SAP Worksheet #1 - Title and Approval Page

FINAL

SAMPLING AND ANALYSIS PLAN FOR

2017 VAPOR INTRUSION STUDY AT KEYPORT OU 2, AREA 8, NAVAL BASE KITSAP KEYPORT, WASHINGTON

November 2017

Prepared for:

Department of the Navy
Naval Facilities Engineering Command Northwest
1101 Tautog Circle
Silverdale, Washington

Prepared by:

Battelle Memorial Institute
505 King Avenue
Columbus, Ohio 43201

and

Trihydro Corporation
1252 Commerce Drive
Laramie, WY 82070

Prepared under:

Contract No.: N44255-14-D-9013
Task Order (TO): 0026

Review Signature: Betsy Branch 2017.11.02 15:05:19 -04'00'

Elizabeth Branch
Battelle QA Officer

Date

Approval Signature:

Teresie Walker
NAVFAC Atlantic QA Officer

Date

SAP Worksheet #1 - Title and Approval Page

DRAFT

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Contract No.: N44255-14-D-9013
Task Order (TO): 0026

Digitally signed by Betsy Cutie
Date: 2017.09.06 13:17:10 -04'00'

Review Signature: _____ _____

Elizabeth Branch — Date
Battelle QA Officer

Approval Signature: WALKER.TERESIE.R.1515870071 Digitally signed by WALKER.TERESIE.R.1515870071 DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=WALKER.TERESIE.R.1515870071 Date: 2017.09.06 16:04:40 -04'00' _____ _____

Teresie Walker — Date
NAVFAC Atlantic QA Officer

EXECUTIVE SUMMARY

This Sampling and Analysis Plan (SAP) for 2017 Keyport Operable Unit (OU) 2, Area 8 Vapor Intrusion (VI) Study, Naval Base Kitsap (NBK) Keyport, Washington has been prepared to support collection and analyses of soil vapor samples in support of VI and risk evaluations adjacent to OU 2, Area 8 at NBK Keyport in Keyport, Washington. This SAP has been prepared by Battelle Memorial Institute (Battelle), and Trihydro Corporation (Trihydro) for the Department of the Navy (DON), Naval Facilities Engineering Command (NAVFAC) Northwest in accordance with Task Order Number 0026, which was issued under contract N44255-14-D-9013.

NBK Keyport OU 2 Area 8 comprises approximately 1 acre on the east side of NBK Keyport (Figure 1), around the location of the former plating shop (Building 72). Building 72 was demolished in 1999 as part of the Area 8 remediation and was replaced by an asphalt-paved parking area. The site is located in a heavily industrialized portion of the facility bordered by Port Orchard Bay to the south and east (Figure 1). The area is predominantly flat and almost entirely paved or covered by buildings. Past releases at Area 8 included the accidental release of chrome plating solutions onto the ground surface, discharge of plating wastes into a utility trench, and the release of plating solutions through cracks in the plating shop floor, waste disposal pipes, and sumps. These chrome plating solutions and plating wastes contained volatile organic compounds (VOCs) and metals. In addition, petroleum hydrocarbons (diesel and heavy oil) have been released to the environment from underground storage tanks and underground concrete vaults located within Area 8. The OU 2 Record of Decision (ROD) was signed in 1994, and selected VOCs and metals were identified as the chemicals of concern (COCs) associated with Area 8. However, VI studies have not been performed in this area, but are warranted based on the presence of trichloroethene (TCE) concentrations in groundwater at or above 5 parts per billion (ppb) within 100 feet of existing buildings (EPA 2015).

This SAP presents the planned field and analytical activities associated with the VI study. All field and laboratory personnel engaged in sampling and analysis during the course of the project will review this SAP and have a copy available during sampling and analysis activities. This SAP includes project quality objectives (PQOs), field sampling procedures, quality assurance/quality control (QA/QC) requirements, and data gathering methods that will be used during the project. This SAP is prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP; U.S. Department of Defense [DoD], 2005).

This SAP addresses the data collection activities and laboratory analyses associated with the VI study.

SAP WORKSHEETS

LIST OF FIGURES

LIST OF ATTACHMENTS

ABBREVIATIONS AND ACRONYMS

APP	Accident Prevention Plan
Battelle	Battelle Memorial Institute
BFB	bromofluorobenzene
bgs	below ground surface
CA	corrective action
CAS	Chemical Abstract Service
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COC	contaminant of concern
CSM	conceptual site model
DL	detection limit
DoD	Department of Defense, United States
DON	Department of the Navy, United States
DQI	data quality indicator
Ecology	Washington State Department of Ecology
EIM	Environmental Information Management
ELAP	Environmental Laboratory Accreditation Program
EPA	Environmental Protection Agency, United States
EXWC	Expeditionary Warfare Center, NAVFAC
GC	gas chromatograph
GC/MS	gas chromatograph/mass spectrometer
GPS	global positioning system
ICAL	initial calibration curve
ICV	initial calibration verification
ID	identification
IS	internal standard
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LDC	Laboratory Data Consultants, Inc.
LOD	limit of detection
LOQ	limit of quantitation
LTM	long-term monitoring

µg/m^3	microgram per cubic meter
MS	matrix spike
MSD	matrix spike duplicate
MTCA	Model Toxics Control Act
N/A	not applicable or not available
NAVFAC	Naval Facilities Engineering Command
NBK	Naval Base Kitsap
NEDD	Naval electronic data deliverable
NELAP	National Environmental Laboratory Accreditation Program
NIRIS	Naval Installation Restoration Information Solution
NUWC	Naval Undersea Warfare Center
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
PAL	project action limit
PARCCS	precision, accuracy, representativeness, completeness, comparability, and sensitivity
PCE	tetrachloroethene
PID	photoionization detector
PQO	project quality objective
QA	quality assurance
QAO	quality assurance officer
QAPP	quality assurance project plan
QC	quality control
QL	quantitation limit
QSM	Quality Systems Manual
ROD	Record of Decision
RPD	relative percent difference
RPM	remedial project manager
RSD	relative standard deviation
RT	retention time
SAP	sampling and analysis plan
SOP	standard operating procedure
SSHO	site safety and health officer
SVOC	semivolatile organic compound

TCD	Thermal Conductivity Detection
TCE	trichloroethene
UFP	uniform federal policy
USACE	United States Army Corps of Engineers
VI	vapor intrusion
VOC	volatile organic compound

SAP Worksheet #2 - SAP Identifying Information

Site Name/Number: NBK Keyport Area 8
Operable Unit (s): 2
Contractor Name: Battelle, with subcontractor Trihydro
Contract Number: N44255-14-D-9013, Task Order 0026
Contract Title: NAVFAC Northwest A&E Environmental Projects IDIQ

1. This SAP conforms to the *Uniform Federal Policy for Quality Assurance Plans* (DoD, 2005), and Environmental Protection Agency (EPA) *Guidance for Quality Assurance Project* Plans, EPA QA/G-5 (EPA, 2002).
2. Regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)
3. This SAP is project specific.
4. List dates of scoping sessions that were held:

Scoping Session	Date
Navy-Stakeholder Meeting	22 February 2017

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation

   N/A

6. List organizational partners (stakeholders) and connection with lead organization:

Organization Partners / Stakeholders	Role
The Suquamish Tribe	Stakeholder
EPA Region 10	Regulator
Washington State Department of Ecology (Ecology)	Lead Regulator

7. Lead organization: Naval Facilities Engineering Command (NAVFAC) Northwest
8. If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

   No special personnel training is required for this project.

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
A. Project Management		
Documentation		
1	Title and Approval Page	Page 1
2	Table of Contents SAP Identifying Information	Page 8

SAP Worksheet #2 - SAP Identifying Information (Continued)

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
3	Distribution List	Page 11
4	Project Personnel Sign-Off Sheet	Page 12
Project Organization		
5	Project Organizational Chart	Page 13
6	Communication Pathways	Page 14
7	Personnel Responsibilities and Qualifications Table	Page 16
8	Special Personnel Training Requirements Table	Page 18
Project Planning/ Problem Definition		
9	Project Planning Session Documentation (including Data Needs tables) Project Scoping Session Participants Sheet	Page 19
10	Problem Definition, Site History, and Background. Site Maps (historical and present)	Page 21
11	Site-Specific Project Quality Objectives	Page 23
12	Measurement Performance Criteria Table	Page 26
13	Sources of Secondary Data and Information Secondary Data Criteria and Limitations Table	Page 27
14	Summary of Project Tasks	Page 28
15	Reference Limits and Evaluation Table	Page 30
16	Project Schedule/Timeline Table	Page 32
B. Measurement Data Acquisition		
Sampling Tasks		
17	Sampling Design and Rationale	Page 33
18	Sampling Locations and Methods/ SOP Requirements Table Sample Location Map(s)	Page 37
19	Analytical Methods/SOP Requirements Table	Page 38
20	Field Quality Control Sample Summary Table	Page 39
21	Project Sampling SOP References Table Sampling SOPs	Page 40
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	Page 42
Analytical Tasks		
23	Analytical SOPs Analytical SOP References Table	Page 44
24	Analytical Instrument Calibration Table	Page 45

SAP Worksheet #2 - SAP Identifying Information (Continued)

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	Page 50
Sample Collection		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal Sample Handling Flow Diagram	Page 53
27	Sample Custody Requirements, Procedures/SOPs Sample Container Identification Example Chain-of-Custody Form and Seal	Page 54
Quality Control Samples		
28	QC Samples Table Screening/Confirmatory Analysis Decision Tree	Page 57
Data Management Tasks		
29	Project Documents and Records Table	Page 59
30	Analytical Services Table Analytical and Data Management SOPs	Page 60
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	Page 61
32	Assessment Findings and Corrective Action Responses Table	Page 62
33	QA Management Reports Table	Page 63
D. Data Review		
34	Verification (Step I) Process Table	Page 64
35	Validation (Steps IIa and IIb) Process Table	Page 66
36	Validation (Steps IIa and IIb) Summary Table	Page 68
37	Usability Assessment	Page 69

QA quality assurance
QAPP quality assurance project plan
QC quality control
SOP standard operating procedure
UFP uniform federal policy

SAP Worksheet #3 - Distribution List

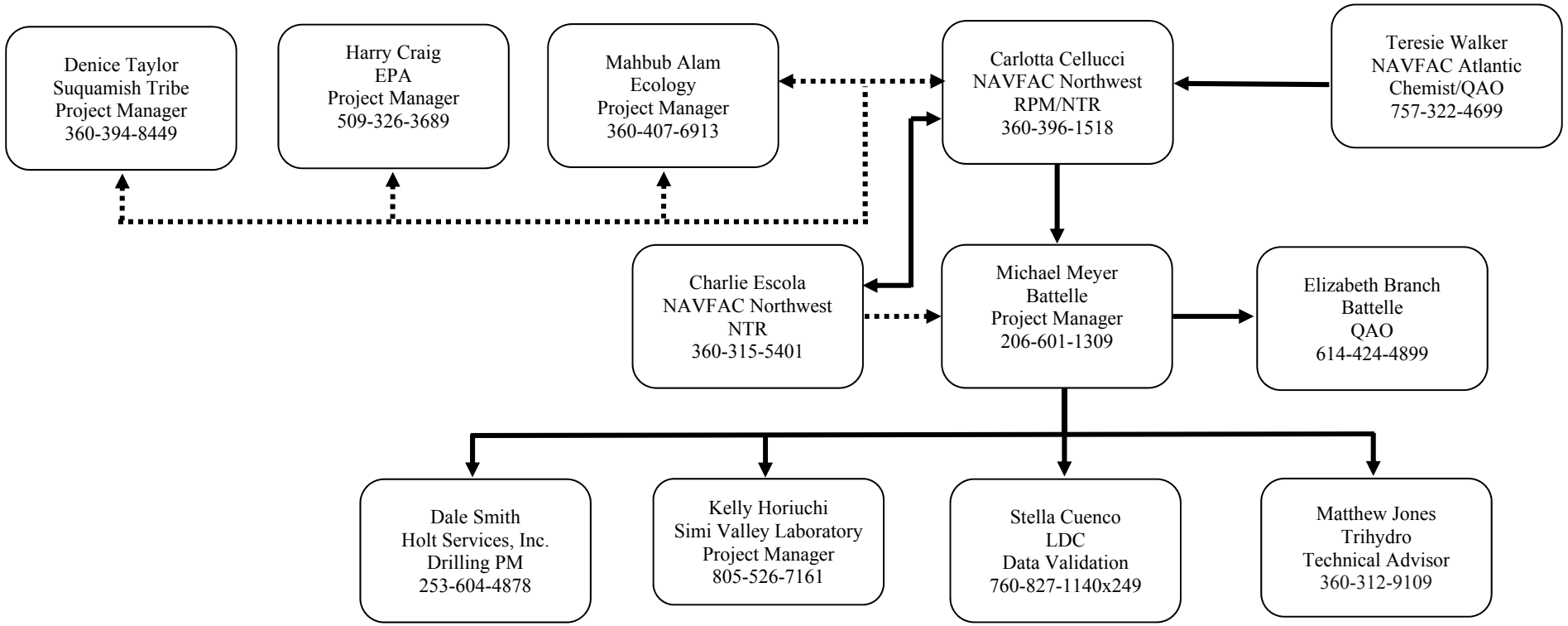
Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address
Carlotta Cellucci	Remedial Project Manager (RPM)/Navy Technical Representative	NAVFAC Northwest	360-396-1518	carlotta.cellucci@navy.mil
Charlie Escola	Navy Technical Representative	NAVFAC Northwest	360-315-5401	charles.escola@navy.mil
Teresie Walker	Navy Quality Assurance Officer (QAO)	NAVFAC Atlantic	757-322-4699	teresie.walker@navy.mil
Harry Craig	Project Manager	EPA	509-326-3689	Craig.harry@epamail.epa.gov
Denice Taylor	Project Manager	Suquamish Tribe	360-394-8449	dtaylor@suquamish.nsn.us
Mahbub Alam	Project Manager	Ecology	360-407-6913	mala461@ecy.wa.gov
Michael Meyer	Project Manager/Field Lead	Battelle	206-601-1309	meyerm@battelle.org
Elizabeth Branch	Project QAO	Battelle	614-424-4899	branche@battelle.org
Matthew Jones	Technical Advisor	Trihydro Corporation	360-312-9109	mjones@trihydro.com
Kelly Horiuchi	Project Manager, Laboratory Director	ALS Simi Valley Laboratory	805-526-7161	kelly.horiuchi@alsglobal.com
Dale Smith	Project Manager	Holt Services, Inc.	253-604-4878	dsmith@holtservicesinc.com
Stella Cuenco	Project Manager	Laboratory Data Consultants (LDC), Inc.	760-827-1140 x249	scuenco@lab-data.com

SAP Worksheet #4 - Project Personnel Sign-Off Sheet

Project Personnel	Title/Organization	Telephone Number	Signature	Date
Michael Meyer	Project Manager/Field Lead, Battelle	206-601-1309		
Matthew Jones	Technical Advisor/Trihydro Corporation	360-312-9109		
Dale Smith	Project Manager/Holt Services, Inc.	253-604-4878		
Kelly Horiuchi	Project Manager/ALS Simi Valley Laboratory	805-526-7161		
Stella Cuenco	Project Manager/LDC	760-827-1140 x249		

SAP Worksheet #5 - Project Organizational Chart

Lines of Authority ——— ····· Lines of Communication



SAP Worksheet #6 - Communication Pathways

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Point of contact for Department of Navy (DON) quality issues	NAVFAC Atlantic QAO	Teresie Walker	757-322-4699	NAVFAC QAO will review and approve this SAP and any amendments to the original SAP.
Project management	Battelle Project Manager	Michael Meyer	206-601-1309	Project Manager will manage the contract, deliverables, and field and project personnel.
QA/QC management	Battelle QAO	Elizabeth Cutié	614-424-4899	Battelle QAO will establish and maintain the project QC program, perform laboratory QC audits, oversee chemical data validation, and will resolve quality issues for the project. QAO will be the point of contact with the NAVFAC Northwest QAO for quality-related matters.
Coordination and communication of fieldwork activities related to sampling	Battelle Field Lead	Michael Meyer	206-601-1309	Field Lead will communicate relevant field information to the Project Manager and Battelle QAO.
Coordination of laboratory for field activities	Battelle Field Lead	Michael Meyer	206-601-1309	Field Lead will contact the laboratory to provide all necessary sample containers and appropriate shipping materials (such as coolers and bubble wrap) to be delivered on-site before field sampling begins and throughout the project.
Coordination of data validation	Battelle QAO	Elizabeth Cutié	614-424-4899	Battelle QAO will contact the laboratory to ensure that data reports are delivered to the data validator.
Submittal of samples to the laboratory	Trihydro Sampling Personnel	Matthew Jones	360-312-9109	Sampling personnel will package and ship samples in accordance with this SAP.
Daily chain of custody reports and shipping documentation	Trihydro Sampling Personnel	Matthew Jones	360-312-9109	Chain of custody forms and shipping documentation will be submitted via fax or e-mail to the Project Manager at the end of each day that samples are collected.
Reporting laboratory data quality issues	ALS Simi Valley Project Manager	Kelly Horiuchi	805-526-7161	All QA/QC issues will be reported by the Laboratory Project Manager to the Battelle QAO in writing within 2 business days.

