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor’s expiration date. Once opened, the hold time is reduced to one year or the vendor’s expiration date (whichever is shorter).

#### **7.1.2 Intermediate Standard Solutions**

Intermediate standards are prepared by quantitative dilution of the stock standard with 80:20 methanol:water. The hold time for intermediate standards is six months or the vendor’s expiration date (whichever is shorter). Intermediate standards may need to be remade if comparisons to other standards indicate analyte degradation or concentration changes. Intermediate standards should be stored in polyethylene vials.



7.1.3 Calibration Standards

Calibration standards for Perfluorinated analytes are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. Calibration standards are prepared in 80:20 methanol:water. The low standard is at a concentration at or below the RL and the remaining standards defines the working range of the detector. Calibration standards must be stored in polyethylene vials.

**Perfluorinated analytes may exist in branched and/or linear form. If a branched form is commercially available, then the calibration standards must contain the branched and linear form. PFHxS and PFOS are currently available in mixes of branched and linear isomers. PFOA is available as a technical mix.**

Calibration standard concentrations are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 HPLC/MS/MS Conditions

7.2.1 HPLC Conditions

3-5ul autosampler injection                      Column temperature – 50.0 °C

Gradient Program

Time (min)	Water (0.1% acetic acid)	MeOH (0.1% acetic acid)	Flow ml/min
0-0.0	65%	35%	0.4
0-7.0	0%	100%	0.4
7.0-10.0	0%	100%	0.7
10.0-11.0	0%	100%	0.7
11.0-15.0	65%	35%	0.4

7.2.2 MS/MS Conditions

Parameter	Value	Parameter	Value
Gas Temp C	250	Sheath Gas Flow (l/min)	10
Gas Flow (l/min)	10	Capillary (V)	4500
Nebulizer (psi)	50	V Charging	600
Sheath Gas Heater	275	Ionization Mode	Neg ESI
Collision Cell Gas (psi)	40	Collision Cell Gas	UHP N2

Fragmentation voltages and collisions energies are optimized for each analyte and are stored in the instrument method. Precursor ions and transition masses are listed in TABLE 1.

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LC/MS/MS conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

### 7.3 Sample Preparation

#### 7.3.1 Low Level Aqueous Samples

A 250ml aliquot of sample (entire bottle) is extracted utilizing a solid phase extraction cartridge. The cartridge is eluted with basic methanol, concentrated and the final volume is adjusted to 1.0ml. Refer to SOP OP072.

### 7.4 HPLC/MS/MS Analysis

Instrument calibration consists of four major sections:

- Mass Tuning and Calibration
- Transition Window Selection
- Initial Calibration Procedures
- Continuing Calibration Verification

#### 7.4.1 Mass Tuning & Calibration and Transition Window Selection

The instrument must have a valid mass calibration prior to any sample analysis. The mass calibration must be updated as needed. (i.e. QC failures, ion masses showing large deviations from known masses, or after major instrument maintenance is performed). It is recommended that the mass calibration be verified weekly through the analysis of a Check Tune. The Agilent Check Tune Masses range from 112.99 to 2233.91 amu for MS1 and 69.00 to 2233.91 for MS2.

The Check Tune Report may show both Positive and Negative ESI Results. Only the Negative results need to be evaluated. The following masses must be within  $\pm 0.5$  amu of the true value.

MS1 (UNIT)	MS2 (UNIT)
	69.00
112.99	112.99
302.00	302.00
601.98	601.98
1033.99	1033.99

Since masses greater than 1033.99 amu are not used for this method, the 1633.95 and 2233.91 amu masses do not need to be within 0.5 amu of the true value.

The mid-point standard is also used to check the analyte retention times. These retention times are used to update the transition windows. The windows must be wide enough to ensure that the branched and linear isomer for PFHxS and PFOS are completely within the transition window. The branched isomer will elute just prior to the linear isomer. If they are partially cut off, adjust the retention time of

the linear isomer or the width of the transition window. Use a similar size window for the other analytes that do not have a branched standard. Later eluting peaks are broader and require a slightly wider transition window.

#### 7.4.2 Initial Calibration Procedures

Before samples can be run, the LC/MS/MS system must be calibrated.

##### 7.4.2.1 Isotope Dilution Standard (Extracted Internal Standard) Calibration

A minimum 5-point calibration curve is created for the Perfluorinated compounds and surrogates using an isotope dilution technique. SGS Orlando routinely performs a 7-point calibration to maximize the calibration range. See TABLE 2.

**The calibration standards for PFHxS and PFOS must consist of both branched and linear isomers. The branched isomer elutes just prior to the linear isomer. PFAS are currently being reported as the sum of the branched and linear isomers so both peaks must be integrated.**

Response factors (RF) for each analyte at each calibration level are determined as follows:

$$RF = (A_{\text{analyte}} \times C_{\text{ids}}) / (A_{\text{ids}} \times C_{\text{analyte}})$$

$A_{\text{analyte}}$	=	area of the analyte
$A_{\text{ids}}$	=	area of the isotope dilution standard
$C_{\text{analyte}}$	=	concentration of the analyte
$C_{\text{ids}}$	=	concentration of the isotope dilution standard.

Use the LC/MS/MS data system to generate a linear regression or quadratic calibration curve for each of the analytes. This curve **must be forced through zero** and may be unweighted or weighted as  $1/x$  or  $1/x^2$ . Forcing zero allows for a better estimate of the background levels of method analytes.

Regression type and weighting should be evaluated to provide the best fit over the entire calibration range while meeting the calibration requirements below and the ICV requirements in section 7.4.2.2.

The Injection Standards and Isotope Dilution Standards are added at a single concentration level to the calibration standards, calibrate each of these using an average response factor. Note: With the MassHunter software a "linear regression" with all values at the same levels is equivalent to an average response factor. The software will display an  $R^2$  curve fit of 0.000000 for those analytes.

The MassHunter software will recalculate the concentration the individual points and the graphics will plot the selected calibration curve and each standard point against that curve. Emphasis should be placed on the %D of the low and mid-point standards. Curve fit order of preference:

Linear Forced  
Quadratic Forced  
Quadratic Weighted Forced

The correlation coefficient (r) should be  $\geq 0.995$  ( $r^2 \geq 0.990$ ). Additionally, each calibration point for each analyte must be recalculated against the current initial calibration.

Linear Curve Fit  $y = ax + b$

y = response ratio      x = concentration ratio

a = linear term          b = constant term

Quadratic Curve Fit  $y = ax^2 + bx + c$

y = response ratio      x = concentration ratio

a = quadratic term      b = linear term      c = constant term

Additionally, each point must be refitted against the initial calibration. The recovery of each analyte must be within 70%-130%, except for the low calibration point which must be within 50%-150% of the expected value.

#### 7.4.2.2 Initial Calibration Verification (ICV) Second Source Standard (SSS)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard (also referred to as a Second Source Standard). The ICV must be prepared from a second source at a mid-range concentration.

**NOTE: Second source standards may consist of linear isomers only.**

**NOTE: Analyze the PFOA Technical Mix to identify the branched isomers. This is a qualitative standard only.**

The recovery of each analyte of interest must be within 70%-130% (%D  $\leq 30\%$ ). If the ICV does not meet these criteria, prepare and analyze a fresh ICV standard. If this ICV meets criteria, proceed with sample analysis.

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If the ICV still does not meet criteria, samples cannot be analyzed. Evaluate the data to determine if one of the sources appears to have concentrated or degraded. It may be advisable to open new ampoules. Make fresh calibration standards and ICV standards as needed and recalibrate the instrument.

#### 7.4.2.3 Retention Time Windows

Retention time windows must be established whenever a new column is installed in an instrument or whenever a major change has been made to an instrument.

The retention time of each analyte and isotope dilution standards must fall within 0.4 minutes of the predicted retention times from the daily calibration verification or from the midpoint standard of the ICAL (on days when an ICAL is performed).

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and isotope dilution standard from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Initial peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks (isotope dilution standards) are used to correct for run-to-run variations in retention times due to temperature, flow, or injector fluctuations. HPLC retention times tend to shift more than GC retention times.

#### 7.4.3 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for the PFAS analytes are prepared at various concentrations. The opening CCV must be prepared at the Reporting Limit in order to verify instrument sensitivity.

Additional CCVs must be analyzed after every 10 samples and at the end of each run to verify that the initial calibration is still valid. CCV must be rotated through low, mid, and high concentrations.

The recovery of each analyte in the low-level CCV must be within 50%-150% of the expected value ( $\%D \leq 50\%$ ).

The recovery of each analyte in the mid-level and high-level CCVs must be within 70%-130% of the expected value ( $\%D \leq 30\%$ ).

The recovery of each isotope dilution standard in all CCVs must be within 70%-130% of the expected value ( $\%D \leq 30\%$ ).

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If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated.

If the recovery is outside the control limits, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reported, i.e. The CCV failed high, the associated QC passed, and the samples were ND.

**NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed. If the sample cannot be reanalyzed, then the data must be qualified.**

**NOTE: Since this may be for Drinking Water Compliance some Regulatory Agencies may require notification prior to reporting any qualified data. Contact the Project Manager or Quality Assurance Office.**

#### 7.4.4 Sample Extract Analysis

7.4.4.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Initial Calibration Standards (or Initial CCV)  
QC Extracts  
Sample Extracts  
CCV Standards

7.4.4.2 Two microliters of injection standard solution is added to every 100ul of extract in the autosampler vial. Generally, 500ul of extract are transferred to the autosampler vial with a gas tight syringe.

7.4.4.3 Three to five microliters (same amount as standards) of extract is injected into the HPLC by the autosampler. The data system then records the resultant peak responses and retention times.

7.4.4.4 Tentative identification of an analyte occurs when the peak from the sample extract falls within the retention time window of the target compound.

7.4.4.5 Positive identification is confirmed by comparing the ion ratio in the sample to the ion ratio of the standards. For the linear isomer, the primary and secondary transition masses must both be present. For the branched isomer the primary and secondary transition masses must both be present. In rare circumstances a particular branched peak may only exhibit a single transition ion.

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The MassHunter software is set to flag the analyte if the ratio of these ions is not within  $\pm 50\%$  of the expected, (e.g., if the ion ratio is expected to be 40% in the standard, the ion ratio in the corresponding sample must be between 20 and 60%).

The signal to noise ratio for the primary transition mass must be at least 10 times that of the background and the secondary transition mass must be at least 3 times that of the background.

- 7.4.4.6 Some of the PFASs may have multiple chromatographic peaks due to the presence of linear and branched isomers. This is prevalent in PFHxS and PFOS. The areas of all the linear and branched isomers peaks present in the standards must be included in the samples and the concentrations reported as a total for each of these analytes.

**NOTE: For PFOA, the branched isomers must be included in the quantitation even if the calibration is based on just the linear isomer. The concentration is reported as the total of the peaks.**

**NOTE: For all others, the areas of any branched isomers that are present in samples but are not present in the calibration standards are not to be included and the concentrations reported as the linear isomer.**

- 7.4.4.7 If the compound identification does not confirm, then the result should be reported as ND or "U".

- 7.4.4.8 If an analyte result exceeds the range of the initial calibration curve, a second smaller aliquot of the sample must be extracted, if available. Dilute an aliquot of the sample with reagent water to 250ml. Add ammonium acetate to a final concentration of 1 g/L and process the diluted sample. Report all concentrations measured in the original sample that do not exceed the calibration range. Report concentrations of analytes that exceeded the calibration range in the original sample based on measurement in a diluted sample.

- 7.4.4.9 If there is no additional sample aliquot available for "dilution", then the result must be reported with an "E" or "L" qualifier.

It may be possible to perform an expanded calibration for select analytes in order to bring the original extract into calibration range. If the expanded ICAL meets method criteria, then reanalyze the MB, BS and any samples that were above the original calibration range.

It may be possible to perform a dilution on the original extract to verify the concentration. This procedure is only valid for dilutions in the 2-10-fold range. The extract is diluted with the 80:20 methanol:water mix. No additional isotope dilution standards are added. The theoretical concentration of the isotope dilution standards in the extract will need to

be entered into MassHunter so that the software can correctly calculate the native analyte concentration. **This dilution procedure is not acceptable for compliance drinking water samples.**

7.4.4.10 If peak identification is prevented by the presence of interferences, further cleanup may be required, or the extract must be diluted so that the interference does not mask any analytes.

## 7.5 Maintenance and Trouble Shooting

7.5.1 Refer to SOP GC001 for routine instrument maintenance and trouble shooting.

7.5.2 All instrument maintenance must be documented in the appropriate "Instrument Repair and Maintenance" log. The log will include such items as problem, action taken, correction verification, date, and analyst.

7.5.3 Repairs performed by outside vendors must also be documented in the log. The analyst or Department Supervisor responsible for the instrument must complete the log if the repair technician does not.

7.5.4 PC and software changes must be documented in the "Instrument Repair and Maintenance" log. Software changes may require additional validation.

## 8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), matrix spike duplicates (MSD) and sample duplicates (DUP). The MB and BS are used to monitor overall method performance, while the MS and MSD or DUP are used to evaluate the method performance and reproducibility in a specific sample matrix.

Blank spike, matrix spike and matrix spike duplicate recoveries are compared to method defined control limits. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

## 9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of isotope dilution standards and by the analysis of a QC set that is prepared and analyzed with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD) or sample duplicate (DUP). All control limits are updated annually and are listed in the LIMS.



9.1 Injection Standards (Isotope Performance Standards)

9.1.1 Perfluoro-[2,3,4-<sup>13</sup>C<sub>3</sub>] butanoic acid, Perfluoro-[1,2-<sup>13</sup>C<sub>2</sub>] octanoic acid (<sup>13</sup>C<sub>2</sub>-PFOA) and Perfluoro-1-[1,2,3,4-<sup>13</sup>C<sub>4</sub>] octane sulfonic acid (<sup>13</sup>C<sub>4</sub>-PFOS) are used as injection standards for this method.

The response of the Injection Standards in all subsequent runs must be 50-150% of the injection standard average response from the initial calibration.

9.1.2 If the injection standard responses are not within limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, integrations, or injection standard solutions. If errors are found, recalculate the data accordingly.

9.1.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.1.2.3 If no problem is found, prepare a second aliquot of extract and reanalyze the sample.

9.1.2.4 If upon reanalysis, the responses are still not within limits, reanalyze the sample at a dilution.

9.1.2.5 If upon analysis of the dilution the responses are within limits, then the sample or select analytes may need to be reported from the dilution or qualified.

9.1.2.6 The responses of the isotope dilution standards can be used to help assess the data too.

9.2 Isotope Dilution Standards

9.2.1 The analytes listed in section 5.7 are used as the isotope dilution standards for this method.

A known amount of isotope dilution standard is added to each sample including the QC set prior to extraction. The recovery for each isotope dilution standard must be 50% to 200%.

The % recovery may be calculated by direct comparison of the isotope dilutions standard responses to the response from the initial calibration midpoint standard or they may be calculated from the calculated concentrations.

$$\% \text{ Recovery} = (\text{Sample Amount} / \text{Amount Spiked}) \times 100$$

Only those isotope dilution standards that directly link to the native analytes being reported need to pass. For example, <sup>13</sup>C<sub>4</sub>-PFBA only needs to pass if PFBA is being reported.

- 9.2.2 If any isotope dilution standard response/recovery is not within the established control limits, the following are required.
- 9.2.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, isotope dilution standard solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.
  - 9.2.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.
  - 9.2.2.3 Check for instrument suppression or enhancement by reanalyzing the sample at a dilution.
  - 9.2.2.4 If no problem is found, re-extract and reanalyze the sample. If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.
  - 9.2.2.5 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Isotope dilution standards from both sets of analysis must be reported on the final report.

### 9.3 Method Blank

- 9.3.1 The method blank is either de-ionized or HPLC grade water to which the surrogate standard has been added. The method blank is then taken through all procedures along with the other samples to determine any contamination from reagents, glassware, or high-level samples. The method blank must be free of any analytes of interest or interferences at 1/3 the required reporting level to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Blank correction is not permitted under this method.
- 9.3.2 Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a "B" or "V" qualifier. This must be approved by the department supervisor.

**NOTE: Samples may be for Drinking Water Compliance; some Regulatory Agencies may require notification prior to reporting any qualified data. Contact the Project Manager or Quality Assurance Office.**

- 9.3.2.1 Hits in Method Blanks should be closely reviewed and confirmed if needed as positive detects greater than 1/3 the RL may be grounds for data rejection by some regulatory agencies. The laboratory must provide enough information for the regulatory agencies to make their decision.

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- 9.3.2.2 If the MB is contaminated but the samples are non-detect, the source of contamination must still be investigated and documented. The samples must be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results.
- 9.3.2.3 If the MB is contaminated but the samples results are <10 times the contamination level, the source of the contamination must be investigated and documented. The samples must be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers (“B” or “V”) must be added to the results.
- 9.3.2.4 If the MB is contaminated but the samples results are >10 times the contamination level, the samples may have been the source of the cross contamination. The department supervisor shall review the data and determine which samples should be re-extracted and reanalyzed for confirmation. It may be necessary prep the high-level samples separately to prevent further cross contamination. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers (“B” or “V”) must be added to the results.

#### 9.4 Blank Spike

- 9.4.1 The blank spike is either de-ionized or HPLC grade water to which the surrogate standard and spike standard have been added. The concentration of the blank spike must be varied from batch to batch. The blank spike is then taken through all procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

$$\% \text{ Recovery} = (\text{Blank Spike Amount} / \text{Amount Spiked}) \times 100$$

The percent recovery for each analyte of interest in the low-level blank spike must fall within 50-150% of the true value, and within 70-130% of true value for the mid-level and high-level blank spike for the results to be acceptable.

**NOTE: Since this may be for Drinking Water Compliance some Regulatory Agencies may require notification prior to reporting any qualified data. Contact the Project Manager or Quality Assurance Office.**

- 9.4.2 If the blank spike recoveries are not within the established control limits, the following are required.
  - 9.4.2.1 Recovery failures in the blank spike should be closely reviewed and confirmed if needed as any recovery failures (including high in BS and non-detect in samples) may be grounds for data rejection by some

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regulatory agencies. The laboratory must provide enough information for the regulatory agencies to make their decision.