SAP Worksheet #6 - Communication Pathways (Continued)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Field and analytical corrective actions	Battelle QAO	Elizabeth Cutié	614-424-4899	The Battelle QAO will immediately notify the Project Manager of any field or analytical procedures that were not performed in accordance with this SAP. The Battelle QAO, will complete documentation of the non-conformance and corrective actions to be taken. The Battelle QAO will verify that the corrective actions have been implemented.
Notification of non-usable analytical data	Battelle QAO	Elizabeth Cutié	614-424-4899	If significant problems are identified by the laboratory or the project team that impact the usability of the data (i.e., the data are rejected or the data quality objectives are not met), the Battelle QAO will notify the Project Manager, who will notify the NAVFAC Northwest RPM and the NAVFAC QAO within 24 hours or the next business day.
SAP procedure revision during field activities	Battelle Field Lead	Michael Meyer	206-601-1309	The Field Lead will prepare a field change request for any changes in sampling procedures that occur as a result of conditions in the field. This request will be submitted to the Project Manager for approval prior to initiation of the change.
SAP	Battelle QAO	Elizabeth Cutié	614-424-4899	Any changes to the SAP will require the Battelle QAO to prepare an addendum that will be approved by the NAVFAC QAO before any field activities begin.
Stop work	NAVFAC Atlantic QAO	Teresie Walker	757-322-4699	If an issue warranting a stop work order is identified, the NAVFAC QAO will immediately call the Battelle QC Manager to order the cessation of all project work.
Stop work	NAVFAC Northwest RPM	Carlotta Cellucci	360-396-1518	If an issue warranting a stop work order is identified, the RPM will immediately call the Battelle Project Manager to order the cessation of all project work.
Stop Work	Battelle QAO	Elizabeth Cutié	614-424-4899	If an issue warranting a stop work order is identified, the Battelle QAO will immediately call the NAVFAC QAO and Battelle Project Manager to order the cessation of all project work.
Stop Work	Trihydro Site Safety and Health Officer (SSHO)	Matthew Jones	360-312-9109	If an issue warranting a stop work order is identified, the Battelle SSHO will immediately call the Battelle Project Manager to order the cessation of all project work.

SAP Worksheet #7 - Personnel Responsibilities Table

Name	Title/Role	Organizational Affiliation	Responsibilities
Carlotta Cellucci	RPM	NAVFAC Northwest	Performing project management for DON. Ensuring that the project scope of work requirements are fulfilled. Overseeing the project cost and schedule. Providing formal technical direction to the project team, as needed. Acting as lead interface with agencies.
Teresie Walker	QAO	NAVFAC Atlantic	Reviewing and approving the SAP. Providing DON oversight of the QA Program. Providing technical and administrative oversight of the surveillance audit activities. Acting as a point of contact for matters concerning QA and DON Laboratory QA Program. Authorizing the suspension of project execution, if QA requirements are not adequately followed.
Michael Meyer	Project Manager	Battelle	Coordinating work activities of subcontractors and ensuring that all personnel adhere to the administrative and technical requirements of the project. Monitoring and reporting the progress of work and ensuring that the project deliverables are completed on time and within project budget. Monitoring the budget and schedule and notifying the RPM of any changes that may require administrative actions. Ensuring adherence to the quality requirements of the contract, project scope of work, and the QC Plans. Ensuring that all work meets the requirements of the technical specifications and complies with applicable codes and regulations. Ensuring that all work activities are conducted in a safe manner in accordance with the site-specific health and safety plan, U.S. Army Corps of Engineers' (USACE's) Safety and Health Requirements (EM-385-1-1), and all applicable Occupational Safety and Health Administration (OSHA) regulations. Serving as the primary contact between DON, Battelle and Trihydro for actions and information related to the work and including appropriate technical personnel in decision-making.

SAP Worksheet #7 - Personnel Responsibilities Table (Continued)

Name	Title/Role	Organizational Affiliation	Responsibilities
			Coordinating satisfactory resolution and completion of evaluation and acceptance report for nonconformance reports.
Elizabeth Branch	QAO	Battelle	Establishing and maintaining the project QC Program. Developing SAP. Overseeing QC, including chemical data validation. Acting as a focal point for coordination for quality matters across all activities and resolving quality issues. Suspending project activities, if quality standards are not maintained. Interfacing with Battelle, Trihydro, and DON, including NAVFAC QAO, on quality-related items. Conducting laboratory QC audits to ensure that project plans are being followed. Performing reviews of audit and surveillance reports conducted by others. Implementing DON technical direction letters related to quality topics.
Kelly Horiuchi	Laboratory Project Manager	ALS Simi Valley Laboratory	Ensuring that analyses are performed in accordance with the SAP Submitting accurate and complete reports within required timelines
Stella Cuenco	Data Validation Project Manager	LDC	Ensuring analytical data validation is performed in accordance with the SAP Submitting accurate and complete validation reports within required timelines.

SAP Worksheet #8 - Special Personnel Training Requirements Table

Project Function	Specialized Training by Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
None						

Note: No Special Personnel Training is required for this project. Field personnel have been trained in sampling procedures and hold OSHA 40-hour Hazardous Waste Operations and Emergency Responses Training certification and appropriate refresher training certification.

SAP Worksheet #9 - Project Scoping Session Participants Sheet

Project Name: Keyport Area 8 Vapor Intrusion Test Project Manager: Michael Meyer			Site Name: Keyport Area 8 Site Location: Keyport, WA	
Date of Session: 22 February 2017 **Scoping Session Purpose:** Scope Overview Discussion				
Name	**Title**	**Affiliation**	**Phone #**	**E-mail Address**
Carlotta Cellucci	Remedial Project Manager	NAVFAC Northwest	360-396-1518	carlotta.cellucci@navy.mil
Mahbub Alam	Project Manager	Department of Ecology	360-407-6913	mala461@ecy.wa.gov
Harry Craig	Project Manager	EPA	509-326-3689	Craig.harry@epamail.epa.gov
Denice Taylor	Project Manager	Suquamish Tribe	360-394-8449	dtaylor@suquamish.nsn.us
JoAnn Grady	Team Facilitator	Grady and Associates	907-321-3213	joanngrady@gmail.com
Eric Zenter	Representative	Boreal Communications	907-723-6430	ejzentner@gmail.com

Comments/Decisions: The Team agreed with the VI study design as presented as a path forward on OU 1 and OU 2 Area 8 (as presented in this SAP).

The team agreed to engage in a collaborative process by which to determine the best administrative option to address the Area 1 hotspots.

Action Item: Ms. Cellucci will contact Ms. Caldwell and discern if methane will also be tested in the VI study at Area 1. She will send the response to the team.

Navy will provide the PDF and redline version of the Keyport Area 1-Phase 2 Investigation Sampling and Analysis Plan to the team by late May 2017.

Ms. Grady will send Mr. Craig and Mr. Alam the summary comments from the last four Keyport meetings.

Ms. Cellucci will contact Mr. Nenninger to review the OUB 1 NEX Gas Station Tidal Influence Study.

Ms. Cellucci will send the Final Area 8 Marine Investigation Report to Ms. Taylor and Mr. Craig.

Ms. Cellucci will send an e-mail to the team extending the comment period for the OU-1 Phase 2 Investigation Report to March 10, 2017.

SAP Worksheet #9 - Project Scoping Session Participants Sheet (Continued)

Project Name: Keyport Area 8 Vapor Intrusion Test Project Manager: Michael Meyer			Site Name: Keyport Area 8 Site Location: Keyport, WA	
Date of Session: 25 October 2017 **Scoping Session Purpose:** Resolution of Comments on Draft SAP				
Name	**Title**	**Affiliation**	**Phone #**	**E-mail Address**
Carlotta Cellucci	Remedial Project Manager	NAVFAC Northwest	360-396-1518	carlotta.cellucci@navy.mil
Mahbub Alam	Project Manager	Department of Ecology	360-407-6913	mala461@ecy.wa.gov
Harry Craig	Project Manager	EPA	509-326-3689	Craig.harry@epamail.epa.gov
Denice Taylor	Project Manager	Suquamish Tribe	360-394-8449	dtaylor@suquamish.nsn.us
Donna Caldwell	Subject Matter Expert	NAVFAC Atlantic	757-322-4816	Donna.caldwell@navy.mil
Matt Jones	Vapor Intrusion Technical Lead	Trihydro Corporation	360-312-9109	mjones@trihydro.com
Michael Meyer	Battelle Project Manager	Battelle	206-601-1309	meyerm@battelle.org
Betsy Branch	Battelle QAO	Battelle		branchb@battelle.org
JoAnn Grady	Team Facilitator	Grady and Associates	907-321-3213	joanngrady@gmail.com
Eric Zenter	Representative	Boreal Communications	907-723-6430	ejzentner@gmail.com

Comments/Decisions: The Team discussed Ecology's comments on the draft SAP. EPA and the Suquamish Tribe did not have any comments on the draft SAP. Ecology agreed with the responses to comments provided by the Navy, except for the General comment and comments 1b and 1c. Following the discussion, the team agreed to revise the responses for these comments, including revisions to the sampling locations. Final sampling locations are shown on Figure 1, and final responses to Ecology's comments are included in Attachment A.

SAP Worksheet #10 - Conceptual Site Model

The locations of NBK Keyport and Area 8 are shown on Figure 1. Keyport is an unincorporated community in Kitsap County, Washington, and NBK Keyport serves as one of two divisions of the Naval Undersea Warfare Center (NUWC), providing Fleet readiness support for submarines, torpedoes, land attack systems and Fleet training systems. NUWC Keyport is located on Puget Sound between the Olympic and Cascade mountain ranges and is surrounded on three sides by water: Dogfish Bay to the west and Liberty Bay to the north, and Port Orchard to the east. Area 8 occupies approximately 1 acre on the eastern portion of NBK Keyport, on a manmade peninsula within a heavily industrialized area and encompasses a parking lot. The parking lot is on the site of a former plating shop (Building 72) which was demolished in 1999. The area is predominantly flat and almost entirely paved, with surrounding industrial buildings.

Past releases at Area 8 include spillage of chrome plating solution containing VOCs onto the ground; discharge of plating wastes into a utility trench; and leakage of plating solutions through cracks in the plating shop floor, waste disposal pipes, and sumps during plating shop operation. These chrome plating solutions and plating wastes contained VOCs and metals. Petroleum hydrocarbons (diesel and heavy oil) were also released to the environment from leaky underground storage tanks and underground concrete vaults located within Area 8. Semivolatile organic compounds (SVOCs) associated with the petroleum release were detected in soil at low concentrations below Washington State Model Toxics Control Act (MTCA) Method B cleanup levels based on soil ingestion, protection of drinking water, and protection of surface water. For groundwater, VOCs and metals (arsenic, cadmium, and chromium) were identified as COCs based on residential use of groundwater as drinking water and inhalation during household use. For subsurface soil, arsenic and cadmium were identified as major contributors to future resident's risk, based on ingestion of produce grown in the soil. The OU 2 ROD was signed in 1994, and identified VOCs and metals as the COCs associated with Area 8. Following the signing of the OU 2 ROD, the Navy performed the following remedial actions:

1. Demolition of building 72 and removal of associated soil hotspots in July 1998 and March 1999, based on cadmium and chromium concentrations exceeding state MTCA Method B cleanup levels for soil ingestion (80 mg/kg for cadmium and 400 mg/kg for chromium) (DON, 1999).

2. Implementation of institutional controls in 2000 to prevent exposure to soil and groundwater during hypothetical future residential land use.

3. Installation and long-term monitoring (LTM) of four new wells starting in 1995 and continuing through 2017.

4. Initiation of sediment and tissue LTM in the intertidal zone of Area 8 starting in 1996 and continuing every 4 years or less thereafter, including 2000, 2004, 2008, 2012 (sediment only), 2015, and 2016.

5. Evaluation of human health and ecological risks based on tissue and sediment data.

6. Execution of independent remedial actions under MTCA related to past petroleum releases.

SAP Worksheet #10 - Conceptual Site Model (Continued)

The ROD also calls for implementation of contingent groundwater control actions, if Area 8 groundwater is found to present an unacceptable risk to human health or the environment, based on sediment and tissue monitoring; however, contingent groundwater control actions have not been implemented. The Navy collected additional sediments and tissue data (in 2015 and 2016) to perform an in-depth human health and ecological risk assessment of contaminant concentrations in intertidal sediment and clam tissue (planned for completion in 2018). Groundwater controls will be implemented if the risk assessment indicates that groundwater migrating from Area 8 has impacted sediment and clam tissue on the adjacent beach resulting in an unacceptable risk to human health or the environment.

Monitoring at Area 8 has been conducted since the signing of the ROD and has included groundwater, sediment, and tissue sampling and analysis.

Inorganics, including arsenic, cadmium, chromium (total), hexavalent chromium, copper, lead, mercury, nickel, silver, thallium, zinc, and cyanide were analyzed in groundwater samples starting in 1995. Chromium was speciated during initial rounds of groundwater sampling to assess the ratio of trivalent to hexavalent chromium. The data report covering the 2000 sampling event recommended that chromium speciation be discontinued based on the conclusion that measured total chromium values could be assumed to be 100 percent hexavalent chromium (DON, 2001). This report also recommended that cyanide be removed from the analyte list for tissue, seep, and sediment because it had not been detected in the groundwater samples since 1998. It was agreed by the Navy and Ecology that another round of cyanide sampling would be collected in groundwater from MW8-12 (historically the highest concentrations were observed at this well) in spring 2002 (DON, 2001). The cyanide concentration at MW8-12 during the 2002 sampling event was found to be well below both groundwater and surface water remedial goals, so groundwater analysis for cyanide was discontinued.

The fourth five-year review (DON, 2015b) concluded that a VI study, which had not been performed as part of historical environmental investigations, was warranted for Area 8, based on new EPA risk based VI guidance (EPA 2015). An evaluation of the VI pathway was recommended because of VOC concentrations detected in groundwater in the vicinity of worker-occupied buildings. The primary potential human health receptors for Area 8 are workers within the buildings surrounding the area, including Buildings 82, 85, and 98. Based on the presence of VOC concentrations exceeding VI screening criteria in groundwater at Area 8, intrusion of vapors emanating from the groundwater plume could impact occupants of nearby buildings.

SAP Worksheet #11 - Project Quality Objectives/Systematic Planning Process Statements

Project quality objectives (PQOs) are an integrated set of qualitative and quantitative decision statements that define data quality requirements based on the end use of the data. The EPA has developed a seven-step process to clarify study objectives, define the appropriate type of data, and specify acceptable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions. The PQO steps addressed in the subsections that follow have been development in accordance with the UFP-QAPP guidance (EPA, 2005).

Step 1 – Problem Statement

This step identifies the issues to be addressed.

Concentrations of VOCs (specifically, TCE) in the groundwater beneath OU 2 Area 8 exceed VI default screening levels, posing a potential risk to human health for workers in buildings within 100 feet of Area 8. No data are available to assess the VI pathway and exposures via this pathway could impact occupants of nearby buildings.

Step 2 - Identify the Decision

The purpose of this step is to define the decision that will be made using data to address the problem. The overall decision to be made based on the data collected under this SAP is to assess the potential for risk to human health through the VI pathway. Specific decisions include:

1. Do the concentrations of VOCs in soil vapor samples indicate the potential for vapor intrusion into nearby buildings that warrants further investigation?

2. Is the lateral or vertical distribution of VOCs in soil vapor indicative of preferential vapor migration pathways that warrant further investigation?

Step 3 - Identify Inputs to the Decision

Inputs to the decision consist of soil vapor samples that will be collected and analyzed for the VOCs TCE, tetrachloroethene (PCE), 1,1-dichloroethene, cis-1,2-dichloroethene, trans-1,2-dichloroethene, vinyl chloride, 1,4-dioxane, 1,1,2-TCA, benzene, carbon tetrachloride, and ethylbenzene.

Step 4 - Define the Study Boundaries

The lateral boundaries of the study consist of the area shown in Figure 1, and encompass the buildings nearest the area where VOCs are present in groundwater.

The vertical boundary of the study will extend to an average depth of 10 feet below ground surface (bgs), anticipated to be the depth of the capillary fringe.

The temporal boundary of the study is that this investigation will consist of a one-time “snapshot” sampling of VOC concentrations in soil vapor.

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

Step 5 – Develop a Decision Rule

The process or "rules" for making the decisions listed under Step 2 are described in this section. Rules include how field decisions will be made, as well as how data will be interpreted.

Decision Rule 1: If the concentrations of any target VOCs in any soil vapor sample exceed the MTCA Method C sub-slab soil gas screening levels established in Ecology's guidance (Ecology, 2016), conclude that additional investigation of the VI pathway is warranted. If concentrations do not exceed the MTCA Method C sub-slab soil gas screening levels, conclude that no additional VI investigation is warranted.

Decision Rule 2: If the concentrations of target VOCs in soil vapor exhibit a vertical or lateral distribution that implies an association with utility backfill or other potential preferential pathways, and VOC concentrations exceed screening levels, conclude that additional investigation of VOC migration along preferential pathways is warranted.