- 9.4.2.2 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.
- 9.4.2.3 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.
- 9.4.2.4 If no problem is found, re-extract and reanalyze all samples associated with the batch.
- 9.4.2.5 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

## 9.5 Matrix Spike and Matrix Spike Duplicate

- 9.5.1 Matrix spike and spike duplicates are replicate sample aliquots to which the spike standard has been added. The concentration of the matrix spike and spike duplicate must be varied from batch to batch. The matrix spike and spike duplicate are then taken through all procedures along with the other samples to monitor the precision and accuracy of the procedure. The percent recovery for each analyte is calculated as follows:

$$\% \text{ Recovery} = [(\text{Spike Amount} - \text{Sample Amount}) / \text{Amount Spiked}] \times 100$$

The percent recovery for each analyte of interest in the low-level matrix spike and spike duplicate must fall within 50-150% of the true value, and within 70-130% of true value for the mid-level and high-level blank spike for the results to be acceptable.

- 9.5.2 If the matrix spike recoveries are not within the established control limits, the following are required.
  - 9.5.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.
  - 9.5.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.5.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extract but are indications of the sample matrix effects.

9.5.3 Precision

Matrix spike and spike duplicate recoveries for each analyte OR sample result and duplicate result are used to calculate the relative percent difference (RPD) for each compound.

$$\text{RPD} = [ | \text{MS Result} - \text{MSD Result} | / \text{Average Result} ] \times 100$$

$$\text{RPD} = [ | \text{Sample Result} - \text{DUP Result} | / \text{Average Result} ] \times 100$$

The RPD for each Perfluorinated compound must be less than 30%. If the RPDs fall outside of the established control limits, the MS and MSD must be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

9.6 Initial Demonstration of Capability

The IDC must be successfully performed prior to analyzing any Field Samples. Prior to conducting the IDC, the analyst must first generate an acceptable Initial Calibration following the procedure outlined in MS022 Section 7.4.2.

9.6.1 Calibration Confirmation – Analyze a Blank Spike (QCS) as described in MS022 Section 9.4.1 to confirm the accuracy of the standards/calibration curve.

9.6.2 Initial Demonstration of Branched vs Linear Isomer Profile for PFOA – Prepare and analyze a qualitative standard used for identifying retention times of branch isomers of PFOA. Identify the retention times of branched isomers of PFOA in the purchased technical grade PFOA standard. This qualitative PFOA standard is not used for quantitation (see MS022 Section 7.4.2.2). This branched isomer identification check must be repeated any time changes occur that affect the analyte retention times.

9.6.3 Initial Demonstration of Low System Background – Any time a new lot of SPE cartridges, solvents, centrifuge tubes, disposable pipets, and autosampler vials are used, it must be demonstrated that an LRB is reasonably free of contamination and that the criteria in MS022 Section 9.3 are met.

9.6.4 Initial Demonstration of Precision (IDP) – Prepare, extract, and analyze seven replicate Blank Spikes (LFBs) fortified near the midrange of the initial calibration curve according to the procedure described in OP072 Section 7.12. Sample preservatives as described in OP072 Section 3.1.3 must be added to these

samples. The relative standard deviation (RSD) of the results of the replicate analyses must be less than 20%.

- 9.6.5 Initial Demonstration of Accuracy (IDA) – Using the same set of replicate data generated above, calculate average recovery. The average recovery of the replicate values must be within  $\pm 30\%$  of the true value.
- 9.6.6 Minimum Reporting Level (MRL) Confirmation – Establish a target concentration for the MRL based on the intended use of the method. The MRL may be established by a laboratory for their specific purpose or may be set by a regulatory agency. Procedure for determining an MRL is listed in QA020 Section 6.0.
- 9.6.7 Detection Limit Determination – While DL determination is not a specific requirement of this method, it may be required by various regulatory bodies associated with compliance monitoring. It is the responsibility of the laboratory to determine if DL determination is required based upon the intended use of the data. Procedure for determining an MDL is listed in QA020 Section 4.2

## 10.0 CALCULATIONS

The concentration of each Perfluorinated compound in the original sample is calculated as follows:

$$\text{Water (ug/l)} = (\text{CONC}_{\text{inst}}) \times (V_F / V_I) \times \text{DF}$$

CONC <sub>inst</sub>	=	Instrument concentration calculated from the initial calibration using mean CF or curve fit
DF	=	Dilution Factor
V <sub>F</sub>	=	Volume of final extract (ml)
V <sub>I</sub>	=	Volume of sample extracted (ml)

## 11.0 SAFETY AND POLLUTION PREVENTION

### 11.1 Safety

The analyst should follow normal safety procedures as outlined in the SGS North America, Inc. Health and Safety Program and SGS Orlando SOP QA033 (Laboratory Safety Procedures), current revision. Safety glasses, a lab coat, and appropriate gloves should be worn when handling samples.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample must be treated as a potential health hazard. Safety Data Sheets (SDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment must be used by all analysts.

## 11.2 Pollution Prevention

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

Wastewater and methanol from the instrument are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

Sample Extracts are archived and stored for 30 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

## 12.0 REFERENCES

EPA Method 533 Revision 1.0, December 2019

**TABLE 1: Precursor and Primary Transition Masses**

Analyte	Type	Precursor Ion	Prod Ion Primary	Prod Ion Secondary
13C3-PFBA	INJ	216	172	
13C4-PFBA	IDS	217	172	
PFBA	Native	213	169	
PFMPA	Native	229	85	
13C5-PFPeA	IDS	268	223	
PFPeA	Native	263	219	
PFMBA	Native	279	85	
13C5-PFHxA	IDS	318	273	
PFHxA	Native	313	269	119
NFDHA	Native	295	201	85
13C4-PFHpA	IDS	367	322	
PFHpA	Native	363	319	169
ADONA	Native	377	251	85
13C2-PFOA	INJ	415	370	
13C8-PFOA	IDS	421	376	
PFOA	Native	413	369	169
13C9-PFNA	IDS	472	427	
PFNA	Native	463	419	219
13C6-PFDA	IDS	519	474	
PFDA	Native	513	469	219
13C7-PFUnDA	IDS	570	525	
PFUnDA	Native	563	519	269
13C2-PFDoDA	IDS	615	570	
PFDoDA	Native	613	569	319



**TABLE 1: Precursor and Primary Transition Masses**

Analyte	Type	Precursor Ion	Prod Ion Primary	Prod Ion Secondary
13C3-PFBS	IDS	302	99	
PFBS	Native	299	80	99
PFEESA	Native	315	135	69
13C3-PFHxS	IDS	402	99	
PFPeS	Native	349	80	99
PFHxS	Native	399	80	99
PFHpS	Native	449	80	99
13C4-PFOS	INJ	503	80	
13C8-PFOS	IDS	507	99	
PFOS	Native	499	80	99
9Cl-PF3ONS	Native	531	351	
11Cl-PF3OUdS	Native	631	451	
13C2-4:2FTS	IDS	329	309	
4:2FTS	Native	327	307	81
13C2-6:2FTS	IDS	429	409	
6:2FTS	Native	427	407	81
13C2-8:2FTS	IDS	529	509	
8:2FTS	Native	527	507	81
13C3-HFPO-DA	IDS	287	169	
HFPO-DA (GenX)	Native	329	285	169

**TABLE 2: Standard Levels**

COMPOUND	EPA 533 LEVELS IN PPB (Instrument Concentration) (x4 for ng/l)										SPIKE			SURR	
	ICAL										ICV1	ICV2	LOW		MID
Perfluorobutanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluoropentanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluorohexanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluoroheptanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluorooctanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0	20.0		1.0	20.0	40.0	
Perfluorononanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluorodecanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluoroundecanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluorododecanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluorobutanesulfonic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluoropentanesulfonic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluorohexanesulfonic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluoroheptanesulfonic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluorooctanesulfonic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
4:2 Fluorotelomer sulfonate	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
6:2 Fluorotelomer sulfonate	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
8:2 Fluorotelomer sulfonate	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluoro-3-methoxypropanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluoro-4-methoxybutanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Nonafluoro-3,6-dioxaheptanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Perfluoro(2-ethoxyethane)sulfonic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
Hexafluoropropylene oxide dimer acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
4,8-Dioxa-3H-perfluorononanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
11-Chloroeicosafuoro-3-oxaundecane-1-sulfonic acid	0.5	1.0	2.0	5.0	10.0	20.0	40.0	50.0	20.0			1.0	20.0	40.0	
MPFAC-24ES Isotope Dilutions STD	10.0	10.0	10.0	10.0	10.0	10.0		10.0	10.0	10.0					10.0
13C3-HFPO-DA	10.0	10.0	10.0	10.0	10.0	10.0		10.0	10.0	10.0					10.0
Perfluoro-[1,2,3-13C2]butanoic acid (13C3-PFBA)	10.0	10.0	10.0	10.0	10.0	10.0		10.0	10.0	10.0					
Perfluoro-[1,2-13C2]octanoic acid INJ STD	10.0	10.0	10.0	10.0	10.0	10.0		10.0	10.0	10.0					
Perfluoro-1-[1,2,3,4-13C4]octanesulfonic acid INJ STD	10.0	10.0	10.0	10.0	10.0	10.0		10.0	10.0	10.0					

Note: 0.5 ppb level must be included for any analyte being reported to 2 ng/l  
Note: 40ppb may be added to curve in place of the 10ppb

$$\text{Mass}_{\text{acid}} = \text{Mass}_{\text{salt}} \times \text{MW}_{\text{acid}} / \text{MW}_{\text{salt}}$$