Step 6 - Specify Limits on Decision Error

The sampling and analysis program described in this SAP is designed to ensure that data quality and type are appropriate for the intended use and to reduce the potential for decision errors. A judgmental sampling approach is used to determine sampling locations to resolve the decision rules. There are two types of decision errors associated with judgmental sampling: sampling design errors and measurement errors.

Sampling design errors are a function of the selection of the sampling locations, number of samples, or analytical methods. Sampling design errors will be minimized by ensuring the design is consistent with the conceptual site model (CSM).

Measurement errors are a function of the procedures used to collect, extract and analyze the samples. This SAP presents acceptable limits on decision errors stemming from field and laboratory measurement errors in the context of detection thresholds and precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS) criteria. Measurement error is further managed by using laboratory standard operating procedures (SOPs), third-party review of the data records, and data quality management.

Analysis of soil vapor samples will be performed by an Environmental Laboratory Accreditation Program (ELAP)- or National Environmental Laboratory Accreditation Program (NELAP)-certified, analytical laboratory that is also accredited in Washington State. For this project, ALS Simi Valley Laboratory and Laboratory Data Consultants (LDC) located in Carlsbad, California will perform analyses and data validation, respectively, in accordance with the SOPs listed in Worksheet #23 and with the analytical requirements specified in this SAP. Laboratory certificates of accreditation are provided in Attachment B. Laboratory SOPs are provided in Attachment C.

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

Step 7 – Optimize the Sampling Design

The sampling design has been optimized by establishing a step-wise approach of collecting and interpreting soil vapor data to assess the need for more invasive sub-slab and indoor air sampling.

SAP Worksheet #12 - Field Quality Control Samples

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria
Field Duplicate	VOCs	One per 10 project samples	Precision	RPD ≤ 25% when analytes detected in field duplicate sample pairs at ≥ LOQ
Field Duplicate	Helium	One per 10 project samples	Precision	RPD ≤ 25% when analytes detected in field duplicate sample pairs at ≥ LOQ

LOQ limit of quantitation
RPD relative percent difference

SAP Worksheet #13 - Secondary Data Criteria and Limitations Table

Secondary Data	Data Source	Data Generator(s)	How Data Will Be Used	Limitations on Data Use
Results of periodic groundwater sampling	LTM reports	U.S. Navy LTM contractor	Comparison to historical results of VOCs in groundwater to assess any trends or changes in lateral extent that may be relevant to VI	VOCs in groundwater data will be used only to make relative comparisons to historical groundwater results, not directly to assess VI risk

SAP Worksheet #14 - Summary of Project Tasks

This worksheet summarizes the project tasks for the VI study.

Site Preparation Tasks:

1. Perform site walk to mark for utility clearance and identify access issues.
2. Prepare traffic control plan to control vehicle movement through parking lot during drilling.
3. Perform utility clearance in accordance with NAVFAC Northwest SOP I-A-6.

Soil Vapor Sampling Tasks:

1. Locate soil vapor sample locations.
2. Install sample point as described in Worksheet #17.
3. Allow well to equilibrate.
4. Label Summa canisters with sample identification (ID), station ID, sampler initials, collection date and time in accordance with NAVFAC Northwest SOP III-E.
5. Record initial vacuum within canister.
6. Setup sample train (fittings and valves, tubing, flow restrictor, Summa can).
7. Perform shut-in test prior to purging.
8. Purge approximately three well casing/line casing volumes.
9. Perform and record field readings of each purge volume.
10. Perform shut-in test prior to sampling.
11. Record start and end times of sample collection and final canister pressure at end of sample collection.
12. Pack Summa canisters for shipment.
13. Complete and QA the chain of custody for collected samples.
14. QA the sample labels and packing against the chain of custody.

Survey Tasks:

1. Record coordinates of sampling locations using a global positioning system (GPS) in accordance with NAVFAC Northwest SOP I-G-2.

Analysis Tasks:

1. Soil vapor samples will be submitted to ALS Simi Valley Laboratory and analyzed for VOCs using EPA Method TO-15 and helium using EPA Method 3C.

Investigation-Derived Waste Tasks:

1. Soil cuttings and decontamination water will be containerized and staged on site pending receipt of laboratory analyses.
2. NBK Keyport Hazardous Waste Department will dispose of containerized waste following receipt of laboratory data.
3. Non-contaminated personal protective equipment will be disposed of as municipal waste in a receptacle designated by the Navy.

Quality Control Tasks:

1. Implement Navy field SOPs for sampling, related field tasks, and sample preparation/analysis methods as listed in Worksheet #18.
2. Collect and analyze field QC samples are described in Worksheets #12 and #20.
3. Fixed-laboratory data for VOC, 1,4-dioxane and helium analyses will undergo Stage III independent data validation. Refer to Worksheet #34–36 for details.

Secondary Data:

1. Incorporate secondary data into interpretations regarding the representativeness of the soil vapor samples based on the similarity of the recent extent of VOCs in groundwater to the historical extent.

Data Management Tasks:

1. Soil boring logs and well construction diagrams will be completed and loaded into Naval Installation Restoration Information Solution (NIRIS).

SAP Worksheet #14 – Summary of Project Tasks (Continued)

2. Analytical data will be submitted for loading to the NIRIS electronic database and Ecology's Environmental Information Management System database following validation and/or verification. Soil boring logs and/or well construction diagrams will be completed and also submitted for loading to NIRIS.
3. Original hard copies of analytical data reports and independent data validation reports will be sent to the NAVFAC Northwest Records Manager for archiving at National Archives and Records Administration.
4. Original hard-copy field data will be retained in the secure central project file, and photocopies will be used for data-reduction project work.

Documentation and Records (NAVFAC Northwest SOP III-E):

1. Field data will be recorded in a bound logbook.
2. Logbooks, chain of custody forms, airbills, and other hard-copy field records will be scanned and retained in the Battelle electronic project file.
3. Upload final validated data to both the Navy Installation Restoration Information Solution (NIRIS) database and the Ecology Environmental Information Management (EIM) System database.

Reporting and Evaluation:

1. Report results of field and laboratory work.
2. Draw conclusions regarding whether additional VI investigation is warranted.

Data Packages:

Laboratory data will be recorded in a Contract Laboratory Program or equivalent format, including sample ID, analysis date, parameter values, method detection limits, and reporting limits. Laboratory data reports must include all information required to perform a comprehensive data validation. The data package elements required to perform data validation are listed below and include both summary forms and instrumental printouts as applicable:

- Initial calibration
- Initial calibration verification
- Continuing calibration verification
- Blank spike results and control charts
- Results from initial calibration and continuing calibration blanks
- Method blank results
- Instrument tuning
- Internal standard results
- Preparation logs
- Matrix spikes (MSs)/matrix spike duplicates (MSDs)
- Dilution tests
- Post-digestive spike
- Any other raw data necessary to fully document the analyses performed on the subject sample group as outlined in DoD QSM v5 (DoD, 2016), Appendix A.

Assessment/Audit Tasks:

Where applicable, the laboratory (or laboratories) selected to perform the analytical testing for this project will have current accreditation under the DoD ELAP, which is based on the review of the laboratory's QA manual, selected SOPs, SOP master list, list of major analytical instrumentation, performance test results, and an on-site assessment performed under DoD ELAP.

Cited NAVFAC Northwest SOPs are provided in Attachment D.
Laboratory chain of custody forms are provided in Attachment E.

SAP Worksheet #15 - Reference Limits and Evaluation Tables

Laboratory: ALS Simi Valley Laboratory
Matrix: Soil Vapor
Analytical Group: VOCs - EPA Method TO-15

Analyte	CAS Number	Project Action Limit (PAL) (µg/m³)	PAL Reference	Project Quantitation Limit (QL) Goal (µg/m³)[3]	Laboratory-Specific (µg/m³)		
					LOQ	LOD	DL
1,1,2-Trichloroethane	79-00-5	6.67	MTCA Method C[2]	0.61	1.3	1.1	0.40
1,1-Dichloroethene	75-35-4	6,667	MTCA Method C[2]	610	1.3	1.1	0.43
1,4-Dioxane	123-91-1	167	MTCA Method C[1]	3.3	1.3	1.1	0.40
Benzene	71-43-2	107	MTCA Method C[1]	2.1	1.3	1.1	0.40
Carbon tetrachloride	56-23-5	139	MTCA Method C[1]	2.8	1.3	1.1	0.38
cis-1,2-Dichloroethene	156-59-2	NE	MTCA Method C	0.5	1.3	1.1	0.40
Ethylbenzene	100-41-4	33,333	MTCA Method C[2]	3,050	1.3	1.1	0.40
Tetrachloroethene	127-18-4	1,333	MTCA Method C[2]	64	1.3	1.1	0.35
trans-1,2-Dichloroethene	156-60-5	2,000	MTCA Method C[2]	183	1.3	1.1	0.48
Trichloroethene	79-01-6	66.7	MTCA Method C[2]	2.5	1.3	1.1	0.35
Vinyl chloride	75-01-4	93.3	MTCA Method C[1]	1.9	1.3	1.1	0.43

NE- Not established; µg/m³ micrograms per cubic meter Project action limits are MTCA Method C sub-slab soil gas screening levels from Table B-1 (updated April 6, 2015) levels, available at: http://www.ecy.wa.gov/programs/tcp/policies/VaporIntrusion/Vapor%20Intrusion%20Table%20update%20April%206%202015.xlsx

[1] – Based on the cancer value

SAP Worksheet #15 – Reference Limits and Evaluation Tables (Continued)

[2]– Based on the non-cancer value
[3]– Project Quantitation Limit Goals were selected as approximately 1/5 of the MTCA Method B screening level (when available), to allow for potential future screening against Method B screening levels.
[5]- Based on CLARC air cleanup level for trans-1,2-dichloroehtene by Method C (non-cancer) of 60.0 ug/m3 (See CLARC Table "Air – Method C", https://fortress.wa.gov/ecy/clarc/FocusSheets/Air%20Method%20B.pdf . Using a 0.03 attenuation factor, the PAL is 2,000 ug/m3.

Laboratory: ALS Simi

Matrix: Soil Vapor

Analytical Group: Helium - EPA Method 3C Mod

Analyte	CAS Number	PAL (µg/m^3)	PAL Reference	Project QL Goal (µg/m^3)	Laboratory-Specific (µg/m^3)		
					LOQ	LOD	DL
Helium	7440-59-7	NE	NE	25	25	25	4.6

NE- Not established

SAP Worksheet #16 - Project Schedule

Deliverable	Timeframe	Planned Completion Date
Final SAP	Following incorporation of comments on draft	11/4/17
Draft Accident Prevention Plan (APP) to Navy	60 Days in advance of planned sampling	9/19/17
Final APP	Following incorporation of Navy comments on draft	11/9/17
Field Work	Fall 2017	11/17/17
Draft Report to Regulators/Stakeholders	Following incorporation of Navy comments on internal draft	4/1/18
Final Report	Following incorporation of comments on draft	6/7/18

SAP Worksheet #17 - Sampling Design and Rationale

This section describes the sampling rationale and design for collection of soil vapor samples.

Sampling Rationale

Soil vapor sampling was selected to meet the PQOs in accordance with Ecology guidance (Ecology, 2016) for sites where groundwater is the source of potential VI. Also in accordance with Ecology's guidance, two depths of soil vapor sampling are planned, one at 5 feet bgs and one just above the capillary zone. Groundwater flow direction at the site is to the east-southeast toward the adjacent water body. All existing buildings within 100 ft. of the site are upgradient of the former plating shop and the groundwater VOC plume. Therefore, soil vapor sample locations were selected to be exterior to the downgradient side of the adjacent buildings, between the building and the VOC plume. To ensure refinement of the conceptual site model (CSM) by understanding diffusion or preferential flow in the vadose zone, vertical soil vapor profile sampling is proposed at the capillary fringe to assess vapor source strength from the groundwater and at 5 feet bgs to assess vertical vapor attenuation and source strength nearest the building foundations. Sample locations have also been selected near utilities that could act as preferential pathways for vapor migration into the adjacent buildings. Soil gas sampling will not occur during a significant rain event and will only occur after five days without a significant rain event (CalEPA, 2015). A significant rain event is defined as 1/2 inch or greater of rainfall during a 24-hour period.

Sampling Design

Six soil vapor wells (SV-1 through SV-6) will be installed with screens at two discrete depths, 5 feet bgs and immediately above the capillary fringe, anticipated to be approximately 10 feet bgs. Samples will not be collected from a depth less than 5 feet bgs. Installation of soil vapor wells will be performed using direct push drill methods by advancing 5-foot long, 2.25-inch diameter, steel rods until the water table has been encountered. Soil will be continuously collected in 5-foot long acetate liners, so that the actual depth of the capillary fringe can be obtained. If the capillary fringe is not detected within the first two rod lengths (10 feet bgs), the boring will be advanced to 15 feet bgs. An aliquot of each 2-foot interval will be sealed in a zip-lock bag to perform a headspace analysis of total organic vapors using a photoionization detector (PID). Vapor concentrations and a description of the soil will be recorded on field boring logs in general accordance with the unified soil classification system.

Upon reaching the target total depth, the drive rod and sampler will be removed. The sampler will be disconnected from the drive rods and an expendable drive point attached. The rods will be re-inserted into the boring to the target sample depth, based on soil core data and the 5-foot rod lengths, and then withdrawn approximately 12 inches, disengaging the expendable drive point. A ¼-inch diameter Nylaflow tube, attached to a sample port, will be inserted into an open slotted polyvinyl chloride (pvc) pipe and set approximately 6 inches off the bottom of the boring. Number 3 washed aquarium sand, or equivalent will be placed through the steel rods, concurrent with its removal, such that sand extends from approximately 6 inches below to 6 inches above the soil vapor sampling point. Approximately 12 inches of fine dry bentonite crumble will be placed in the boring on top of the sand pack (Ecology, 2016). Additional bentonite crumble will be hydrated in a container at the surface and placed in the hole concurrent with the removal of the drive rods. The above process will also be repeated for the shallow target depth. That is, each soil gas

SAP Worksheet #17 – Sampling Design and Rationale (Continued)

well will include two nested sample points within the same boring. In the event that completion integrity of nested soil gas wells is compromised, separate, but closely spaced borings may be used to target different depths.

After the multi-depth probe is installed, and the annular seal placed, the drive rods will be removed from the hole. The remaining open hole will be tremmied with hydrated bentonite to the ground surface. The tops of the Nylaflow tubes will be equipped with a ball valve, and labeled as to depth. A diagram of the typical nested vapor point construction is shown on Figure 2. Soil gas sampling procedures, as described below, will be conducted a minimum of two hours after temporary monitoring point installation to allow time for the seals to hydrate and for subsurface conditions to stabilize.

Shut-in testing will be conducted to confirm the integrity of the sample train (Figure 3) prior to conducting soil gas purging and collecting the final sample for laboratory analysis. Shut-in testing will be performed by closing the ball valve to the soil gas probe, inducing a vacuum of approximately 15 inches of mercury on the sample equipment, the valves at both ends of the tubing will then be closed and the vacuum gage observed to ensure the vacuum does not dissipate. If the vacuum dissipates, the leaky component in the sample train will be identified and repaired, or replaced (if necessary), and the shut-in test will be repeated until the sample train can hold a constant vacuum of 200 milliliters per minute. If groundwater is pulled through the Nylaflow tubing during purging, the boring location and/or depth will be deemed inadequate for soil gas sampling collection and a new boring will be advanced for sampling immediately adjacent to the original location. Shut-in test results will be recorded on the soil vapor monitoring field forms (Attachment F).

After completion of the initial shut-in testing, the soil vapor wells will be purged using an approximate 200 milliliter per minute flow controller, vacuum pump, Tedlar bag, and lung box. After each volume of soil gas is purged (approximately 5 to 8 minutes and 1 to 1.5 liters of soil vapor), the total organic vapor concentration will be measured using a PID. In addition, oxygen, carbon dioxide, and methane concentrations in the purged sample will be measured using a fixed gas meter. Approximately 3 purge volumes of soil vapor will be removed.

Helium will be used as a tracer gas for measuring the potential for leakage of ambient air through the annular seal of the soil vapor probe or connections within the sample train. A shroud will be placed around each well, flow controller, Summa canister, and fittings during purging. Helium gas will be added to the shroud through a small port. The concentration of helium will be recorded using a multi-gas detector to confirm that a minimum helium content of 10% is maintained beneath the shroud during purging. The range of helium maintained in the shroud will be recorded during each purge interval.

The concentrations of helium in the soil gas samples recovered in the Tedlar bag during each purge interval will be screened using the multi-gas detector. The concentrations of helium in the soil gas sample will be confirmed to be less than 5% of the concentration in the shroud. If the helium concentration in the soil gas sample exceeds 5% of the shroud concentration, the probe and sampling equipment will be isolated at points along the sample train and inspected for leaks,

SAP Worksheet #17 – Sampling Design and Rationale (Continued)

repaired or replaced as needed, and additional purging will be conducted to ensure collection of a representative soil gas sample.