MW<sub>acid</sub> = Molecular weight of PFAS  
MW<sub>salt</sub> = Molecular weight of the salt

**APPENDIX OF SIGNIFICANT CHANGES**

<b>Revision Date</b>	<b>Revision Number</b>	<b>Affected Section(s)</b>	<b>Revision Description</b>
06/2023	6	1.1.3	Added ranges of reporting limits
06/2023	6	6.2	Added instrument models
06/2023	6	7.4.2.1	Expanded procedure for Isotope Dilution Standard (Extracted Internal Standard) calibration
06/2023	6	7.4.4.5	Added MassHunter software flagging information for ion ratios
06/2023	6	9.3	Added "Blank correction is not permitted under this method."
06/2023	6	11.1	Updated Health and Safety section. Added SGS Orlando SOP QA033 (Laboratory Safety Procedures) reference
06/2023	6	11.2	Updated Pollution Prevention Section. Added SGS Orlando SOP SAM108 (Laboratory Waste Disposal) reference
06/2023	6	Appendix of Significant Changes	Added

# ANALYSIS OF PER- and POLYFLUORINATED ALKYL SUBSTANCES BY LC/MS/MS AND ISOTOPE DILUTION

## SOP Acknowledgement Form

I have read and understand this SOP. I will not knowingly deviate from this approved SOP without approval of the Department Supervisor, QA Officer, or Technical Director. If I notice any discrepancies between this SOP and the routine procedure, I will notify the Department Supervisor so that either the SOP or procedure can be changed. Furthermore, I understand that this SOP is property of SGS North America Inc. – Orlando and may not be printed nor duplicated in any manner.

Internal SOPs referenced within this SOP: OP072, GC001, QA029, QA033, SAM108, current revisions

Print Name	Signature	Date

Print the SOP Acknowledgement Form, sign, and submit to the SGS Orlando QA department.



## ANALYSIS OF PERFLUORINATED ALKYL ACIDS BY LC/MS/MS

Prepared by: Norm Farmer Date: 06/09/2023

Approved by: Natasha Gumtie Date: 06/12/2023

### Annual Review

Reviewed by: \_\_\_\_\_ Date: \_\_\_\_\_

Reviewed by: \_\_\_\_\_ Date: \_\_\_\_\_

Reviewed by: \_\_\_\_\_ Date: \_\_\_\_\_

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# TITLE: ANALYSIS OF PERFLUORINATED ALKYL ACIDS BY LC/MS/MS

## REFERENCES: EPA 537 and 537.1

### 1.0 SCOPE AND APPLICATION, SUMMARY

#### 1.1 Scope and Application

1.1.1 This method is used to determine the concentrations of select Perfluorinated Alkyl Acids (PFAA) in potable and finished drinking water utilizing an HPLC equipped with a tandem mass spectrometer (MS/MS).

1.1.2 The following compounds can be reported by this method:

TARGET	Synonym
Perfluorohexanoic acid	PFHxA
Perfluoroheptanoic acid	PFHpA
Perfluorooctanoic acid	PFOA
Perfluorononanoic acid	PFNA
Perfluorodecanoic acid	PFDA
Perfluoroundecanoic acid	PFUnDA
Perfluorododecanoic acid	PFDoDA
Perfluorotridecanoic acid	PFTTrDA
Perfluorotetradecanoic acid	PFTeDA
Perfluorobutanesulfonic acid	PFBS
Perfluorohexanesulfonic acid	PFHxS
Perfluorooctanesulfonic acid	PFOS
N-Methyl perfluorooctanesulfonamidoacetic acid	N-MeFOSAA
N-Ethyl perfluorooctanesulfonamidoacetic acid	N-EtFOSAA
Hexafluoropropylene oxide dimer acid (GenX)	HFPO-DA
4,8-dioxa-3H-perfluorononanoic acid	ADONA
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid	9Cl-PF3ONS
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS

Additional analytes can be found in TABLE 1.

- 1.1.3 The reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. Reporting limits may vary depending on matrix complications and volumes. Reporting limits for this method are in the range of 0.002-0.010 ug/l or 2.0 to 10 ng/l.
- 1.1.4 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.
- 1.1.5 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a "J" or "I" qualifier. Some program or project specifications may require that no values below the RL be reported.

## 1.2 Summary

- 1.2.1 This method is adapted from EPA 537 and 537.1 for the analysis of PFAA's in potable and finished drinking water.
- 1.2.2 Samples are received, stored, and extracted within the appropriate holding times.
- 1.2.3 Low level waters are extracted by SPE in accordance to SOP OP064.
- 1.2.4 Perfluorinated compounds are separated, detected, and quantitated using an LC/MS/MS. After HPLC separation and ionization, the specific Perfluorinated compound is isolated in the first mass spectrometer and transferred to a collision cell for fragmentation. The resulting fragments are introduced into the second mass spectrometer where they are detected and quantified.
- 1.2.5 Manual integrations are performed in accordance with SOP QA029.

## 2.0 PRESERVATION AND HOLDING TIME

### 2.1 Preservation

- 2.1.1 Samples shall be collected in 250mL polypropylene (PP) or polyethylene (HDPE) bottles. Caps must not have Teflon liners. Various studies have shown that HDPE is preferable to PP for this analysis.
- 2.1.2 5.0 g/l of Trizma<sup>®</sup> should be added to each sample as a buffering reagent and to remove free chlorine.
- 2.1.3 The samples must be chilled to  $\leq 10^{\circ}\text{C}$  from the time of collection until arrival at the laboratory. Samples must not exceed  $10^{\circ}\text{C}$  during the first 48 hours after

collection. The samples must be refrigerated at  $\leq 6^{\circ}\text{C}$  from the time of receipt until extraction.

2.1.4 The extracts must be stored at room temperature to minimize analyte adsorption to the container walls. All extracts must be vortexed just prior to transfer to the autosampler vials.

## 2.2 Holding Time

2.2.1 Aqueous samples must be extracted within 14 days of collection.

2.2.2 Extracts must be analyzed within 28 days of extraction.

## 3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences. Method interferences may be caused by contaminants in solvents, reagents, or glassware. The analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) or Teflon products, HPLC solvent lines, methanol, aluminum foil, SPE transfer lines, bottle caps, etc. All of these materials must be demonstrated to be free from interferences.

3.2 Contact with glass containers, pipettes, or syringes should be minimized since the Perfluorinated compounds can potentially adsorb to glass surfaces.

3.3 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 of the water. Humic and/or fulvic material can be co-extracted during SPE and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent. Total organic carbon (TOC) is a good indicator of the humic content of the sample.

3.4 The d5-EtFOSAA surrogate exhibits low recoveries when iron is present in the samples. Some regulatory agencies such as Michigan Drinking Water and Environmental Health Division will now except sample results if d5-EtFOSAA is the only surrogate that is failing.

3.5 SPE cartridges can be a source of interferences. The analysis of field and method blanks can provide important information regarding the presence or absence of such interferences. Brands and lots of SPE devices must be tested to ensure that contamination does not preclude analyte identification and quantitation.

3.6 The background from method analytes or other contaminants that interfere with the measurement of method analytes must be below 1/3 of the Reporting Limit.

3.7 Water and containers used for equipment blanks or field blanks must be tested prior to use. For smaller sampling events DI water will be provided in the same type of bottle used for sample collection. For larger sampling events, four-liter HDPE containers should be used. Containers should be filled with DI water and allowed to sit for several hours

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before testing. The DI water and container blanks must be free of any analytes of interest or interferences at 1/3 the required reporting level to be acceptable.

- 3.8 A field blank must be collected with each set of samples at each site per sampling event. Each field blank consists of 4 bottles. Two bottles are filled with DI water at the lab and the other two bottles are empty. If Trizma<sup>®</sup> is being used for the samples then the two empty bottles must also contain Trizma<sup>®</sup>. At the sampling site the sampler should open the two empty bottles and transfer the DI water from the full bottles into them. Cap the bottles, label as field blanks, and return them to the laboratory along with the samples for analysis.

## **4.0 DEFINITIONS**

- 4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.
- 4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS) or Quality Control Sample (QCS).
- 4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.
- 4.4 Field Blank (FB): 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, preservation, and all analytical procedures. The purpose of the FB is to determine if method analytes or other interferences are present in the field environment.
- 4.5 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still considered valid.
- 4.6 Internal Standards (ISTD): A compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples.
- 4.7 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point must be at a level equal to or below the reporting level.
- 4.8 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor must be used whenever possible. The ICV is

used to verify the validity of an Initial Calibration. This may also be called a QC check standard or Second Source standard.

- 4.9 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in each sample matrix.
- 4.10 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in each sample matrix.
- 4.11 Method Blank (MB): 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 processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.
- 4.12 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.
- 4.13 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

## 5.0 REAGENTS

- 5.1 Water – HPLC grade or equivalent
  - Water with 0.1% acetic acid: Add 1ml of acetic acid to 1.0 L of HPLC Water
- 5.2 Methanol – HPLC grade or equivalent
  - Methanol with 0.1% acetic acid: Add 1ml of acetic acid to 1.0 L of Methanol
- 5.3 Methanol:Water Solution – 96% methanol and 4% water volume:volume
- 5.4 Acetic Acid – HPLC grade or equivalent
- 5.5 Trizma® - A premixed blend of Tris [Tris(hydroxymethyl)aminomethane] and Tris HCL [Tris(hydroxymethyl)aminomethane hydrochloride] – Sigma cat# T-7193 or equivalent.
- 5.6 Technical PFOA standard – Qualitative for branched isomer retention time determination.
- 5.7 Perfluorinated Alkyl Acids stock standards – Traceable to Certificate of Analysis.
- 5.8 Mass labeled – Internal Standards

Perfluoro-n-[3,4,5-13C3]pentanoic acid (13C3-PFPeA)  
Perfluoro-[1,2-13C2]octanoic acid (13C2-PFOA)  
Perfluoro-1-[1,2,3,4-13C4]octanesulfonic acid (13C4-PFOS)  
N-methyl-d3-perfluoro-1-octanesulfonamidoacetic acid (d3-NMeFOSAA)

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5.9 Mass labeled – Surrogates

Perfluoro-n-[1,2-<sup>13</sup>C<sub>2</sub>]hexanoic acid (13C<sub>2</sub>-PFHxA)  
Perfluoro-n-[1,2-<sup>13</sup>C<sub>2</sub>]decanoic acid (13C<sub>2</sub>-PFDA)  
N-ethyl-d<sub>5</sub>-perfluoro-1-octanesulfonamidoacetic acid (d<sub>5</sub>-NEtFOSAA)  
Tetrafluoro-2-heptafluoropropoxy-<sup>13</sup>C<sub>3</sub>-propanoic acid (13C<sub>3</sub>-HFPO-DA)

## 6.0 APPARATUS

6.1 HPLC – Agilent Technologies 1260 or 1290

Suitable HPLC equipped with an autosampler, pump, and column compartment. System should have a membrane degasser.

6.2 MS/MS – Agilent Technologies 6460A, 6470x or 6495B

LC/MS/MS must be capable of negative ion electrospray ionization near the required flow rate of the HPLC Column. The system must be capable of performing MS/MS to produce unique precursor and product ions for the PFAA method analytes within the specified retention time segments. A minimum of 10 scans across each peak is required to ensure adequate precision.

6.3 Data System – Agilent Technologies MassHunter B.07.0x, B.08.0x, B10.0x.

6.3.1 A computer system interfaced to the HPLC/MS/MS that allows for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

6.3.2 The software must allow for the viewing of the specific MS/MS Spectra acquired over the analytical run. Comparisons can then be made between spectra from standards and samples.

6.3.3 Data is archived to a backup server for long term storage.

6.4 Columns: Agilent Poroshell 120 EC C18 2.7 $\mu$ m, 100 x 2.1 mm ID or equivalent

6.5 Disposable polyethylene transfer pipettes

6.6 15ml Centrifuge tubes - HDPE or PP

6.7 Polyethylene screw cap and autosampler vials

6.8 Volumetric Pipettors and volumetric “plasticware” for dilutions of standards and extracts.

6.9 4-liter LDPE or HDPE containers – For DI water for Equipment Blanks, shown to be PFAA free

6.10 250ml polyethylene or polypropylene bottles – shown to be PFAA free

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## 7.0 PROCEDURE

### 7.1 Standards Preparation

Standards are prepared from commercially available certified neat or reference standards. All standards must be logged in the HPLC Standards Logbook. All standards shall be traceable to their original source. The standards must be stored at  $\leq 6^{\circ}\text{C}$ , or as recommended by the manufacturer. All standards must be allowed to come to room temperature and vortexed prior to use. Calibration levels, spike and surrogate concentrations, preparation information, and vendor part numbers can be found in the MS STD Summary in the Active SOP directory. A summary of the calibration concentrations can be found in Table 3.

#### 7.1.1 Stock Standard Solutions

Stock standards are available from some commercial vendors. All vendors must supply a "Certificate of Analysis" with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor's expiration date. Once opened, the hold time is reduced to one year or the vendor's expiration date (whichever is shorter).

#### 7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with 96:4 methanol:water. The hold time for intermediate standards is six months or the vendor's expiration date (whichever is shorter). Intermediate standards may need to be remade if comparisons to other standards indicate analyte degradation or concentration changes. Intermediate standards should be stored in polyethylene vials.

#### 7.1.3 Calibration Standards

Calibration standards for Perfluorinated analytes are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. Calibration standards are prepared in 96:4 methanol:water. The low standard is at a concentration at or below the RL and the remaining standards defines the working range of the detector. Calibration standards should be stored in polyethylene vials.

**Perfluorinated analytes may exist in branched and linear form. If a branched form is commercially available, then the calibration standards must contain the branched and linear form. PFHxS, PFOS, MeFOSAA, and EtFOSAA are currently available in mixes of branched and linear isomers. PFOA is available as a technical mix.**

Calibration standard concentrations are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 HPLC/MS/MS Conditions

7.2.1 HPLC Conditions - Poroshell Column

3-5ul autosampler injection                      Column temperature – 50.0 °C

7.2.2 Gradient Table

Time (min)	Water (0.1% acetic acid)	MeOH (0.1% acetic acid)	Flow ml/min
0-0.0	65%	35%	0.4
0-7.0	0%	100%	0.4
7.0-10.0	0%	100%	0.7
10.0-11.0	0%	100%	0.7
11.0-15.0	65%	35%	0.4

7.2.3 MS/MS Conditions

Parameter	Value	Parameter	Value
Gas Temp C	250	Sheath Gas Flow (l/min)	10
Gas Flow (l/min)	10	Capillary (V)	4500
Nebulizer (psi)	50	V Charging	600
Sheath Gas Heater	275	Ionization Mode	Neg ESI
Collision Cell Gas (psi)	40	Collision Cell Gas	UHP N2

Fragmentation voltages and collisions energies are optimized for each analyte and are stored in the instrument method. Precursor ions and transition masses are listed in TABLE 2

LC/MS/MS conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3 Sample Preparation

A 250ml aliquot of sample (entire bottle) is extracted utilizing a solid phase extraction cartridge. The cartridge is eluted with methanol. The extract is concentrated to dryness, ISTD solution is added, and the final volume is adjusted to 1.0ml. Refer to SOP OP064.

7.4 HPLC/MS/MS Analysis

Instrument calibration consists of four major sections:

- Mass Tuning and Calibration
- Transition Window Selection
- Initial Calibration Procedures
- Continuing Calibration Verification

#### 7.4.1 Mass Calibration and Transition Window Selection

The instrument must have a valid mass calibration prior to any sample analysis. The mass calibration must be updated as needed. (i.e. QC failures, ion masses showing large deviations from known masses, or after major instrument maintenance is performed). It is recommended that the mass calibration be verified weekly through the analysis of a Check Tune. The Agilent Check Tune Masses range from 112.99 to 2233.91 amu for MS1 and 69.00 to 2233.91 for MS2.

The Check Tune Report may show both Positive and Negative ESI Results. Only the Negative results need to be evaluated. The following masses must be within  $\pm 0.5$  amu of the true value.

MS1 (UNIT)	MS2 (UNIT)
	69.00
112.99	112.99
302.00	302.00
601.98	601.98
1033.99	1033.99

Since masses greater than 1033.99 amu are not used for this method, the 1633.95 and 2233.91 amu masses do not need to be within 0.5 amu of the true value.

The mid-point standard is used to check the analyte retention times. These retention times are used to update the transition windows. The windows must be wide enough to ensure that the branched and linear isomer for PFOA, PFHxS, PFOS, MeFOSAA, and EtFOSAA are completely within the transition window. The branched isomers will elute just prior to the linear isomer. If they are partially cut off, adjust the retention time of the linear isomer or the width of the transition window. The branched isomers for MeFOSAA and EtFOSAA may be poorly resolved from the linear isomer (they may be evaluated in the ICV). Use a similar size window for the other analytes that do not have a branched standard. Later eluting peaks are broader and require a slightly wider transition window.

#### 7.4.2 Initial Calibration Procedures

Before samples can be run, the LC/MS/MS system must be calibrated.

##### 7.4.2.1 Internal Standard Calibration

A minimum 5-point calibration curve is created for the Perfluorinated compounds and surrogates using an internal standard technique. SGS Orlando routinely performs an 8-point calibration to maximize the calibration range. See TABLE 3

**The calibration standards for MeFOSAA, EtFOSAA, PFHxS and PFOS must consist of both branched and linear isomers. The branched isomer elutes just prior to the linear isomer. PFAAs are**

**currently being reported as the sum of the branched and linear isomers so both peaks must be integrated.**

Use the LC/MS/MS data system to generate a linear regression or quadratic calibration curve for each of the analytes. This curve **must be forced through zero** and may be concentration weighted. Forcing zero allows for a better estimate of the background levels of method analytes.

Regression type and weighting should be evaluated to provide the best fit over the entire calibration range while meeting the calibration requirements below and the ICV requirements in section 7.4.2.2.