After purging and confirmation of the integrity of the probe and sampling equipment using helium as a tracer gas, the soil gas sample will be collected using a laboratory–supplied pre-evacuated 1-liter Summa canister with a 5-micron in-line filter and flow controller set at a constant vacuum of 200 milliliters per minute. The vacuum of the canisters will be recorded prior to sample collection (initial) and after sample collection (final) with a dedicated gauge. Additionally, the vacuum will be observed during the sample collection process with separate individual gauges included on the sample train. To obtain an appropriate sample volume, the final vacuum is anticipated to be between approximately -1 and -3 inches of mercury.

Samples for VOCs and laboratory confirmation of the helium concentration will be collected. The Summa canister, in-line filter, and flow controller will be placed beneath the shroud and a helium content of at least 10% will be maintained during collection of the final soil gas sample. Following completion of sampling activities, the ball valve on the Summa canister will be closed and capped.

A completed chain of custody form will accompany the samples to the analytical laboratory. Each Summa canister will have an attached label that includes the following information:

- Site Name
- Date/Time
- Unique Summa Canister ID
- Unique Sample ID
- Sampler Name
- Requested Laboratory Analyses
- Initial Summa Canister Vacuum Upon Receipt from the Laboratory
- Final Summa Canister Vacuum Following Soil Gas Sample Collection

Sampling locations will be documented to the nearest foot using GPS technology. Soil vapor samples will be sent to ALS Simi Valley laboratory and will be analyzed for the VI constituents TCE, PCE, 1,1-dichloroethene, cis-1,2-dichloroethene, trans-1,2-dichloroethene, vinyl chloride, 1,4-dioxane, 1,1,2-TCA, benzene, carbon tetrachloride, and ethylbenzene using EPA Method TO-15 to evaluate VI risks, and for helium using EPA Method 3C to ensure sample competency.

All field activities, including sample collection and analyses, will be conducted in accordance with approved project plans. All required samples will be analyzed in accordance with ELAP and Washington State approved methods. Laboratory analyses will include all appropriate sample preparation methods required to obtain accurate and defensible data.

SAP Worksheet #17 – Sampling Design and Rationale (Continued)

All analytical data will be validated by a third-party data validator. Laboratory analytical SOPs are provided in Attachment C. Samples will be analyzed as shown in Worksheet #18. QC samples will be collected as shown in Worksheet #20.

SAP Worksheet #18 - Sampling Locations and Methods/SOP Requirements Table

Sampling Location / ID Number	Matrix	Depth	Analytical Group	Number of Samples	Sampling SOP Reference
OU2A8-SV-1-##	Soil Vapor	~5 and ~10 feet bgs	VOCs by TO-15 Helium by EPA Method 3C	2	Worksheet #21
OU2A8-SV-2-##	Soil Vapor	~5 and ~10 feet bgs	VOCs by TO-15 Helium by EPA Method 3C	2	Worksheet #21
OU2A8-SV-3-##	Soil Vapor	~5 and ~10 feet bgs	VOCs by TO-15 Helium by EPA Method 3C	2	Worksheet #21
OU2A8-SV-4-##	Soil Vapor	~5 and ~10 feet bgs	VOCs by TO-15 Helium by EPA Method 3C	2	Worksheet #21
OU2A8-SV-5-##	Soil Vapor	~5 and ~10 feet bgs	VOCs by TO-15 Helium by EPA Method 3C	2	Worksheet #21
OU2A8-SV-6-##	Soil Vapor	~5 and ~10 feet bgs	VOCs by TO-15 Helium by EPA Method 3C	2	Worksheet #21
OU2A8-SV-7-##[1]	Soil Vapor	~5 and ~10 feet bgs	VOCs by TO-15 Helium by EPA Method 3C	1	Worksheet #21
OU2A8-SV-8-##[1]	Soil Vapor	~5 and ~10 feet bgs	VOCs by TO-15 Helium by EPA Method 3C	1	Worksheet #21

depth of sample collection
bgs below ground surface
ID identification
OU operable unit
SOP standard operating procedure
1 - blind duplicate sample

SAP Worksheet #19 - Field Sampling Requirements Table

Matrix	**Analytical Group**	**Analytical and Preparation Method/SOP Reference**	**Containers** (number, size, and type)	**Sample Volume** (units)	**Preservation Requirements** (chemical, temperature, light protected)	**Maximum Holding Time** (preparation/ analysis)
Soil Vapor	VOCs	EPA Method TO-15	One 1 Liter Summa Canister	1 Liter	None	30 Days
Soil Vapor	Helium	EPA Method 3C Mod	One 1 Liter Summa Canister	1 Liter	None	30 Days

SAP Worksheet #20 - Field Quality Control Sample Summary Table

Matrix	Analytical Group	No. of Sampling Locations[1]	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equip. Blanks	No. of Trip Blanks	No. of PT Samples	Total No. of Samples to Lab
Soil Vapor	VOCs, Helium	12	2	N/A	N/A	N/A	N/A	0	14

[1] Total number of samples to be collected. Six soil vapor probes will be installed with one screen set at 5 feet bgs and one screen set at a maximum of 10 feet bgs.

N/A not applicable

SAP Worksheet #21 - Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
I-A-6	Standard Operating Procedure, Utility Clearance, February 2015	NAVFAC Northwest	N/A	N	
I-A-9	Standard Operating Procedure, General Field Operation, February 2015	NAVFAC Northwest	See Attachment D (I-A-9-1: Field Equipment Checklist)	N	
I-A-10	Standard Operating Procedure, Monitoring/Sampling Location Recording, February 2015	NAVFAC Northwest	See Attachment D (I-A-10-1: Monitoring/sampling Location Information Form)	N	
I-A-11	Standard Operating Procedure, Sample naming, February 2015	NAVFAC Northwest	N/A	N	
I-B-4	Standard Operating Procedure, Borehole Abandonment, February 2015	NAVFAC Northwest	Drill rig, wheelbarrow or drum, filter pack material, bentonite powder, bentonite pellets or chips, Portland cement, tremie pipe	Y	Clip the top of tubing off and perform minor sealing on top by hand.
I-G-2	Standard Operating Procedure, GPS Surveying, August 2014	NAVFAC Northwest	GPS device	N	
III-D	Standard Operating Procedure, Logbooks, April 2015	NAVFAC Northwest	Waterproof hardbound field logbook with numbered pages, indelible marking pen, ruler, clipboard	N	
III-E	Standard Operating Procedure, Record Keeping, Sample Labeling, and Chain-of-Custody Procedures, April 2015	NAVFAC Northwest	N/A	N	

SAP Worksheet #21 - Project Sampling SOP References Table (Continued)

III-G	Standard Operating Procedure, Sample Handling, Storage, and Shipping, April 2015	NAVFAC Northwest	N/A	N	
III-I	Standard Operating Procedure, Equipment Decontamination, April 2015	NAVFAC Northwest	See Section 2.4 – Cleaning Solutions and Techniques	N	
III-J	Standard Operating Procedure, Equipment Calibration, Operation, and Maintenance, April 2015	NAVFAC Northwest	N/A	N	
III-C	Standard Operating Procedure, Field QC Samples (Air), April 2015	NAVFAC Northwest	Glass sorbent tubes, filters, liquid impingers, Tedlar bags, Summa canisters	Y	Procedures in WS#17 take precedence over the SOP

N no
N/A not applicable or not available
NAVFAC Naval Facilities Engineering Command
SOP standard operating procedure
Y yes

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

Field Equipment	Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference	Comments
PID (Mini RAE or equivalent)	Maintenance	As needed	Per instrument specifications	In-house repair or return to manufacturer or rental vendor	Field Team Leader	Manufacturer's user manual	Replace batteries as needed
	Calibration	At the start of each sampling event and as needed based on daily calibration check	Per instrument specifications	Recalibrate until within the acceptable range or return to manufacturer or rental vendor	Field Team Leader	Manufacturer's user manual	
Micromanometer (The Energy Conservatory DG-500 or equivalent)	Maintenance	Check if operating properly daily	Per instrument specifications	Return to manufacturer or rental vendor	Field Team Leader	Manufacturer's user manual	Replace batteries as needed
Differential Pressure Recorder (Omniguard or equivalent)	Maintenance	Check if operating properly daily	Per instrument specifications	Manually reset zero point (if needed)	Field Team Leader	Manufacturer's user manual	Replace thermal printer paper as needed
Pressure gauges	Maintenance	Check if operating properly daily	Per instrument specifications	Return to manufacturer or rental vendor	Field Team Leader	Manufacturer's user manual	
Portable vacuum lung box and hand pump	Maintenance	Check if operating properly daily	Pumping at required flow rate to evacuate adequate volume prior to sampling	In-house repair or return to manufacturer	Field Team Leader	Manufacturer's user manual	Replace batteries as needed
Landfill gas meter (Landtec GEM500 or equivalent)	Calibration	At the start of each sampling event and as needed based on daily calibration check	Per instrument specifications	Recalibrate until within the acceptable range or return to manufacturer or rental vendor	Field Team Leader	Manufacturer's user manual	Replace batteries as needed

SAP Worksheet #22 - Field Equipment Calibration, Maintenance, Testing, and Inspection Table (Continued)

Field Equipment	Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference	Comments
Helium meter (Dielectric MGD-2002 or equivalent)	Calibration	At the start of each sampling event and as needed based on daily calibration check	Per instrument specifications	Recalibrate until within the acceptable range or return to manufacturer or rental vendor	Field Team Leader	Manufacturer's user manual	Replace batteries as needed

PID photoionization detector
SOP standard operating procedure

SAP Worksheet #23 - Analytical SOP References Table

Laboratory SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
VOA-TO15	Determination of Volatile Organic Compounds in Air Samples Collected in Specially Prepared Canister and Gas Collection Bags by Gas Chromatography/Mass Spectrometry (GC/MS), EPA TO-15, Rev 24, 6/3/17	Definitive	Soil Vapor / VOCs	GC/MS	ALS Simi Valley Laboratory	N
VOA-HHE	Analysis of Hydrogen and Helium Using Gas Chromatography with Thermal Conductivity Detection (TCD), EPA 3C Modified, Rev 6, 9/20/14	Definitive	Soil Vapor / Helium	GC	ALS Simi Valley Laboratory	N

GC gas chromatograph
GC/MS gas chromatograph/mass spectrometer

SAP Worksheet #24 - Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS	Bromofluorobenzene (BFB) Tuning Verification	Once every 24 hours or analytical batch	Ion abundance criteria as described in Table 3 of Method TO-15	1) Repeat BFB analysis 2) Retune instrument	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-TO15, Rev.24
GC/MS	Initial Calibration (ICAL) – minimum of five levels	Initially or if continuing calibration no longer meets criteria	Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: r2 ≥ 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99.	1) May repeat 1 point (if 5 levels) or 2 points (if 6 levels) 2) Inspect the system for problems and perform required maintenance 3) Repeat initial calibration Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-TO15, Rev.24

SAP Worksheet #24 - Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS	Initial Calibration Verification (ICV)	Following every ICAL	All reported analytes within ± 20% of true value.	1) Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Problem must be corrected. Samples may not be analyzed until there is a valid ICV.	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-TO15, Rev.24
GC/MS	Continuing Calibration Verification (CCV)	Once every 24 hours prior to sample analysis, if an ICAL has not been performed (within the last 24 hours). [DoD Quality Systems Manual 5.1 - A CCV standard must be analyzed at the end of the analytical batch]	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV.	1) Reanalyze CCV [DoD: Analyze two additional CCVs] 2) Identify and correct problem; re-analyze or if necessary qualify the data. 3) Repeat initial calibration if CCV corrective action is unsuccessful.	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-TO15, Rev.24
GC/MS	Internal Standards (IS)	All samples, duplicates, blanks and standards	Retention time (RT) must be ± 10 sec from most recent valid calibration (ICAL midpoint)	1) Identify and correct the problem 2) Reanalyze the sample unless obvious matrix interference exists. 3) Problem persists, qualify data.	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-TO15, Rev.24

SAP Worksheet #24 - Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/MS	Surrogate Standards	All samples, duplicates, blanks and standards	Appendix B, DoD QSM v5 limits	1) Identify and correct the problem 2) Reanalyze the sample unless obvious matrix interference exists 3) If problem persists, qualify data	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-TO15, Rev.24
GC/MS	Limit of Quantitation (LOQ)	Annual verification. [DoD: Quarterly verification required]	1) At or above the low standard of the current initial calibration. 2) % recovery for each analyte within laboratory generated control limits.	1) Reanalyze 2) Identify and correct problem; re-analyze. 3) Repeat verification at higher level to set higher LOQ if corrective action is unsuccessful.	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-TO15, Rev.24
GC/MS	Detection Limit (DL) with Limit of Detection Verification	Initially and verification performed once per 12-month period. [DoD: Quarterly limit of detection (LOD) verification required]	LOD Verification - Response with a minimum signal to noise ratio of 3:1	1) Repeat detection limit determination and LOD verification at higher concentration or perform and pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration.	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-TO15, Rev.24

SAP Worksheet #24 - Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/TCD	Initial Calibration (ICAL) – minimum of five levels	Initially and if continuing calibration no longer meets criteria	1) Analytes calibrated using average RF, RSD ≤15%. 2) Quadratic, ≥0.99% (utilized for hydrogen only, where necessary)	1) May repeat one point (if analyzing 5 levels) or two points (if analyzing 6 levels) 2) Inspect the system for problems and perform required maintenance 3) Repeat initial calibration Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-HHE Rev 6.0
GC/TCD	Initial Calibration Verification (ICV)	Following every ICAL	Percent recovery for each analyte 85-115%	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Problem must be corrected. Samples may not be analyzed until there is a valid ICV.	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-HHE Rev 6.0
GC/TCD	Continuing Calibration Verification (CCV)	Initial run of batch, every 10 samples, and end of batch (all samples must be bracketed by two CCVs)	1) Percent difference of ≤10% 2) RT for each analyte in the standard within 0.33 min. from mean RT from the ICAL.	1) Reanalyze CCV 2) Identify and correct problem; re-analyze or where appropriate qualify the data. 3) Repeat initial calibration if CCV corrective action is unsuccessful.	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-HHE Rev 6.0

SAP Worksheet #24 - Analytical Instrument Calibration Table (Continued)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GC/TCD	Laboratory Method Blank	Once every analytical batch of 20 or fewer samples	No analyte detected above the LOQ	1) Reanalyze blank 2) Identify and correct problem 3) Reanalyze blank and affected samples 4) Qualify data, if necessary	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-HHE Rev 6.0
GC/TCD	Laboratory Control Sample (LCS)	Once every analytical batch of 20 or fewer samples	Sum of all fixed gases in lab air within 90% and 110% (oxygen, nitrogen and carbon dioxide)	1) Reanalyze 2) Identify and correct the problem 3) Qualify data	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-HHE Rev 6.0
GC/TCD	Laboratory Duplicates	Once every analytical batch of 20 or fewer samples	Relative percent difference (RPD) within laboratory generated limits	1) Analyze third aliquot 2) Flag data if third aliquot is unacceptable	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-HHE Rev 6.0
GC/TCD	Holding Time	N/A	30 days (Canisters) 72 hours (Tedlar Bags)	Contact client and qualify data	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-HHE Rev 6.0

NA = not applicable

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

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Concentrating Trap	ICAL and CCV	ICAL and CCV	As needed indicated by calibration and QC difficulties.	Clean blank, sufficient sensitivity, and ICAL meets linearity criteria.	Routine maintenance includes periodic solvent cleaning of Silco steel lines in the valve oven if contamination is suspected. Also, periodic replacement of multi-sorbent or partial replacement of the trap if analyte specific deterioration is detected.	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-TO15, Rev.24
GC/MS	Column performance	ICAL and CCV	ICAL and CCV	Monitored by observing both peak shapes and column bleed.	Acceptable resolution and peak shape	Cut or replace column	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-TO15, Rev.24
GC/MS	Vacuum System / Pump Oil	ICAL and CCV	ICAL and CCV	Every six months, including changing the pump oil and checking the molecular sieve in the backstreaming trap.	Level of oil and quality is sufficient	Change oil	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-TO15, Rev.24

SAP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table (Continued)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Mass Selective Detector ion source cleaning	ICAL and CCV	ICAL and CCV	When tune difficulties or fluctuating internal standard areas are encountered.	Sufficient sensitivity and ICAL meets linearity criteria	Re-clean or replace source parts	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-TO15, Rev.24
GC/MS	Filament	ICAL and CCV	ICAL and CCV	As needed.	NA	Replace Filament	Dept. Supervisor, however other trained analysts in the team may be responsible	VOA-TO15, Rev.24

SAP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table (Continued)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC	Injection Port	NA	NA	As Needed	Passing QC	Replace septum and/or injection liner	Analyst	VOA-HHE Rev 6.0
GC	Column	NA	NA	Monitored by observing both peak shapes and column bleed	Passing QC	Reposition, trim or replace column	Analyst	VOA-HHE Rev 6.0
GC	Carrier Gas Purifier	NA	NA	As recommended by supplier	NA	NA	Analyst	VOA-HHE Rev 6.0
GC	Detector	NA	NA	As needed	NA	NA	Analyst	VOA-HHE Rev 6.0
GC	Major/Additional Service	NA	NA	Performed as needed by trained technician	NA	NA	Analyst	VOA-HHE Rev 6.0

NA = not applicable

SAP Worksheet #26 - Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Trihydro Field Technician
Sample Packaging (Personnel/Organization): Trihydro Field Technician
Coordination of Shipment (Personnel/Organization): Trihydro Field Technician
Type of Shipment/Carrier: Laboratory courier or commercial carrier
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): ALS Simi Valley Laboratory sample custodian
Sample Custody and Storage (Personnel/Organization): ALS Simi Valley Laboratory sample custodian
Sample Preparation (Personnel/Organization): ALS Simi Valley Laboratory sample preparation group
Sample Determinative Analysis (Personnel/Organization): ALS Simi Valley Laboratory bench chemist
SAMPLE ARCHIVING
Field Sample Storage (Number of days from sample collection): Samples from the reusable canisters are disposed after analysis and verification of results against lab and project requirements.
Sample Extract/Digestate Storage (Number of days from extraction/digestion): not applicable
Biological Sample Storage (Number of days from sample collection): not applicable
SAMPLE DISPOSAL
Personnel/Organization: ALS Simi Valley Laboratory sample custodian
Number of Days from Analysis: Samples from the reusable canisters are disposed after analysis and verification of results against lab and project requirements.