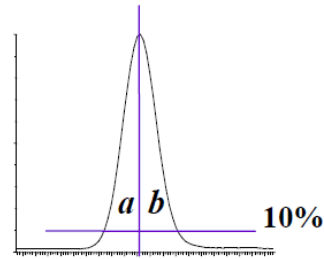
The MassHunter software will recalculate the concentration the individual points and the graphics will plot the selected calibration curve and each standard point against that curve. Emphasis should be placed on the %D of the low and mid-point standards. Curve fit order of preference:

Linear Forced  
Quadratic Forced  
Quadratic Weighted Forced

The correlation coefficient ( $r$ ) must be  $\geq 0.995$  ( $r^2 \geq 0.990$ ). Additionally, each calibration point for each analyte must be recalculated against the current initial calibration. The recovery of each analyte must be within 70%-130%, except for the low calibration point which must be within 50%-150% of the expected value.

The peak asymmetry factor must be calculated for the first two eluting peaks (that are being reported) in the mid-level calibration standard. The value must fall between 0.8 and 1.5. The MassHunter software is set to do this automatically. If the peak shape does not meet criteria the mobile phase may need to be adjusted to a higher aqueous content.

$$A_s = \frac{b}{a}$$



where:

$A_s$  = peak asymmetry factor

$B$  = width of the back half of the peak measured (at 10% peak height) from the trailing edge of the peak to a line dropped perpendicularly from the peak apex

$a$  = the width of the front half of the peak measured (at 10% peak height) from the leading edge of the peak to a line dropped perpendicularly from the apex.

The peak asymmetry factor must also be re-evaluated whenever changes are made to the system that may affect peak shape.

#### 7.4.2.2 Initial Calibration Verification (ICV) Second Source Standard (SSS)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard (also referred to as a Second Source Standard). The ICV must be prepared from a second source at a mid-range concentration.

**NOTE: Second source standards may consist of linear isomers only.**

**NOTE: Analyze the PFOA Technical Mix to identify the branched isomers. This is a qualitative standard only.**

The recovery of each analyte of interest must be within 70%-130% (%D  $\leq$  30%). If the ICV does not meet these criteria, prepare and analyze a fresh ICV standard. If this ICV meets criteria, proceed with sample analysis.

If the ICV still does not meet criteria, samples cannot be analyzed. Evaluate the data to determine if one of the sources appears to have concentrated or degraded. It may be advisable to open new ampoules. Make fresh calibration standards and ICV standards as needed and recalibrate the instrument.

#### 7.4.2.3 Retention Time Windows

The retention time of each analyte and internal standard must fall within 0.4 minutes of the predicted retention times from the daily calibration

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verification or from the midpoint standard of the ICAL (on days when an ICAL is performed).

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and internal standard from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Initial peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks (internal standards) are used to correct for run-to-run variations in retention times due to temperature, flow, or injector fluctuations. HPLC retention times tend to shift more than GC retention times.

#### 7.4.3 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for the PFAAs are prepared at various concentrations. The opening CCV must be prepared at the Reporting Limit in order to verify instrument sensitivity.

Additional CCVs must be analyzed after every 10 samples and at the end of each run to verify that the initial calibration is still valid. CCVs must be rotated through low, mid, and high concentrations.

The recovery of each analyte in the low-level CCV must be within 50%-150% of the expected value ( $\%D \leq 50\%$ ).

The recovery of each analyte in the mid-level and high-level CCVs must be within 70%-130% of the expected value ( $\%D \leq 30\%$ ).

Internal standard criteria is listed in section 9.1.1.

If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated.

If the recovery is outside the control limits, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reported, i.e. The CCV failed high, the associated QC passed, and the samples were ND.

**NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed. If the sample cannot be reanalyzed, then the data must be qualified.**

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**NOTE: Since this may be for Drinking Water Compliance some Regulatory Agencies may require notification prior to reporting any qualified data. Contact the Project Manager or Quality Assurance Office.**

The peak asymmetry factor must be re-evaluated whenever changes are made to the system that may affect peak shape. If the peak asymmetry factor needs to be re-evaluated, analyze a mid-level CCV after the initial low-level opening CCV.

See section 7.4.2.1 on how to evaluate the peak asymmetry factor.

#### 7.4.4 Sample Extract Analysis

7.4.4.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Initial Calibration Standards (or Initial CCV)  
QC Extracts  
Sample Extracts  
CCV Standards

7.4.4.2 Twenty microliters of internal standard solution are added to each extract in the centrifuge tube prior to the volume being adjusted to 1.0ml. Extract must be vortexed before transferring to the autosampler vial. Generally, 200-300ul of extract is transferred to the autosampler vial with a disposable pipette.

Autosampler vials should not be re-injected as the caps generally do not reseal after injection. If the available extract volume is getting low, the analyst may reseal the autosampler vial immediately after injection.

7.4.4.3 Three to five microliters (same amount as standards) of extract is injected into the HPLC by the autosampler. The data system then records the resultant peak responses and retention times.

7.4.4.4 Tentative identification of an analyte occurs when the peak from the sample extract fall within the retention time window of the target compound.

7.4.4.5 Positive identification is confirmed by comparing the mass spectra in the sample to the mass spectra of the standards.

7.4.4.6 Some of the PFAAs may have multiple chromatographic peaks due to the presence of linear and branched isomers. This is prevalent in PFOA, PFHxS and PFOS (and to some degree MeFOSAA and EtFOSAA). The areas of all the linear and branched isomers peaks must be included and the concentrations reported as a total for each of these analytes.

**NOTE: The branched isomers must be included in the quantitation even if the calibration is based on just the linear isomer.**

- 7.4.4.7 If the compound identification does not confirm, then the result should be reported as ND or “U”.
- 7.4.4.8 If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. Extracts should be diluted with 96:4 methanol:water solution. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.
- 7.4.4.9 Since the ISTD solution was added to the entire extract, the analyst will need to supplement the amount of ISTD in the diluted extract for proper quantitation.
- 7.4.4.10 If peak identification is prevented by the presence of interferences, further cleanup may be required, or the extract must be diluted so that the interference does not mask any analytes.

## 7.5 Maintenance and Trouble Shooting

- 7.6.1 Refer to SOP GC001 for routine instrument maintenance and trouble shooting.
- 7.6.2 All instrument maintenance must be documented in the appropriate “Instrument Repair and Maintenance” log. The log will include such items as problem, action taken, correction verification, date, and analyst.
- 7.6.3 Repairs performed by outside vendors must also be documented in the log. The analyst or Department Supervisor responsible for the instrument must complete the log if the repair technician does not.
- 7.6.4 PC and software changes must be documented in the “Instrument Repair and Maintenance” log. Software changes may require additional validation.

## 8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), matrix spike duplicates (MSD) and sample duplicates (DUP). The MB and BS are used to monitor overall method performance, while the MS and MSD or DUP are used to evaluate the method performance and reproducibility in a specific sample matrix.

Blank spike, matrix spike and matrix spike duplicate recoveries are compared to method defined control limits. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

## 9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), matrix spike duplicate (MSD) and/or sample duplicate (DUP). All control limits for this method are mandated by the method and stored in LIMS.

### 9.1 Internal Standards

9.1.1 Perfluoro-[1,2-<sup>13</sup>C<sub>2</sub>]octanoic acid (13C<sub>2</sub>-PFOA), Perfluoro-1-[1,2,3,4-<sup>13</sup>C<sub>4</sub>]octanesulfonic acid (13C<sub>4</sub>-PFOS) and N-methyl-d<sub>3</sub>-perfluoro-1-octanesulfonamidoacetic acid (d<sub>3</sub>-NMeFOSAA) are used as internal standards for this method. Additional analytes listed in Section 5.5 may be used for internal standards when reporting additional analytes.

The response of the internal standard in all subsequent runs must be 70-140% of the internal standard response in the most recent CCV and 50-150% of the internal standard average response from the initial calibration.

The target analytes associated with each internal standard are listed in TABLE 2.

9.1.2 If the internal standard responses are not within limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, integrations, or internal standards solutions. If errors are found, recalculate the data accordingly.

9.1.2.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.2.3 If no problem is found, prepare a second aliquot of extract and reanalyze the sample.

9.1.2.4 If upon reanalysis, the responses are still not within limits, the problem is considered matrix interference.

9.1.2.5 The sample may need to be reported from the dilution or the results qualified.

### 9.2 Surrogates

9.2.1 Perfluoro-n-[1,2-<sup>13</sup>C<sub>2</sub>]hexanoic acid (13C<sub>2</sub>-PFHxA) and Perfluoro-n-[1,2-<sup>13</sup>C<sub>2</sub>]decanoic acid (13C<sub>2</sub>-PFDA) are used as the surrogate standards to monitor the efficiency of the extraction. Additional analytes such as N-ethyl-d<sub>5</sub>-perfluoro-1-octanesulfonamidoacetic acid (d<sub>5</sub>-NEtFOSAA) and Tetrafluoro-2-

heptafluoropropoxy-13C3-propanoic acid (13C3-HFPO-DA) must be used for surrogate standards when reporting additional analytes.

A known amount of surrogate standard is added to each sample including the QC set prior to extraction. The percent recovery for each surrogate is calculated as follows:

$$\% \text{ Recovery} = (\text{Sample Amount} / \text{Amount Spiked}) \times 100$$

The percent recovery for each surrogate must fall within 70-130% of true value for the results to be acceptable.