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SAP Worksheet #27 - Sample Custody Requirements

Sample Number

Samples submitted to the analytical laboratory will be uniquely numbered as shown in Worksheet #18. The field duplicate sample will be sequentially numbered and identified as sample OU2A8-SV-7 and the original sample corresponding to the field duplicate sample will be noted on the field forms and in the associated field logbook.

Sample Labeling

Sample labels/tags are necessary to prevent misidentification of samples. Sample labels/tags will be filled out in indelible ink and affixed to sample containers at the time of sample collection. At a minimum, each sample container will be labeled with the following:

- Sample ID number;
- Canister/flow controller identification numbers;
- Before and after canister pressure measurements;
- Sample collection date (month/day/year);
- Time of collection (24-hour clock) or start/stop/total time for time integrated samples;
- Sampler's initials; and
- Analyses required.

Sample Handling and Shipping

Immediately after samples are collected and labeled, each will be placed in a resealable plastic bag and then placed into a sample shipping box. Sampling will be shipped to the laboratory using a commercial carrier. If samples are collected on a weekend or a holiday, they will be stored and shipped off site the next business day.

Chain-of-Custody

To establish the documentation necessary to trace sample possession from the time of collection through analysis and disposal, a chain of custody record will be completely filled out and will accompany every sample. Samples will be delivered to the laboratory for analysis as soon as practicable.

A sample is considered to be in a person's custody if the following conditions have been observed:

- The item is in the actual possession of the person;
- The item is in the view of the person after being in actual possession of the person;
- The item is locked in a secure area;
- The item is placed in an area restricted to authorized personnel; or
- The item is placed in a container and secured with a tamper indicating seal, such that the sample cannot be reached without breaking the seal.

SAP Worksheet #27 - Sample Custody Requirements (Continued)

The chain of custody record will be the controlling document to ensure that the sample custody is maintained. Sampling personnel will initiate the chain of custody record in the field once a sample is collected. Each time the sample custody is transferred, the former custodian will sign the chain of custody on the "Relinquished By" line, and the new custodian will sign the chain of custody on the "Received By" line. The date, time, and the name of their project or company affiliation will accompany each signature. The waybill number and carrier company name will be recorded on the chain of custody for the commercial carrier used. The shipping container will be secured with two custody seals (one on the front and one on the side), thereby allowing for custody to be maintained by the shipping personnel until receipt by the laboratory.

Sample custody will be the responsibility of sampling personnel from the time of sample collection until the samples are accepted by the laboratory via commercial carrier. Thereafter, the laboratory performing the analysis will maintain custody.

Field Logbooks

A permanently bound field logbook with consecutively numbered pages, used for sampling activities only, will be assigned to this project. All entries will be recorded in indelible ink. At the end of each workday, the logbook pages will be signed by the responsible sampler, and any unused portions of the logbook page will be crossed out, signed, and dated.

If it is necessary to transfer the logbook to another person, the person relinquishing the logbook will crossed out unused portions of the last logbook page used and sign and date the page. The person receiving the logbook will sign and date the top of the next page to be used.

At a minimum, the logbook will contain the following information:

- Project name and location;
- Date and time;
- Personnel in attendance;
- General weather information;
- Work performed;
- Field observations;
- The name and association of any site visitors, the reason for the visit, and the time of arrival and departure;
- Sampling performed, including specifics such as location, type of sample, type of analyses, and sample ID;
- Field analyses performed, including results, instrument checks, problems, and calibration records for field instruments;
- Descriptions of deviations from this SAP;
- Problems encountered and corrective action taken;
- Identification of field QC samples;

SAP Worksheet #27 - Sample Custody Requirements (Continued)

- QC activities;
- Verbal or written instructions; and
- Any other events that may affect the samples.

Document Corrections

Changes or corrections on any project documentation will be made by crossing out the erroneous item with a single line and initialing (by the person performing the correction) and dating the correction. The original item, although erroneous, must remain legible beneath the cross-out line. The new information should be written clearly above the crossed-out item.

SAP Worksheet #28 - Laboratory QC Samples Tables

Matrix: Air

Analytical Group: VOCs

Analytical Method / SOP Reference: EPA TO-15 (Standard)

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method blank	Once every analytical batch of 20 or fewer samples	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected >LOQ.	1) Reanalyze blank 2) Identify and correct problem 3) Reanalyze blank and affected samples 4) Qualify data.	Dept. Supervisor, however other trained analysts in the team may be responsible	Bias	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.
Laboratory control sample (spiked with analytes of interest)	Once every analytical batch of 20 or fewer samples	See QC Limits for Soil Vapor Samples Table	1) Reanalyze 2) Identify and correct problem 3) Qualify data *DoD projects require corrective action for all exceedances	Dept. Supervisor, however other trained analysts in the team may be responsible	Accuracy	See QC Limits for Soil Vapor Samples Table

QC Limits for Soil Vapor Samples

Analyte	LCS/LCSD (%) Recovery	RPD (%)
1,1,2-Trichloroethane	73-119	25
1,1-Dichloroethene	61-133	25
1,4-Dioxane	71-122	25
Benzene	69-119	25
Carbon tetrachloride	69-132	25
cis-1,2-Dichloroethene	70-121	25
Ethylbenzene	70-124	25
Tetrachloroethene	66-124	25
trans-1,2-Dichloroethene	67-124	25
Trichloroethene	71-123	25
Vinyl chloride	64-127	25

SAP Worksheet #28 - Laboratory QC Samples Tables (Continued)

Matrix: Air

Analytical Group: Helium

Analytical Method / SOP Reference: EPA 3C modified

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method blank	Once every analytical batch of 20 or fewer samples	No analyte detected equal to or above the LOQ	1) Reanalyze blank 2) Identify and correct problem 3) Reanalyze blank and affected samples 4) Qualify data.	Analyst	Bias	No analytes detected less than half the reporting limit
Laboratory control sample	Once every analytical batch of 20 or fewer samples	Sum of all fixed gases in lab air within 90% and 110% (oxygen, nitrogen and carbon dioxide)	1) Reanalyze blank 2) Identify and correct problem 3) Qualify data *DoD projects require corrective action for all exceedances	Analyst	Accuracy	Sum of all fixed gases in lab air within 90% and 110% (oxygen, nitrogen and carbon dioxide)

SAP Worksheet #29 - Project Documents and Records Table

Document	Where Maintained
Planning documents (Work Plan, SAP, Health & Safety Plan)	Battelle project file NAVFAC Northwest Site File
Field forms/logbooks	Battelle project file NAVFAC Northwest Site File
Sample labels	ALS Simi Valley Laboratory
Chain of Custody Forms	Battelle project file and ALS Simi Valley Laboratory
Shipping records	Battelle project file
Field audit and nonconformance reports	Battelle project file
Laboratory data package including: Sample receipt and login Laboratory internal chain of custody Instrument calibration logs Sample preparation logs Sample analysis/run logs Nonconformance reports including corrective actions	ALS Simi Valley Laboratory and Battelle project file; NAVFAC Northwest Site File
Data validation report	LDC validator and Battelle project file NAVFAC Northwest Site File

SAP Worksheet #30 - Analytical Services Table

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method/ SOP Reference	Data Package Turnaround Time	Laboratory (Name and Address, Contact Person and Telephone Number)	Backup Laboratory (Name and Address, Contact Person and Telephone Number
Soil Vapor	VOCs	OU2A8-SV-1 through 6	U.S. EPA TO-15/VOA-TO15	10 business days	ALS Simi Valley Laboratory 2655 Park Center Drive, Suite A Simi Valley, CA 93065	TBD
Soil Vapor	Helium	OU2A8-SV-1 through 6	U.S. EPA 3C Modified/VOA-HHE	10 business days	ALS Simi Valley Laboratory 2655 Park Center Drive, Suite A Simi Valley, CA 93065	TBD

TBD = to be determined

SAP Worksheet #31 - Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing CA	Person(s) Responsible for Monitoring Effectiveness of CA
Field Readiness Review	Prior to mobilization of the project and prior to initiating major phases of work	Internal	Battelle	Project Manager	Field Lead	Field Lead	Project Manager
Field Sampling Surveillance	Once at the beginning of field sampling activities	Internal	Battelle	Field Lead	Project Manager	Project Manager	Field Lead
Field Documentation Review	Once at the end of sampling	Internal	Battelle	Field Lead	Project Manager	Project Manager	Field Lead
Data Review	Once for project	Internal	Battelle	Battelle QAO	Project Manager	Project Manager	Battelle QAO

SAP Worksheet #32 - Assessment Findings and Corrective Action Response Table

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response	Timeframe for Response
Field Sampling Surveillance	Surveillance Report	Project Manager and Battelle QAO	7 days after completion of the inspection	Corrective Action Report	Project Manager	5 days after notification
Data Review	Data Review Report	Project Manager	7 days after completion of the review	Corrective Action Report	Battelle QAO	14 days after notification

SAP Worksheet #33 - Quality Assurance Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation	Report Recipient(s)
Field Sampling Surveillance Report	One at start of sampling	14 days	Field Lead	Project Manager and Battelle QAO
Data Review Report	One after all data are generated and reviewed	14 days	Battelle QAO	Project Manager

SAP Worksheet #34 - Data Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification
Field logbook	Field logbooks will be reviewed and placed in the project file upon project completion.	Internal	Field Team Leader QAO (Battelle)
Chain-of-custody forms	Chain-of-custody forms will be reviewed for completeness. A copy of the forms will be retained in the project file, and the original and any remaining copies will be taped inside the container for shipment.	Internal	Field Team Leader
Sample logins	Sample login information will be reviewed and verified for completeness in accordance with the chain-of-custody forms. Battelle QAO will review Sample login information provided by the laboratory after sample login to verify correct analyses requested and logged in.	Internal External	Laboratory Project Manager (ALS) QAO (Battelle)
Holding Times	Verify that samples were analyzed within holding times specified in methods and this SAP. If holding times were not met, then confirm that deviations were documented and that appropriate notifications were made by the laboratory. Will be performed by examining the laboratory data package after generation of the package but prior to release to the third-party validator or Battelle.	Internal	Analyst-primary, supervisor or peer- secondary (ALS)
Analytes	Verify that required lists of analytes were reported as specified on the chain-of-custody form. Will be performed by examining the laboratory data package after generation of the package but prior to release to the third-party validator or Battelle. Verification will be documented in the final report.	Internal	Analyst-primary, supervisor or peer- secondary (ALS)
Analytical Methods and Procedures	Verify that the required analytical methods were used and that any deviations were noted. Verify that the quality control samples met performance criteria and that any deviations were documented. Will be performed by examining the laboratory data package after generation of the package but prior to release to the third-party validator or Battelle. Verification will be documented in the final report.	Internal	Analyst-primary, supervisor or peer- secondary (ALS)

SAP Worksheet #34 - Data Verification (Step I) Process Table (Continued)

Data Flags	Verify that the laboratory data flags were defined in the laboratory data package and applied as specified. Will be performed by examining the laboratory data package after generation of the package but prior to release to the third-party validator or Battelle. Verification will be documented in the final report.	Internal	Analyst-primary, supervisor or peer- secondary (ALS)
Laboratory data prior to release	Laboratory data will be reviewed and verified for completeness against analyses requested on the chain-of-custody forms.	Internal	Laboratory Project Manager (ALS),
Laboratory Data Packages	All laboratory data packages will be verified by the laboratory performing the work for completeness and technical accuracy prior to submittal. Issues identified by the laboratory will then be reviewed by the Battelle QAO. Subsequently, data packages will be evaluated externally according to the data validation procedures specified in Worksheet #36 of this SAP.	Internal External External	Laboratory Project Manager (ALS) QAO (Battelle) Third-party Data Validator (LDC)
EDD	All EDDs will be verified by the laboratory performing the work for completeness and technical accuracy prior to submittal. All received EDDs will be verified against the hard copy laboratory data packages.	Internal External	Laboratory Project Manager (ALS) QAO (Battelle)

SAP Worksheet #35 - Data Validation (Steps IIa and IIb) Process Table

Step IIa/IIb	Validation Input	Description	Responsible for Validation
IIa	Sampling Methods and Procedures	Establish that the required sampling methods were used and that any deviations were noted. Determine whether the sampling procedures and field measurements met performance criteria and that any deviations were documented. Will be performed during site surveillance when samples are being collected in the field.	Field Team Leader (Battelle)
IIa	Holding Times	Determine whether samples were analyzed within holding times specified in methods and this SAP. If holding times were not met, then confirm that deviations were documented in the laboratory case narrative.	Third-party Data Validator (LDC)
IIa	Analytes	Determine whether required lists of analytes were reported as specified on the chain-of-custody form. Will be performed by examining the laboratory data package. Validation will be documented in the data validation report.	Third-party Data Validator (LDC)
IIa	Analytical Methods and Procedures	Determine whether the required analytical methods were used and that any deviations were noted. Verify that the quality control samples met performance criteria and that any deviations were documented. Will be performed by examining the laboratory data package. Validation will be documented in the data validation report.	Third-party Data Validator (LDC)
IIa	Data Flags	Determine whether the laboratory data flags were defined in the laboratory data package and applied as specified. Will be performed by examining the laboratory data package. Validation will be documented in the data validation report.	Third-party Data Validator (LDC)
IIb	Sampling Plan	Determine whether the sampling plan was executed as specified (the number, location, etc.) and the type of field samples were collected and analyzed as specified in the SAP. Will be performed by examining the laboratory data package. Validation will be documented in the data validation report.	QAO (Battelle)
IIb	Sampling Procedures	Evaluate whether sampling procedures were followed with respect to equipment and proper sampling methods (e.g., techniques, equipment, decontamination, volume, temperature, preservation). Will be performed during inspection when samples are being collected in the field. Verification will be documented in the final report.	Field Team Leader (Battelle)
IIa/IIb	Field Duplicates	Compare results of field duplicates with criteria established in the SAP. Will be performed after receipt of the data packages by examining the laboratory results and comparing relative percent difference values to project criteria in Worksheet #12. Verification will be documented in the final report.	QAO (Battelle) Third-party validator (LDC)

SAP Worksheet #35 - Data Validation (Steps IIa and IIb) Process Table (Continued)

Step IIa/IIb	Validation Input	Description	Responsible for Validation
IIa/IIb	Project LOQs	Determine whether LOQs were achieved as outlined in the SAP. Will be performed after receipt of the data packages by examining the laboratory results and comparing achieved LOQs to project criteria in Worksheet #15. Verification will be documented in the final report.	Third-party Data Validator (LDC)
IIa/IIb	Performance Criteria	Evaluate QC data against performance criteria in the SAP. Will be performed after receipt of the data packages by examining the laboratory data and comparing to project criteria in Worksheets #12 and #28. Verification will be documented in the final report.	Third-party Data Validator (LDC)

SAP Worksheet #36 - Data Validation (Steps IIa and IIb) Summary Table

Step IIa / IIb [a]	Matrix	Analytical Group	Validation Criteria [b]	Data Validator
IIa / IIb	Soil Vapor	VOCs	In accordance with SOP VOA-TO15, NAVFAC Northwest Series II SOPs (2015) and EPA National Functional Guidelines for Organics (EPA, 2017a).	Third-party Data Validator
IIa / IIb	Soil Vapor	Helium	In accordance with SOP VOA-HHe, NAVFAC Northwest Series II SOPs (2015) and EPA National Functional Guidelines for Inorganics (EPA, 2017b).	

[b] Analytical Laboratory SOPs provided in Attachment C.

SAP Worksheet #37 - Usability Assessment

Data Quality Assessment

Data validation reports will be reviewed and assessed for meeting data quality objectives. The Battelle QAO will review the data validation reports for any deviations and to ensure that the third-party reviewer has assigned data qualifiers. The following data qualifiers will be used:

J = Result is estimated

U = Analyte is not detected at or above the stated LOQ

R = Data are rejected

UJ = Analyte is not detected, but there is an uncertainty about the LOQ

Data qualifiers are used to indicate uncertainties associated with the data. The assigned qualifiers will be entered into the validation code field in the database. The Battelle QAO will prepare a data quality assessment report that will summarize the findings of the data assessment and discuss usability of the data to be included in the report.

Data will be reported in tabular format to be included in the report. The electronic data in the NIRIS Naval electronic data deliverable (NEDD) format will be submitted to the DON as described in the *NEDD Standard Operating Procedure* (DON NIRIS Working Group, 2013). An e-mail confirmation will be saved in the project file.

Measurement Quality Objective for Chemical Data

The primary measurement quality objective for the monitoring program relates to the precision and accuracy, including detections and LOQs, for the analytical methods performed. All analytical results will be evaluated in accordance with PARCCS parameters to document the quality of the data and to ensure that the data are of sufficient quality to meet the project objectives. The following subsections describe each of the PARCCS parameters and how they will be assessed within this project.

Precision

Precision is the degree of mutual agreement between individual measurements of the same property under similar conditions and expressed as RPD. Laboratory analytical precision is determined by analyzing laboratory duplicates or LCSs in duplicate, and calculating the RPD between the duplicate results. Field analytical precision is determined by analyzing field duplicates where samples are collected simultaneously (if possible) and is a measurement of the precision of both sample collection and handling and analytical procedures. RPD is calculated as follows:

$$\text{RPD} = \frac{|\text{Sample 1 result} - \text{Sample 2 result}|}{\left(\frac{\text{Sample 1 result} + \text{Sample 2 result}}{2}\right)} \times 100\%$$

Field and laboratory measurement performance criteria are provided in Worksheets #12.

SAP Worksheet #37 - Usability Assessment (Continued)

Accuracy

A program of sample spiking will be conducted to evaluate laboratory accuracy. This program includes analysis of LCSs or blank spikes, surrogate standards, and method blanks. LCSs are analyzed at a frequency of 5 percent (1 in 20 project samples). Surrogate standards, as applicable to the method, are added to every sample analyzed for organic constituents. The results of the spiked samples are used to calculate the percent recovery for evaluating accuracy.

$$\%\ \mathrm{R} = \frac{\text{Spiked sample result}}{\text{True concentration}} \times 100\%$$

Results that fall outside the accuracy goals will be further evaluated based on the results of other QC samples. Refer to tables in Worksheet #28 for accuracy limits for each method.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent the characteristics of a population, variations in a parameter at a sampling point, or an environmental condition that they are intended to represent. Representative data will be obtained for this project through careful selection of sampling locations and analytical parameters. Representative data will also be obtained through proper collection and handling of samples to avoid interference and minimize contamination.

Representativeness of data will also be ensured through the consistent application of established field and laboratory procedures. Laboratory blank samples will be evaluated for the presence of contaminants to aid in evaluating the representativeness of sample results. Data determined to be nonrepresentative, by comparison with existing data, will be used only if accompanied by appropriate qualifiers and limits of uncertainty.

Completeness

Completeness is a measure of the percentage of project-specific data that are considered valid. Valid data are obtained when samples are collected and analyzed in accordance with QC procedures outlined in this SAP, and when none of the QC criteria that affect data usability is exceeded to the extent that data are rejected (e.g., data qualified as estimated [J qualified] are included in the usable data). When all data validation is completed, the percent completeness value will be calculated by dividing the number of useable sample results (i.e., not rejected [R-qualified] data) by the total number of sample results generated for this investigation.

$$\%\ \text{completeness} = \frac{\text{number of valid analyte results}}{\text{number of expected results}} \times 100\%$$

Completeness will also be evaluated as part of the data quality assessment process against project-specific requirements. This evaluation will help determine whether any limitations are associated with the decisions to be made based on the data collected. The completeness goal for the VI project data is 90 percent.

SAP Worksheet #37 - Usability Assessment (Continued)

Comparability

Comparability expresses the confidence with which one data set can be compared with another. Comparability of data will be achieved by consistently following standard field and laboratory procedures and by using standard measurement units in reporting analytical data.

Sensitivity

Sensitivity assesses the ability of the laboratory to detect target analytes using the methods and instruments selected for this project. Worksheet #15 lists the project action limits and laboratory DLs. Based on laboratory DL studies, the EPA methods and laboratory instrumentation have been selected for use on this project in order to detect target analytes at the project action limits.

Usability Assessment

A usability assessment to determine how well the data collected support the project objectives and decisions to be made will be performed by the project team. Any deviations from proposed field activities will be reviewed, and their effect on data usability evaluated. The analytical results will be compared to the DQIs presented on Worksheets #12 and #28 to determine whether the measurement performance criteria were met. Upon completion of the verification and validation processes noted on Worksheet #34 and Worksheet #35, the DQIs will be evaluated for each analytical group. Based on the results of this examination, conclusions regarding the validity and usability of data for each analytical group will be drawn and a data quality assessment report will be prepared. The report will include discussions of conclusions drawn and any limitations on the use of project data as a result of this assessment and will be included in a final report for this phase of the project.

REFERENCES

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FIGURES

Legend

- Former Buildings
- Tanks
- Soil Vapor Location (Proposed)
- Monitoring Well
- Spring-Seep
- Manhole
- Water Valve
- Electrical Structure (underground)
- Gas Valve

Main Utility Lines

- Distribution Main
- Hydrant Lateral
- Transmission Main
- Force Main (old South Pier)
- Electrical General
- Gas Service Line
- Water Service Line
- Storm Drain

TCE Isoconcentration contours

- Inferred Remediation Goal Contour
- Inferred Contour
- Contour

N

Feet
0
50
100

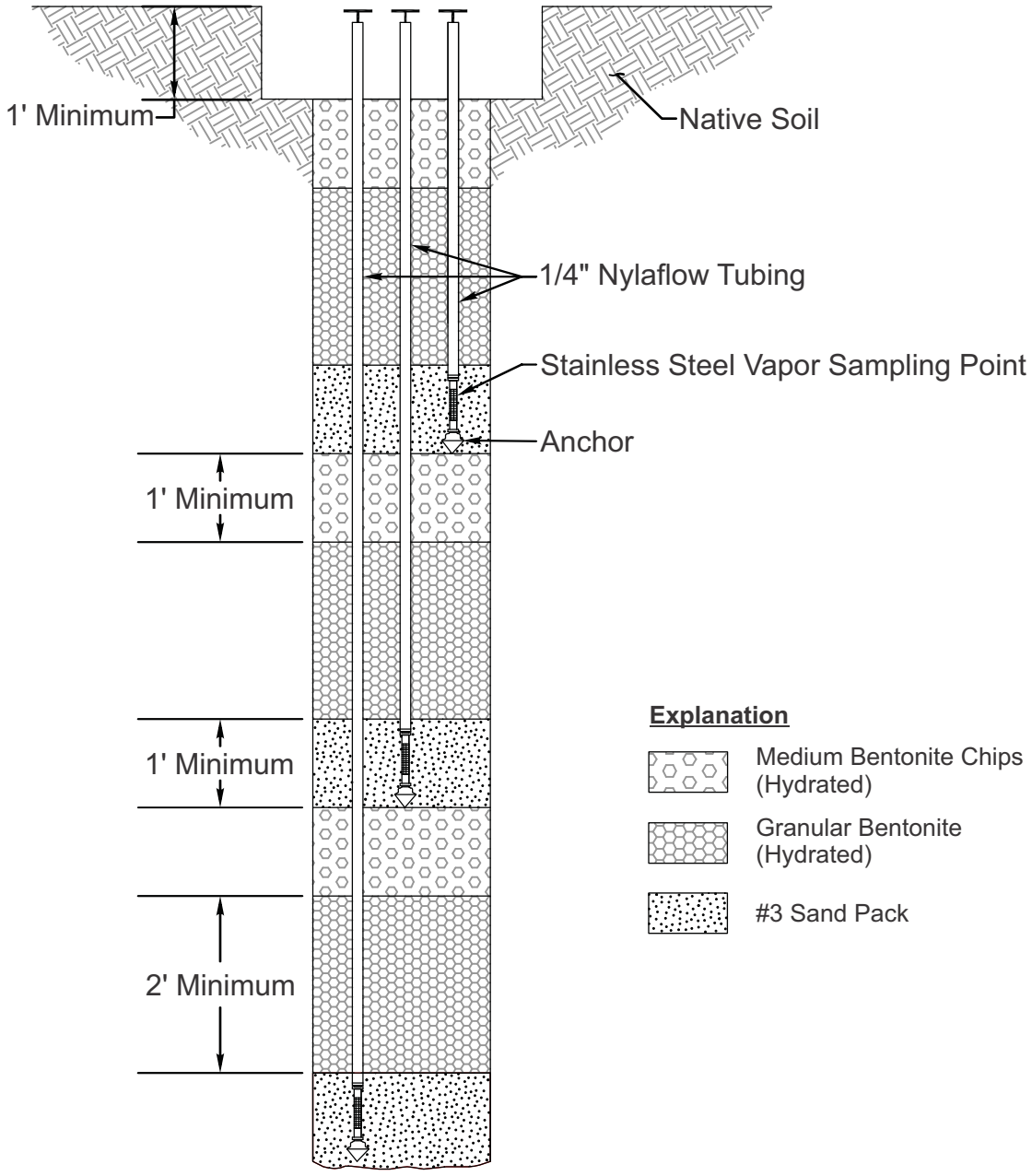
BATTELLE

Figure 1
OU 2 Area 8 Location Map
and Site Plan View

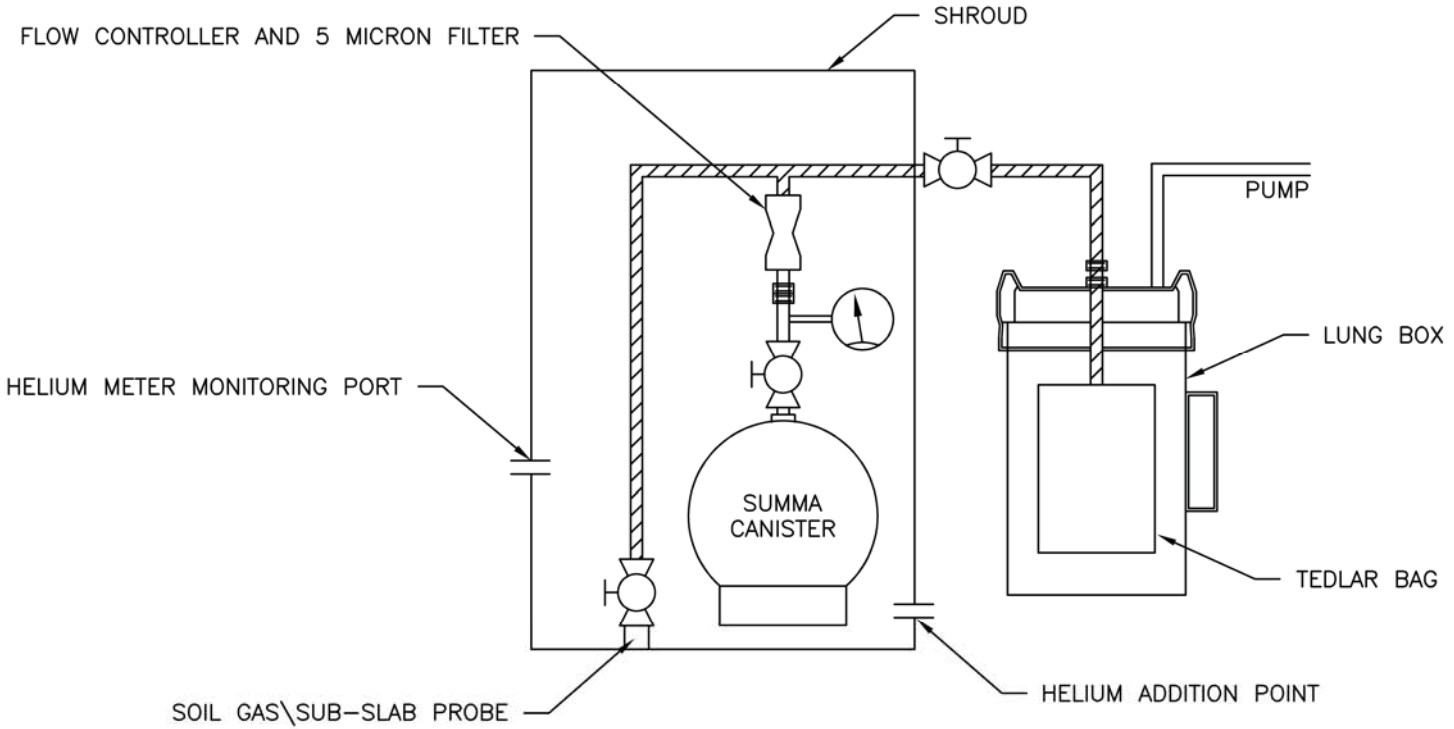
Keyport Naval Undersea
Warfare Center

DATE: 10/25/2017 ANALYST: HICKSJ
REV. 0 APPROVED: MEYER

FILE: C:\GIS\Keyport\Maps\OU2_Area_8_r04.mxd



SOURCE Trihydro 2011	BATTELLE		
DRAWN BY VS	**Figure 2. Typical Temporary Nested Soil Vapor Well**		
CHECKED BY MM	NUWC DIVISION KEYPORT, WA		
	PROJECT 100100157-026NW21	FILE NESTED SOIL VAPOR WELL.CDR	DATE 06/17



NOTE:
ACTUAL SAMPLE TRAIN MAY BE SLIGHTLY MODIFIED IN THE FIELD.

SOURCE Trihydro 2012	BATTELLE		
DRAWN BY VS	**Figure 3. General Soil Gas Sampling Train**		
CHECKED BY MM	FORMER SAFETY-KLEEN SYSTEMS, INC. RECYCLE CENTER – REEDLEY, CA		
	PROJECT 100100157-026NW21	FILE SOIL SAMPLING TRAIN.CDR	DATE 07/17

ATTACHMENT A

RESPONSES TO COMMENTS

Document Title:
DRAFT Sampling and Analysis Plan2017 Keyport Operable Unit (OU) 2, Area 8 Vapor Intrusion (VI) Study, Naval Base Kitsap (NBK) Keyport

Document dated: September 6, 2017

Comments from: Mahbub Alam, Ecology PM
Comments dated: October 9, 2017

#	Page No./ Line No.	Comment	Proposed Response	Response Accepted?
0	General	Washington State Department of Ecology (Ecology) appreciates the opportunity to review and comment on the above referenced document. The sampling and analysis plan (SAP) for the Area 8 vapor intrusion (VI) study is written very well. It is organized and contains all relevant information for a successful study. However, Ecology would like to see couple of more sample locations based on of the following rationale. Ecology generally recommends collecting soil gas samples right above the subsurface contamination (VI source) which often display less spatial viability. Since samples are collected away from the VI source, a more dense sampling design is necessary to account for spatial variability. Given the site condition (significant number of utility lines, mostly paved surfaces), there are greater likelihood of having preferential pathways and having more sampling locations will add confidence to the measured concentration levels. Please see the attached Figure for 3 more proposed sampling locations. Ecology would like to emphasize the sampling location near the north-east corner of the site (Southeast corner of the building in the force main trench). MW 8-11 show increased TCE and 1,4 Dioxane results. The long term TCE data suggests the plume may have shifted towards northerly direction.	The Navy appreciates Ecology's thoughtful comments. We can see your rationale for increased sample location density. We note, however, that the existing buildings are located relatively far from a low-concentration chlorinated solvent plume moving away from the buildings. The locations proposed by the Navy are near the buildings and near preferential pathways, between the plume and the buildings. We note that the two sample depths proposed at each location provide vertical data density to supplement the horizontal data density. While the Navy is amenable to adjusting the position of the originally proposed locations, we believe that the proposed data density is sufficient and request that Ecology reconsider requesting additional locations.	Sample locations will be moved per discussions at the comment resolution meeting to the points shown on the attached map.
1a	Page 29, SAP worksheet #15	Add a new column or add to the PAL reference column whether the PAL is based on cancer or non-cancer basis.	We will add footnotes to identify the basis of each PAL as cancer or non-cancer.	Yes.

Document Title:
DRAFT Sampling and Analysis Plan2017 Keyport Operable Unit (OU) 2, Area 8 Vapor Intrusion (VI) Study, Naval Base Kitsap (NBK) Keyport

Document dated: September 6, 2017

#	Page No./ Line No.	Comment	Proposed Response	Response Accepted?
1b		The Table showed the PAL for 1,4-Dioxane as 18.7 ug/m3. Explain how this number was derived with source reference. Soil gas screening level for this chemical was not calculated in the Table B-1 of the Draft Ecology VI guidance. However, The air cleanup level for 1,4-Dioxane by Method B cancer is 0.5 ug/m3 (See CLARC Table "Air – Method B" https://fortress.wa.gov/ecy/clarc/FocusSheets/Air%20Method%20B.pdf . Using the 0.03 attenuation factor as provided in the footnote of the SAP worksheet, the PAL becomes 16.7 ug/m3. Ecology recommends to use this value as the PAL.	The PALs are based on Ecology's vapor intrusion screening Table B-1 updated April 6, 2015. Based on additional consideration of the receptors at the site, the Navy realized that the original PALs proposed in the QAPP were based on residential exposure. Given that this is an industrial site with land use controls (LUCs) that prevent residential use, the Navy proposes to update the PALs to reflect MTCA Method C values, as presented in Ecology's VI screening Table B-1. The air cleanup value for 1,4-dioxane in the CLARC Table "Air-Method C" is 5.0 µg/m^3. Using the 0.03 attenuation factor as provided in the footnote of the SAP worksheet, the PAL becomes 167 ug/m3. As stated above, the site is covered by a institutional control plan with LUCs that limit the site to industrial use only, the Navy feels that the use of MTCA Method C is appropriate at this time.	Yes.