- 9.2.2 If any surrogate recovery is not within the established control limits, the following are required.
- 9.2.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, surrogate solutions or internal standard solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.
  - 9.2.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.
  - 9.2.2.3 If no problem is found, re-extract and reanalyze the sample. **NOTE:** If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary, but the resulting data must be qualified accordingly. If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.
  - 9.2.2.4 If upon re-extraction and reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis must be reported on the final report to show the corrective action that was taken. For Drinking Water Compliance samples, it may be necessary to report both sets of results separately.

### 9.3 Method Blank

- 9.3.1 The method blank is either de-ionized or HPLC grade water to which the surrogate standard has been added. The method blank is then taken through all procedures along with the other samples to determine any contamination from reagents, glassware, or high-level samples. The method blank must be free of any analytes of interest or interferences at 1/3 the required reporting level to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Blank correction is not permitted under this method.

- 9.3.2 Samples associated with a contaminated method blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a “B” or “V” qualifier. This must be approved by the department supervisor.

**NOTE: Samples may be for Drinking Water Compliance; some Regulatory Agencies may require notification prior to reporting any qualified data. Contact the Project Manager or Quality Assurance Office.**

- 9.3.2.1 Hits in Method Blanks should be closely reviewed and confirmed if needed as positive detects greater than 1/3 the RL may be grounds for data rejection by some regulatory agencies. The laboratory must provide enough information for the regulatory agencies to make their decision.

- 9.3.2.2 If the MB is contaminated but the samples are non-detect, the source of contamination must still be investigated and documented. The samples must be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results.

- 9.3.2.3 If the MB is contaminated but the samples results are <10 times the contamination level, the source of the contamination must be investigated and documented. The samples must be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers (“B” or “V”) must be added to the results.

- 9.3.2.4 If the MB is contaminated but the samples results are >10 times the contamination level, the samples may have been the source of the cross contamination. The department supervisor shall review the data and determine which samples should be re-extracted and reanalyzed for confirmation. It may be necessary prep the high-level samples separately to prevent further cross contamination. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers (“B” or “V”) must be added to the results.

#### 9.4 Blank Spike

- 9.4.1 The blank spike is either de-ionized or HPLC grade water to which the surrogate standard and spike standard have been added. The concentration of the blank spike must be varied from batch to batch. The blank spike is then taken through all procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

$$\% \text{ Recovery} = (\text{Blank Spike Amount} / \text{Amount Spiked}) \times 100$$

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The percent recovery for each analyte of interest in the low-level blank spike must fall within 50-150% of the true value, and within 70-130% of true value for the mid-level and high-level blank spike for the results to be acceptable.

**NOTE: Since this may be for Drinking Water Compliance some Regulatory Agencies may require notification prior to reporting any qualified data. Contact the Project Manager or Quality Assurance Office.**

- 9.4.2 If the blank spike recoveries are not within the established control limits, the following are required.
  - 9.4.2.1 Recovery failures in the blank spike should be closely reviewed and confirmed if needed as any recovery failures (including high in BS and non-detect in samples) may be grounds for data rejection by some regulatory agencies. The laboratory must provide enough information for the regulatory agencies to make their decision.
  - 9.4.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the blank spike to verify.
  - 9.4.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the samples.
  - 9.4.2.3 If no problem is found, re-extract and reanalyze all samples associated with the batch.
  - 9.4.2.4 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers must be added to the results. This must be approved by the department supervisor.

## 9.5 Matrix Spike and Matrix Spike Duplicate

- 9.5.1 Matrix spike and spike duplicates are replicate sample aliquots to which the spike standard has been added. The concentration of the matrix spike and spike duplicate must be varied from batch to batch. The matrix spike and spike duplicate are then taken through all procedures along with the other samples to monitor the precision and accuracy of the procedure. The percent recovery for each analyte is calculated as follows:

$$\% \text{ Recovery} = [(\text{Spike Amount} - \text{Sample Amount}) / \text{Amount Spiked}] \times 100$$

The percent recovery for each analyte of interest in the low-level matrix spike and spike duplicate must fall within 50-150% of the true value, and within 70-130% of

true value for the mid-level and high-level blank spike for the results to be acceptable.

9.5.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.5.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.5.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the samples.

9.5.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extract but are indications of the sample matrix effects.

9.5.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

$$\text{RPD} = [ | \text{MS Result} - \text{MSD Result} | / \text{Average Result} ] \times 100$$

$$\text{RPD} = [ | \text{Sample Result} - \text{DUP Result} | / \text{Average Result} ] \times 100$$

The RPD for each Perfluorinated compound should be less than 30%. If the RPDs fall outside of the established control limits, the MS and MSD should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary. RPD failures are generally not grounds for re-extraction.

9.6 Field Blanks

9.6.1 The purpose of the FB is to determine if method analytes or other interferences are present in the field environment.

9.6.1.1 A field blank must be collected with each set of samples at each site per sampling event. Each field blank consists of 4 bottles. Two bottles are filled with DI water at the lab and the other two bottles are empty. If Trizma® is being used for the samples then the two empty bottles must also contain Trizma® from the same lot as the sample bottles. At the sampling site the sampler should open the two empty bottles and



transfer the DI water from the full bottles into them. Cap the bottles, label as field blanks, and return them to the laboratory along with the samples for analysis.

- 9.6.1.2 If it was noted that the Trizma® lots differed between the samples and Field Blank, note this in the analytical report.
- 9.6.2 Hits in Field Blanks should be closely reviewed and confirmed if needed as positive detects greater than 1/3 the RL may be grounds for data rejection by some regulatory agencies. The laboratory must provide enough information for the regulatory agencies to make their decision.
  - 9.6.2.1 Analytes detected in both the samples and an associated Field Blank must be footnoted in the results. [Wisconsin DNR requires that the resulting data be qualified with a B Flag.](#)
  - 9.6.2.2 If compounds are detected in the Field Blank but there are no detections of the compounds in the associated field samples, the results can be accepted with appropriate qualification. [Wisconsin DNR requires that the resulting data be qualified with a B Flag.](#)
  - 9.6.2.3 If the Field Blank is contaminated but the samples results are >10 times the contamination level, the samples may have been the source of the cross contamination. The results may still be accepted, but the appropriate footnote must be added to the results. [Wisconsin DNR requires that the resulting data be qualified with a B Flag.](#)

## 9.7 Initial Demonstration of Capability

The IDC must be successfully performed prior to analyzing any Field Samples. Prior to conducting the IDC, the analyst must first generate an acceptable Initial Calibration following the procedure outlined in MS017 Section 7.4.2.

- 9.7.1 Calibration Confirmation – Analyze a Blank Spike (QCS) as described in MS017 Section 9.4.1 to confirm the accuracy of the standards/calibration curve.
- 9.7.2 Initial Demonstration of Branched vs Linear Isomer Profile for PFOA – Prepare and analyze a qualitative standard used for identifying retention times of branch isomers of PFOA. Identify the retention times of branched isomers of PFOA in the purchased technical grade PFOA standard. This qualitative PFOA standard is not used for quantitation (see MS017 Section 7.4.2.2). This branched isomer identification check must be repeated any time changes occur that affect the analyte retention times.
- 9.7.3 Initial Demonstration of Low System Background – Any time a new lot of SPE cartridges, solvents, centrifuge tubes, disposable pipets, and autosampler vials are used, it must be demonstrated that an LRB is reasonably free of contamination and that the criteria in MS017 Section 9.3 are met.

- 9.7.4 Initial Demonstration of Precision (IDP) – Prepare, extract, and analyze four replicate Blank Spikes (LFBs) fortified near the midrange of the initial calibration curve according to the procedure described in OP064 Section 7.11. Sample preservatives as described in OP064 Section 3.1.3 must be added to these samples. The relative standard deviation (RSD) of the results of the replicate analyses must be less than 20%.
- 9.7.5 Initial Demonstration of Accuracy (IDA) – Using the same set of replicate data generated above, calculate average recovery. The average recovery of the replicate values must be within  $\pm 30\%$  of the true value.
- 9.7.6 Initial Demonstration of Peak Asymmetry Factor – Peak asymmetry factors must be calculated using the equation in MS017 Section 7.4.2.1 for the first two eluting peaks in the mid-level CAL standard. The peak asymmetry factors must fall in the range of 0.8 to 1.5.
- 9.7.7 Minimum Reporting Level (MRL) Confirmation – Establish a target concentration for the MRL based on the intended use of the method. The MRL may be established by a laboratory for their specific purpose or may be set by a regulatory agency. Procedure for determining an MRL is listed in QA020 Section 6.0.
- 9.7.8 Detection Limit Determination – While DL determination is not a specific requirement of this method, it may be required by various regulatory bodies associated with compliance monitoring. It is the responsibility of the laboratory to determine if DL determination is required based upon the intended use of the data. Procedure for determining an MDL is listed in QA020 Section 4.2.

## 10.0 CALCULATIONS

The concentration of each Perfluorinated compound in the original sample is calculated as follows:

$$\text{Water (ug/l)} = (\text{CONC}_{\text{inst}}) \times (V_F / V_I) \times \text{DF}$$

CONC <sub>inst</sub>	=	Instrument concentration calculated from the initial Calibration using mean CF or curve fit
DF	=	Dilution Factor
V <sub>F</sub>	=	Volume of final extract (ml)
V <sub>I</sub>	=	Volume of sample extracted (ml)

## **11.0 SAFETY AND POLLUTION PREVENTION**

### **11.1 Safety**

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

The analyst should follow normal safety procedures as outlined in the SGS North America, Inc. Health and Safety Program and SGS Orlando SOP QA033 (Laboratory Safety Procedures), current revision. Safety glasses, a lab coat, and appropriate gloves should be worn when handling samples.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample must be treated as a potential health hazard. Safety Data Sheets (SDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment must be used by all analysts.