Document Title:
DRAFT Sampling and Analysis Plan2017 Keyport Operable Unit (OU) 2, Area 8 Vapor Intrusion (VI) Study, Naval Base Kitsap (NBK) Keyport

Document dated: September 6, 2017

#	Page No./ Line No.	Comment	Proposed Response	Response Accepted?
1c		Screening level for trans-1,2-Dichloroethene was not established in the SAP worksheet. The air cleanup level for trans-1,2-Dichloroethene by Method B non cancer is 27.4 ug/m3 (See CLARC Table "Air – Method B" https://fortress.wa.gov/ecy/clarc/FocusSheets/Air%20Method%20B.pdf . Using the 0.03 attenuation factor, the screening level becomes 913 ug/m3. Ecology recommends to use this value as the PAL.	We relied on Table B-1 of the Draft Ecology VI guidance, which does not establish a value screening value for trans-1,2-DCE. We also see that Ecology removed the inhalation value from CLARC in August 2015. However, we propose to revise the PAL to the non-cancer value proposed by Ecology, adjusted to Method C (9,130 ug/m^3), based on the industrial use of the site and the existing LUCs, as mentioned in the response to Comment 1b above.	Method C level for trans-1,2-dichloroethene will be changed to 2000 ug/m3 based on recalculation of the Method C level and the applicable attenuation factor.
1d		Remove PAL reference of Method B for cis-1,2-Dichloroethene as no screening level was established using method B.	We will remove the PAL as requested.	Yes.
1e		Explain how the project QL goal was established. In most cases, it seems it was determined by a factor of 2 from the PAL. Note project QL for carbon tetrachloride does not make sense as it is higher than PAL when LOQ is lower than the PAL. Ecology recommends using a higher factor (e.g. 5 to 10) whenever possible to establish the project QL as this provides better protection of the PAL. Sometimes a higher factor is not feasible as that can make the project QL lower than LOQ. In that case, LOQ should be used as the project QL.	We will revise the project QL goal to be 1/5 of the PAL wherever possible, and default to the LOQ if this target is not achievable.	Will be based on 1/5 of Method B values to allow for future comparison, if land use changes.
2	Page 32, SAP Worksheet #17	What is the diameter of the 5 feet long drive rod? Figure 2 illustrates nested soil vapor well. Does this mean there will be no separate boreholes for different target depths?	The outside diameter of the rod will be 2.25 inches. Each soil gas well is anticipated to have two nested sample points at discrete depths within the same boring. In the event that completion integrity of nested soil gas wells is compromised, separate, but closely spaced borings may be used to target different depths.	Yes.

Document Title:
DRAFT Sampling and Analysis Plan2017 Keyport Operable Unit (OU) 2, Area 8 Vapor Intrusion (VI) Study, Naval Base Kitsap (NBK) Keyport

Document dated: September 6, 2017

#	Page No./ Line No.	Comment	Proposed Response	Response Accepted?
3	Page 37, SAP Worksheet #20	Since the number of proposed sampling locations are 12 (not including Ecology's additional proposed ones), it is recommended to have two field duplicates per the QC criteria of 1 duplicate per 10 samples. In addition, since soil gas sampling data show significant spatial variability, more duplicate sampling is necessary to better understand the precision of sampling.	The Navy agrees that the standard QC criterion for field duplicates is one per 10 samples, and therefore the Navy proposes to update the worksheet to include two field duplicates.	Yes.
4	Page 67-68, SAP Worksheet #37	The last line of the precision paragraph noted laboratory measurement performance criteria for precision are provided in worksheet #28. Worksheet #28 measures accuracy and bias not precision. Show which worksheet measures laboratory analytical precision. For Accuracy, percent recovery (%R) was stated as the measurement parameter. However, worksheet #28 noted "Appendix B, DoD QSM v5 Limits" as the QC acceptance criteria. What are the %R criteria? Reproduce the criteria in the worksheet. Also, state clearly if blank or LCS samples will be spiked. Are there any MS/MSD samples?	The last line of the precision paragraph as cited in the comment will be revised to cite WS#12 for precision criteria. Appendix B, DoD QSM v5 Limits will be added to WS #28 (VOCs) as the QC acceptance criteria. A statement will be added to WS #28 to indicate LCS samples will be spiked. The methods for soil vapor analysis do not require MS/MSD samples; however, the methods require laboratory duplicates. Laboratory duplicates and their acceptance criteria will be added to WS #28.	Yes.

ATTACHMENT B

LABORATORY CERTIFICATES OF ACCREDITATION

PERRY JOHNSON LABORATORY ACCREDITATION, INC.

Certificate of Accreditation

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

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065

(Hereinafter called the Organization) and hereby declares that Organization has met the requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the DoD Quality Systems Manual for Environmental Laboratories Version 5.0 July 2013 and is accredited is accordance with the:

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

This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system
(as outlined by the joint ISO-ILAC-IAF Communiqué dated January 2009):

This accreditation demonstrates technical competence for the defined scope:

Environmental Testing

(As detailed in the supplement)

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

For PJLA:

Tracy Szerszen
President/Operations Manager

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

Initial Accreditation Date:	*Issue Date:*	*Expiration Date:*
January 11, 2010	December 3, 2015	February 28, 2018

Accreditation No:	*Certificate No:*
65818	L15-398

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

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

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

Matrix	Standard Method	Technology	Analyte
Air	ASTM D 1946-90	GC/TCD	Carbon Dioxide
Air	ASTM D 1946-90	GC/TCD	Carbon Monoxide
Air	ASTM D 1946-90	GC/TCD	Hydrogen
Air	ASTM D 1946-90	GC/TCD	Methane
Air	ASTM D 1946-90	GC/TCD	Nitrogen
Air	ASTM D 1946-90	GC/TCD	Oxygen
Air	EPA 3C	GC/TCD	Carbon Dioxide
Air	EPA 3C	GC/TCD	Methane
Air	EPA 3C	GC/TCD	Nitrogen
Air	EPA 3C	GC/TCD	Oxygen
Air	EPA TO-15	GC/MS	1,1,1-Trichloroethane
Air	EPA TO-15	GC/MS	1,1,2,2-Tetrachloroethane
Air	EPA TO-15	GC/MS	1,1,2-Trichloroethane
Air	EPA TO-15	GC/MS	1,1-Dichloroethane
Air	EPA TO-15	GC/MS	1,1-Dichloroethene
Air	EPA TO-15	GC/MS	1,2,3-Trimethylbenzene
Air	EPA TO-15	GC/MS	1,2,4-Trichlorobenzene
Air	EPA TO-15	GC/MS	1,2,4-Trimethylbenzene
Air	EPA TO-15	GC/MS	1,2,Dibromo-3-Chloropropane
Air	EPA TO-15	GC/MS	1,2-Dibromoethane
Air	EPA TO-15	GC/MS	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)
Air	EPA TO-15	GC/MS	1,2-Dichlorobenzene
Air	EPA TO-15	GC/MS	1,2-Dichloroethane
Air	EPA TO-15	GC/MS	1,2-Dichloropropane
Air	EPA TO-15	GC/MS	1,3,5-Trimethylbenzene
Air	EPA TO-15	GC/MS	1,3-Butadiene
Air	EPA TO-15	GC/MS	1,3-Dichlorobenzene
Air	EPA TO-15	GC/MS	1,4-Dichlorobenzene
Air	EPA TO-15	GC/MS	1,4-Dioxane
Air	EPA TO-15	GC/MS	1-Butanol
Air	EPA TO-15	GC/MS	2-Butanone (MEK)
Air	EPA TO-15	GC/MS	2-Hexanone
Air	EPA TO-15	GC/MS	3-Ethyltoluene
Air	EPA TO-15	GC/MS	4-Ethyltoluene
Air	EPA TO-15	GC/MS	4-Methyl-2-Pentanone
Air	EPA TO-15	GC/MS	Acetone

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Matrix	Standard Method	Technology	Analyte
Air	EPA TO-15	GC/MS	Acetonitrile
Air	EPA TO-15	GC/MS	Acrolein
Air	EPA TO-15	GC/MS	Acrylonitrile
Air	EPA TO-15	GC/MS	Allyl Chloride
Air	EPA TO-15	GC/MS	alpha-Methylstyrene
Air	EPA TO-15	GC/MS	alpha-Pinene
Air	EPA TO-15	GC/MS	Benzene
Air	EPA TO-15	GC/MS	Benzyl Chloride
Air	EPA TO-15	GC/MS	Bromodichloromethane
Air	EPA TO-15	GC/MS	Bromoform
Air	EPA TO-15	GC/MS	Bromomethane
Air	EPA TO-15	GC/MS	Carbon Disulfide
Air	EPA TO-15	GC/MS	Carbon Tetrachloride
Air	EPA TO-15	GC/MS	Chlorobenzene
Air	EPA TO-15	GC/MS	Chloroethane
Air	EPA TO-15	GC/MS	Chloroform
Air	EPA TO-15	GC/MS	Chloromethane
Air	EPA TO-15	GC/MS	cis-1,2-Dichloroethene
Air	EPA TO-15	GC/MS	cis-1,3-Dichloropropene
Air	EPA TO-15	GC/MS	Cumene
Air	EPA TO-15	GC/MS	Cyclohexane
Air	EPA TO-15	GC/MS	Cyclohexanone
Air	EPA TO-15	GC/MS	Dibromochloromethane
Air	EPA TO-15	GC/MS	Dichlorodifluoromethane (CFC 12)
Air	EPA TO-15	GC/MS	Diisopropyl Ether
Air	EPA TO-15	GC/MS	d-Limonene
Air	EPA TO-15	GC/MS	Ethanol
Air	EPA TO-15	GC/MS	Ethyl Acetate
Air	EPA TO-15	GC/MS	Ethyl tert-Butyl Ether
Air	EPA TO-15	GC/MS	Ethylbenzene
Air	EPA TO-15	GC/MS	Hexachlorobutadiene
Air	EPA TO-15	GC/MS	Isooctane
Air	EPA TO-15	GC/MS	Isopropyl acetate
Air	EPA TO-15	GC/MS	Isopropyl Alcohol
Air	EPA TO-15	GC/MS	m-&, p-Xylenes
Air	EPA TO-15	GC/MS	Methyl Methacrylate

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

Matrix	Standard Method	Technology	Analyte
Air	EPA TO-15	GC/MS	Methyl tert-Butyl Ether
Air	EPA TO-15	GC/MS	Methylene Chloride
Air	EPA TO-15	GC/MS	Naphthalene
Air	EPA TO-15	GC/MS	n-Butyl Acetate
Air	EPA TO-15	GC/MS	n-Butylbenzene
Air	EPA TO-15	GC/MS	n-Decane
Air	EPA TO-15	GC/MS	n-Dodecane
Air	EPA TO-15	GC/MS	n-Heptane
Air	EPA TO-15	GC/MS	n-Hexane
Air	EPA TO-15	GC/MS	n-Nonane
Air	EPA TO-15	GC/MS	o-Xylene
Air	EPA TO-15	GC/MS	sec-Butylbenzene
Air	EPA TO-15	GC/MS	tert-Amyl Methyl Ether
Air	EPA TO-15	GC/MS	tert-Butanol
Air	EPA TO-15	GC/MS	Tetrachloroethene
Air	EPA TO-15	GC/MS	trans-1,3-Dichloropropene
Air	EPA TO-15	GC/MS	Trichlorotrifluoroethane
Air	EPA TO-15	GC/MS	Vinyl Chloride
Air	EPA TO-15	GC/MS	2-Ethyltoluene
Air	EPA TO-15	GC/MS	n-Octane
Air	EPA TO-15	GC/MS	n-Propylbenzene
Air	EPA TO-15	GC/MS	n-Undecane
Air	EPA TO-15	GC/MS	p-Isopropyltoluene
Air	EPA TO-15	GC/MS	Propene
Air	EPA TO-15	GC/MS	Styrene
Air	EPA TO-15	GC/MS	tert-Butylbenzene
Air	EPA TO-15	GC/MS	Tetrahydrofuran
Air	EPA TO-15	GC/MS	Toluene
Air	EPA TO-15	GC/MS	trans-1,2-Dichloroethene
Air	EPA TO-15	GC/MS	Trichloroethene
Air	EPA TO-15	GC/MS	Trichlorofluoromethane
Air	EPA TO-15	GC/MS	Vinyl Acetate
Air	Simi Valley SOP VOA-EPA3C	GC/TCD	Carbon Dioxide
Air	Simi Valley SOP VOA-EPA 3C	GC/TCD	Carbon Monoxide
Air	Simi Valley SOP VOA-EPA 3C	GC/TCD	Hydrogen
Air	Simi Valley SOP VOA-EPA 3C	GC/TCD	Methane

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ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

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

Matrix	Standard Method	Technology	Analyte
Air	Simi Valley SOP VOA-EPA 3C	GC/TCD	Nitrogen
Air	Simi Valley SOP VOA-EPA 3C	GC/TCD	Oxygen
Air	Simi Valley SOP VOA TO-15	GC/MS	1,1,1-Trichloroethane
Air	Simi Valley SOP VOA TO-15	GC/MS	1,1,2,2-Tetrachloroethane
Air	Simi Valley SOP VOA TO-15	GC/MS	1,1,2-Trichloroethane
Air	Simi Valley SOP VOA TO-15	GC/MS	1,1-Dichloroethane
Air	Simi Valley SOP VOA TO-15	GC/MS	1,1-Dichloroethene
Air	Simi Valley SOP VOA TO-15	GC/MS	1,2,3-Trimethylbenzene
Air	Simi Valley SOP VOA TO-15	GC/MS	1,2,4-Trichlorobenzene
Air	Simi Valley SOP VOA TO-15	GC/MS	1,2,4-Trimethylbenzene
Air	Simi Valley SOP VOA TO-15	GC/MS	1,2-Dibromo-3-Chloropropane
Air	Simi Valley SOP VOA TO-15	GC/MS	1,2-Dibromoethane
Air	Simi Valley SOP VOA TO-15	GC/MS	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)
Air	Simi Valley SOP VOA TO-15	GC/MS	1,2-Dichlorobenzene
Air	Simi Valley SOP VOA TO-15	GC/MS	1,2-Dichloroethane
Air	Simi Valley SOP VOA TO-15	GC/MS	1,2-Dichloropropane
Air	Simi Valley SOP VOA TO-15	GC/MS	1,3,5-Trimethylbenzene
Air	Simi Valley SOP VOA TO-15	GC/MS	1,3-Butadiene
Air	Simi Valley SOP VOA TO-15	GC/MS	1,3-Dichlorobenzene
Air	Simi Valley SOP VOA TO-15	GC/MS	1,4-Dichlorobenzene
Air	Simi Valley SOP VOA TO-15	GC/MS	1,4-Dioxane
Air	Simi Valley SOP VOA TO-15	GC/MS	1-Butanol
Air	Simi Valley SOP VOA TO-15	GC/MS	2-Butanone (MEK)
Air	Simi Valley SOP VOA TO-15	GC/MS	2-Ethyltoluene
Air	Simi Valley SOP VOA TO-15	GC/MS	2-Hexanone
Air	Simi Valley SOP VOA TO-15	GC/MS	3-Ethyltoluene
Air	Simi Valley SOP VOA TO-15	GC/MS	4-Ethyltoluene
Air	Simi Valley SOP VOA TO-15	GC/MS	4-Methyl-2-Pentanone
Air	Simi Valley SOP VOA TO-15	GC/MS	Acetone
Air	Simi Valley SOP VOA TO-15	GC/MS	Acetonitrile
Air	Simi Valley SOP VOA TO-15	GC/MS	Acrolein
Air	Simi Valley SOP VOA TO-15	GC/MS	Acrylonitrile
Air	Simi Valley SOP VOA TO-15	GC/MS	Allyl Chloride
Air	Simi Valley SOP VOA TO-15	GC/MS	alpha-Methylstyrene
Air	Simi Valley SOP VOA TO-15	GC/MS	alpha-Pinene