### **11.2 Pollution Prevention**

Wastewater and methanol from the instrument are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

Sample Extracts are archived and stored for 30 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

## **12.0 REFERENCES**

EPA Method 537 Revision 1.1, September 2009

EPA Method 537.1 Revision 1.0, November 2018

EPA Technical Advisory: Laboratory Analysis of Drinking Water Samples for PFOA Using EPA Method 537 Rev. 1.1, EPA 815-B-16-021, September 2016

**TABLE 1: Analytes**

Perfluoropentanoic acid*	PFPeA	2706-90-3
<b>Perfluorohexanoic acid</b>	PFHxA	307-24-4
<b>Perfluoroheptanoic acid</b>	PFHpA	375-85-9
<b>Perfluorooctanoic acid</b>	PFOA	335-67-1
<b>Perfluorononanoic acid</b>	PFNA	375-95-1
<b>Perfluorodecanoic acid</b>	PFDA	335-76-2
<b>Perfluoroundecanoic acid</b>	PFUnDA	2058-94-8
<b>Perfluorododecanoic acid</b>	PFDoDA	307-55-1
<b>Perfluorotridecanoic acid</b>	PFTrDA	72629-94-8
<b>Perfluorotetradecanoic acid</b>	PFTeDA	376-06-7
<b>Perfluorobutanesulfonic acid</b>	PFBS	375-73-5
<b>Perfluorohexanesulfonic acid</b>	PFHxS	355-46-4
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
<b>Perfluorooctanesulfonic acid</b>	PFOS	1763-23-1
6:2 Fluorotelomer sulfonate	6:2FTS	27619-97-2
8:2 Fluorotelomer sulfonate	8:2FTS	39108-34-4
<b>N-Methyl perfluorooctanesulfonamidoacetic acid</b>	MeFOSAA	2355-31-9
<b>N-Ethyl perfluorooctanesulfonamidoacetic acid</b>	EtFOSAA	2991-50-6
<b>Hexafluoropropylene oxide dimer acid (GenX)</b>	HFPO-DA	13252-13-6
<b>4,8-dioxa-3H-perfluorononanoic acid</b>	ADONA	919005-14-4
<b>9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid</b>	9Cl-PF3ONS	756426-58-1
<b>11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid</b>	11Cl-PF3OUdS	763051-92-9

**Bolded** analytes are listed in the EPA method.

**NOTE:** \*PFPeA may recover outside of the 50-150% recovery limit for the low-level blank spike or the 70-130% recovery limit for the mid and high level blank spikes. This may not be acceptable to some regulatory agencies.

**TABLE 2: Precursor and Primary Transition Masses**

Compound	ISTD/SURR	Precursor Ion	Product Ion Primary	Product Ion Secondary	ISTD REF.
13C3-PFPeA	ISTD	266	222		
PFPeA		263	219		13C3-PFPeA
13C2-PFOA	ISTD	415	370		
13C2-PFHxA	SURR	315	270		13C2-PFOA
13C2-PFDA	SURR	515	470		13C2-PFOA
13C3-HFPO-DA	SURR	287	169		13C2-PFOA
HFPO-DA (GenX)		329	285	169	13C2-PFOA
PFHxA		313	269	119/169	13C2-PFOA
PFHpA		363	319	169	13C2-PFOA
PFOA		413	369	169	13C2-PFOA
PFNA		463	419	219	13C2-PFOA
PFDA		513	469	219	13C2-PFOA
ADONA		377	251	85	13C2-PFOA
9Cl-PF3ONS		531	351		13C2-PFOA
11Cl-PF3OUdS		631	451		13C2-PFOA
13C4-PFOS	ISTD	503	80		
PFBS		299	80	99	13C4-PFOS
PFHxS		399	80	99	13C4-PFOS
PFOS		499	80	99	13C4-PFOS
PFUnDA		563	519	269	13C4-PFOS
PFDODA		613	569	319	13C4-PFOS
PFTrDA		663	619	369	13C4-PFOS
PFTeDA		713	669	219	13C4-PFOS
d3-MeFOSAA	ISTD	573	419		
d5-EtFOSAA	SURR	589	419		d3-MeFOSAA
EtFOSAA		584	419	483	d3-MeFOSAA
MeFOSAA		570	419	512	d3-MeFOSAA
13C2-6:2FTS	ISTD	429	409		
6:2FTS		427	407	81	13C2-6:2FTS
8:2FTS		527	507	81	13C2-6:2FTS

**TABLE 3: Standard Levels**

COMPOUND	EPA 537 LEVELS IN PPB (Instrument Concentration)										SPIKE			
	ICAL										ICV1	LOW	MID	HIGH
Perfluoropentanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
Perfluorohexanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
Perfluoroheptanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
Perfluorooctanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
Perfluorononanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
Perfluorodecanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
Perfluoroundecanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
Perfluorododecanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
Perfluorotridecanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
Perfluorotetradecanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
Perfluorobutanesulfonic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
Perfluorohexanesulfonic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
Perfluorooctanesulfonic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
N-MeFOSAA	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
N-EtFOSAA	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
6:2 Fluorotelomer sulfonate	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
8:2 Fluorotelomer sulfonate	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
Hexafluoropropylene oxide dimer acid (GenX)	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
4,8-dioxo-3H-perfluorononanoic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
11-chloroeicosafuoro-3-oxaundecane-1-sulfonic acid	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0	20.0	1.0	20.0	80.0	
Perfluoro-n-[1,2-13C2]hexanoic acid SURR	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0		20.0	20.0	20.0	20.0
Perfluoro-n-[1,2-13C2]decanoic acid SURR	0.5	1.0	2.0	5.0	10.0	20.0	50.0	80.0	100.0		20.0	20.0	20.0	20.0
d5-EtFOSAA SURR	1.0	2.0	4.0	10.0	20.0	40.0	100.0	160.0	200.0		40.0	40.0	40.0	40.0
13C3-HFPO-DA SURR	1.0	2.0	4.0	10.0	20.0	40.0	100.0	160.0	200.0		40.0	40.0	40.0	40.0
Perfluoro-n-[3,4,5-13C3]pentanoic acid ISTD	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0				
Perfluoro-[1,2-13C2]octanoic acid ISTD	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0				
Perfluoro-1-[1,2,3,4-13C4]octanesulfonic acid ISTD	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0				
d3-MeFOSAA ISTD	40.0	40.0	40.0	40.0	40.0	40.0	40.0	40.0	40.0	40.0				
6:2 (13C2)-Fluorotelomer sulfonate ISTD	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20.0				

Note: 0.5 ppb level must be included for any analyte being reported to 2 ng/l  
 Note: 80ppb may be added to curve in place of the 10ppb  
 Note: All standards, spikes, surrogate solutions are to be prepared in 96:4 MeOH:H2O

$$\text{Mass}_{\text{acid}} = \text{Mass}_{\text{salt}} \times \text{MW}_{\text{acid}} / \text{MW}_{\text{salt}}$$

MW<sub>acid</sub> = Molecular weight of PFAA  
 MW<sub>salt</sub> = Molecular weight of the salt

**APPENDIX OF SIGNIFICANT CHANGES**

<b>Revision Date</b>	<b>Revision Number</b>	<b>Affected Section(s)</b>	<b>Revision Description</b>
06/2023	12	9.3.1	Added "Blank correction is not permitted under this method."
06/2023	12	Table 1 Analytes	Added "Note: *PFPeA may recover outside of the 50-150% recovery limit for the low-level blank spike or the 70-130% recovery limit for the mid and high level blank spikes. This may not be acceptable to some regulatory agencies."
06/2023	12	Table 2 Precursor and Primary Transition Masses	Removed Perfluorobutanoic acid and Perfluoroheptanesulfonic acid
06/2023	12	Table 3 Standard Levels	Removed Perfluorobutanoic acid and Perfluoroheptanesulfonic acid
06/2023	12	11.1	Updated Safety section. Added SGS Orlando SOP QA033 (Laboratory Safety Procedures) reference
06/2023	12	11.2	Updated Pollution Prevention Section. Added SGS Orlando SOP SAM108 (Laboratory Waste Disposal) reference
06/2023	12	Appendix of Significant Changes	Added

# ANALYSIS OF PERFLUORINATED ALKYL ACIDS BY LC/MS/MS

## SOP Acknowledgement Form

I have read and understand this SOP. I will not knowingly deviate from this approved SOP without approval of the Department Supervisor, QA Officer, or Technical Director. If I notice any discrepancies between this SOP and the routine procedure, I will notify the Department Supervisor so that either the SOP or procedure can be changed. Furthermore, I understand that this SOP is property of SGS North America Inc. – Orlando and may not be printed nor duplicated in any manner.

Internal SOPs referenced within this SOP: OP064, GC001, QA029, QA033, SAM108, current revisions

Print Name	Signature	Date

Print the SOP Acknowledgement Form, sign, and submit to the SGS Orlando QA department.