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

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

Matrix	Standard Method	Technology	Analyte
Air	Simi Valley SOP VOA TO-15	GC/MS	Benzene
Air	Simi Valley SOP VOA TO-15	GC/MS	Benzyl Chloride
Air	Simi Valley SOP VOA TO-15	GC/MS	Bromodichloromethane
Air	Simi Valley SOP VOA TO-15	GC/MS	Bromoform
Air	Simi Valley SOP VOA TO-15	GC/MS	Bromomethane
Air	Simi Valley SOP VOA TO-15	GC/MS	Carbon Disulfide
Air	Simi Valley SOP VOA TO-15	GC/MS	Carbon Tetrachloride
Air	Simi Valley SOP VOA TO-15	GC/MS	Chlorobenzene
Air	Simi Valley SOP VOA TO-15	GC/MS	Chloroethane
Air	Simi Valley SOP VOA TO-15	GC/MS	Chloroform
Air	Simi Valley SOP VOA TO-15	GC/MS	Chloromethane
Air	Simi Valley SOP VOA TO-15	GC/MS	cis-1,2-Dichloroethene
Air	Simi Valley SOP VOA TO-15	GC/MS	cis-1,3-Dichloropropene
Air	Simi Valley SOP VOA TO-15	GC/MS	Cumene
Air	Simi Valley SOP VOA TO-15	GC/MS	Cyclohexane
Air	Simi Valley SOP VOA TO-15	GC/MS	Cyclohexanone
Air	Simi Valley SOP VOA TO-15	GC/MS	Dibromochloromethane
Air	Simi Valley SOP VOA TO-15	GC/MS	Dichlorodifluoromethane (CFC 12)
Air	Simi Valley SOP VOA TO-15	GC/MS	Diisopropyl Ether
Air	Simi Valley SOP VOA TO-15	GC/MS	d-Limonene
Air	Simi Valley SOP VOA TO-15	GC/MS	Ethanol
Air	Simi Valley SOP VOA TO-15	GC/MS	Ethyl Acetate
Air	Simi Valley SOP VOA TO-15	GC/MS	Ethyl tert-Butyl Ether
Air	Simi Valley SOP VOA TO-15	GC/MS	Ethylbenzene
Air	Simi Valley SOP VOA TO-15	GC/MS	Hexachlorobutadiene
Air	Simi Valley SOP VOA TO-15	GC/MS	Isooctane
Air	Simi Valley SOP VOA TO-15	GC/MS	Isopropyl acetate
Air	Simi Valley SOP VOA TO-15	GC/MS	Isopropyl Alcohol
Air	Simi Valley SOP VOA TO-15	GC/MS	m-&,p-Xylenes
Air	Simi Valley SOP VOA TO-15	GC/MS	Methyl Methacrylate
Air	Simi Valley SOP VOA TO-15	GC/MS	Methyl tert-Butyl Ether
Air	Simi Valley SOP VOA TO-15	GC/MS	Methylene Chloride
Air	Simi Valley SOP VOA TO-15	GC/MS	Naphthalene
Air	Simi Valley SOP VOA TO-15	GC/MS	n-Butyl Acetate
Air	Simi Valley SOP VOA TO-15	GC/MS	n-Butylbenzene
Air	Simi Valley SOP VOA TO-15	GC/MS	n-Decane

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

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

Matrix	Standard Method	Technology	Analyte
Air	Simi Valley SOP VOA TO-15	GC/MS	n-Dodecane
Air	Simi Valley SOP VOA TO-15	GC/MS	n-Heptane
Air	Simi Valley SOP VOA TO-15	GC/MS	n-Hexane
Air	Simi Valley SOP VOA TO-15	GC/MS	n-Nonane
Air	Simi Valley SOP VOA TO-15	GC/MS	n-Octane
Air	Simi Valley SOP VOA TO-15	GC/MS	n-Propylbenzene
Air	Simi Valley SOP VOA TO-15	GC/MS	n-Undecane
Air	Simi Valley SOP VOA TO-15	GC/MS	o-Xylene
Air	Simi Valley SOP VOA TO-15	GC/MS	p-Isopropyltoluene
Air	Simi Valley SOP VOA TO-15	GC/MS	Propene
Air	Simi Valley SOP VOA TO-15	GC/MS	sec-Butylbenzene
Air	Simi Valley SOP VOA TO-15	GC/MS	Styrene
Air	Simi Valley SOP VOA TO-15	GC/MS	t-Butanol
Air	Simi Valley SOP VOA TO-15	GC/MS	tert-Amyl Methyl Ether
Air	Simi Valley SOP VOA TO-15	GC/MS	tert-Butylbenzene
Air	Simi Valley SOP VOA TO-15	GC/MS	Tetrachloroethene
Air	Simi Valley SOP VOA TO-15	GC/MS	Tetrahydrofuran
Air	Simi Valley SOP VOA TO-15	GC/MS	Toluene
Air	Simi Valley SOP VOA TO-15	GC/MS	trans-1,2-Dichloroethene
Air	Simi Valley SOP VOA TO-15	GC/MS	trans-1,3-Dichloropropene
Air	Simi Valley SOP VOA TO-15	GC/MS	Trichloroethene
Air	Simi Valley SOP VOA TO-15	GC/MS	Trichlorofluoromethane
Air	Simi Valley SOP VOA TO-15	GC/MS	Trichlorotrifluoroethane
Air	Simi Valley SOP VOA TO-15	GC/MS	Vinyl Acetate
Air	Simi Valley SOP VOA TO-15	GC/MS	Vinyl Chloride
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,1,1-Trichloroethane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,1,2,2-Tetrachloroethane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,1,2-Trichloroethane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,1-Dichloroethane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,1-Dichloroethene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,2,4-Trichlorobenzene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,2,4-Trimethylbenzene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,2-Dibromo-3-chloropropane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,2-Dibromoethane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,2-Dichloro-1,1,2,2-tetrafluoroethane (CFC 114)
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,2-Dichlorobenzene

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental

2655 Park Center Drive, Suite A, Simi Valley, CA 93065
Chaney Humphrey Phone: 805-526-7161

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

Matrix	Standard Method	Technology	Analyte
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,2-Dichloroethane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,2-Dichloropropane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,3,5-Trimethylbenzene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,3-Butadiene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,3-Dichlorobenzene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,4-Dichlorobenzene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	1,4-Dioxane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	2,2,4-Trimethylpentane (Isooctane)
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	2-Butanone (MEK)
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	2-Hexanone
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	2-Propanol (Isopropyl Alcohol)
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	4-Methyl-2-pentanone
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Acetone
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Acetonitrile
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Benzene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Bromodichloromethane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Bromoform
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Carbon Disulfide
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Carbon Tetrachloride
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Chlorobenzene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Chloroethane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Chloroform
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Chloromethane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	cis-1,2-Dichloroethene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	cis-1,3-Dichloropropene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Cumene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Cyclohexane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Dibromochloromethane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Dichlorodifluoromethane (CFC 12)
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Ethanol
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Ethylbenzene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Hexachlorobutadiene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	m,p-Xylenes
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Methyl tert-Butyl Ether
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Methylene Chloride
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Naphthalene

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ISO/IEC 17025:2005 and DoD-ELAP

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Chaney Humphrey Phone: 805-526-7161

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

Matrix	Standard Method	Technology	Analyte
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	n-Heptane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	n-Hexane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	n-Octane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	o-Xylene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Styrene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Tetrachloroethene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Tetrahydrofuran (THF)
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Toluene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	trans-1,2-Dichloroethene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	trans-1,3-Dichloropropene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Trichloroethene
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Trichlorofluoromethane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Trichlorotrifluoroethane
Air	Simi Valley SOP VOA TO-17/EPA TO-17	GC/MS	Vinyl Chloride
Air	Simi Valley SOP VOA-TO3C1C6	GC/FID	C1 - C6+
Air	Simi Valley SOP VOA-TO3C1C6	GC/FID	Ethane
Air	Simi Valley SOP VOA-TO3C1C6	GC/FID	Methane
Air	Simi Valley SOP VOA-TO3C1C6	GC/FID	n-Butane
Air	Simi Valley SOP VOA-TO3C1C6	GC/FID	n-Hexane
Air	Simi Valley SOP VOA-TO3C1C6	GC/FID	n-Pentane
Air	Simi Valley SOP VOA-TO3C1C6	GC/FID	Propane
Air	Simi Valley SOP VOA-TO3C1C6	GC/FID	Total Volatile Petroleum Hydrocarbons (TVPH) as Hexane
Air	Simi Valley SOP VOA-TPHG_TO3	GC/FID	Total Petroleum Hydrocarbons Gasoline (TPHG)
Aqueous	RSK 175	GC/TCD	Carbon Dioxide
Aqueous	RSK 175	GC/FID	Ethane
Aqueous	RSK 175	GC/FID	Ethene
Aqueous	RSK 175	GC/FID	Methane

The State of Washington
Department of Ecology

ALS Environmental - Simi Valley
Simi Valley, CA

has complied with provisions set forth in Chapter 173-50 WAC and is hereby recognized by the Department of Ecology as an ACCREDITED LABORATORY for the analytical parameters listed on the accompanying Scope of Accreditation. This certificate is effective May 13, 2017 and shall expire May 12, 2018.

Witnessed under my hand on April 14, 2017

Alan D. Rue
Lab Accreditation Unit Supervisor

Laboratory ID
C946

WASHINGTON STATE DEPARTMENT OF ECOLOGY

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

SCOPE OF ACCREDITATION

ALS Environmental - Simi Valley

Simi Valley, CA

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

Matrix/Analyte	Method	Notes
Air		
Carbon monoxide	ALS-SOP-VOA-EPA 25CM	2,3
1,1,1-Trichloroethane	EPA TO-15 Rev. 2 (1999)	1
1,1,2,2-Tetrachloroethane	EPA TO-15 Rev. 2 (1999)	1
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA TO-15 Rev. 2 (1999)	1
1,1,2-Trichloroethane	EPA TO-15 Rev. 2 (1999)	1
1,1-Dichloroethane	EPA TO-15 Rev. 2 (1999)	1
1,1-Dichloroethylene	EPA TO-15 Rev. 2 (1999)	1
1,2,3-Trimethylbenzene	EPA TO-15 Rev. 2 (1999)	1
1,2,4-Trichlorobenzene	EPA TO-15 Rev. 2 (1999)	1
1,2,4-Trimethylbenzene	EPA TO-15 Rev. 2 (1999)	1
1,2-Dibromo-3-chloropropane (DBCP)	EPA TO-15 Rev. 2 (1999)	1
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA TO-15 Rev. 2 (1999)	1
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	EPA TO-15 Rev. 2 (1999)	1
1,2-Dichlorobenzene	EPA TO-15 Rev. 2 (1999)	1
1,2-Dichloroethane (Ethylene dichloride)	EPA TO-15 Rev. 2 (1999)	1
1,2-Dichloropropane	EPA TO-15 Rev. 2 (1999)	1
1,3,5-Trimethylbenzene	EPA TO-15 Rev. 2 (1999)	1
1,3-Butadiene	EPA TO-15 Rev. 2 (1999)	1
1,3-Dichlorobenzene	EPA TO-15 Rev. 2 (1999)	1
1,4-Dichlorobenzene	EPA TO-15 Rev. 2 (1999)	1
1,4-Dioxane (1,4- Diethyleneoxide)	EPA TO-15 Rev. 2 (1999)	1

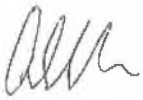
Matrix/Analyte	Method	Notes
1-Butanol (n-Butanol)	EPA TO-15 Rev. 2 (1999)	1
1-Propene	EPA TO-15 Rev. 2 (1999)	1
2-Butanone (Methyl ethyl ketone, MEK)	EPA TO-15 Rev. 2 (1999)	1
2-Ethyltoluene	EPA TO-15 Rev. 2 (1999)	1
2-Hexanone	EPA TO-15 Rev. 2 (1999)	1
3-Ethyltoluene	EPA TO-15 Rev. 2 (1999)	1
4-Ethyltoluene	EPA TO-15 Rev. 2 (1999)	1
4-Isopropyltoluene (p-Cymene)	EPA TO-15 Rev. 2 (1999)	1
4-Methyl-2-pentanone (MIBK)	EPA TO-15 Rev. 2 (1999)	1
Acetone	EPA TO-15 Rev. 2 (1999)	1
Acetonitrile	EPA TO-15 Rev. 2 (1999)	1
Acrolein (Propenal)	EPA TO-15 Rev. 2 (1999)	1
Acrylonitrile	EPA TO-15 Rev. 2 (1999)	1
Allyl chloride (3-Chloropropene)	EPA TO-15 Rev. 2 (1999)	1
alpha-Methylstyrene	EPA TO-15 Rev. 2 (1999)	1
alpha-Pinene	EPA TO-15 Rev. 2 (1999)	1
Benzene	EPA TO-15 Rev. 2 (1999)	1
Benzyl chloride	EPA TO-15 Rev. 2 (1999)	1
Bromodichloromethane	EPA TO-15 Rev. 2 (1999)	1
Bromoform	EPA TO-15 Rev. 2 (1999)	1
Carbon disulfide	EPA TO-15 Rev. 2 (1999)	1
Carbon tetrachloride	EPA TO-15 Rev. 2 (1999)	1
Chlorobenzene	EPA TO-15 Rev. 2 (1999)	1
Chlorodibromomethane	EPA TO-15 Rev. 2 (1999)	1
Chloroethane (Ethyl chloride)	EPA TO-15 Rev. 2 (1999)	1
Chloroform	EPA TO-15 Rev. 2 (1999)	1
cis-1,2-Dichloroethylene	EPA TO-15 Rev. 2 (1999)	1
cis-1,3-Dichloropropene	EPA TO-15 Rev. 2 (1999)	1
Cyclohexane	EPA TO-15 Rev. 2 (1999)	1
Cyclohexanone	EPA TO-15 Rev. 2 (1999)	1
Dichlorodifluoromethane (Freon-12)	EPA TO-15 Rev. 2 (1999)	1
Di-isopropylether (DIPE)	EPA TO-15 Rev. 2 (1999)	1
d-Limonene	EPA TO-15 Rev. 2 (1999)	1
Ethanol	EPA TO-15 Rev. 2 (1999)	1
Ethyl acetate	EPA TO-15 Rev. 2 (1999)	1
Ethyl tert-Butyl Ether	EPA TO-15 Rev. 2 (1999)	1

Matrix/Analyte	Method	Notes
Ethylbenzene	EPA TO-15 Rev. 2 (1999)	1
Hexachlorobutadiene	EPA TO-15 Rev. 2 (1999)	1
Isooctane	EPA TO-15 Rev. 2 (1999)	1
Isopropyl acetate	EPA TO-15 Rev. 2 (1999)	1
Isopropyl alcohol (2-Propanol, Isopropanol)	EPA TO-15 Rev. 2 (1999)	1
Isopropylbenzene	EPA TO-15 Rev. 2 (1999)	1
m+p-xylene	EPA TO-15 Rev. 2 (1999)	1
Methyl bromide (Bromomethane)	EPA TO-15 Rev. 2 (1999)	1
Methyl chloride (Chloromethane)	EPA TO-15 Rev. 2 (1999)	1
Methyl methacrylate	EPA TO-15 Rev. 2 (1999)	1
Methyl tert-butyl ether (MTBE)	EPA TO-15 Rev. 2 (1999)	1
Methylene chloride (Dichloromethane)	EPA TO-15 Rev. 2 (1999)	1
Naphthalene	EPA TO-15 Rev. 2 (1999)	1
n-Butyl-acetate	EPA TO-15 Rev. 2 (1999)	1
n-Butylbenzene	EPA TO-15 Rev. 2 (1999)	1
n-Decane	EPA TO-15 Rev. 2 (1999)	1
n-Dodecane	EPA TO-15 Rev. 2 (1999)	1
n-Heptane	EPA TO-15 Rev. 2 (1999)	1
n-Hexane	EPA TO-15 Rev. 2 (1999)	1
n-Nonane	EPA TO-15 Rev. 2 (1999)	1
n-Octane	EPA TO-15 Rev. 2 (1999)	1
n-Propylbenzene	EPA TO-15 Rev. 2 (1999)	1
n-Undecane	EPA TO-15 Rev. 2 (1999)	1
o-Xylene	EPA TO-15 Rev. 2 (1999)	1
sec-Butylbenzene	EPA TO-15 Rev. 2 (1999)	1
Styrene	EPA TO-15 Rev. 2 (1999)	1
tert-amylmethylether (TAME)	EPA TO-15 Rev. 2 (1999)	1
tert-Butyl alcohol	EPA TO-15 Rev. 2 (1999)	1
tert-Butylbenzene	EPA TO-15 Rev. 2 (1999)	1
Tetrachloroethylene (Perchloroethylene)	EPA TO-15 Rev. 2 (1999)	1
Tetrahydrofuran (THF)	EPA TO-15 Rev. 2 (1999)	1
Toluene	EPA TO-15 Rev. 2 (1999)	1
trans-1,2-Dichloroethylene	EPA TO-15 Rev. 2 (1999)	1
trans-1,3-Dichloropropylene	EPA TO-15 Rev. 2 (1999)	1
Trichloroethene (Trichloroethylene)	EPA TO-15 Rev. 2 (1999)	1
Trichlorofluoromethane (Freon 11)	EPA TO-15 Rev. 2 (1999)	1

Matrix/Analyte	Method	Notes
Vinyl acetate	EPA TO-15 Rev. 2 (1999)	1
Vinyl chloride	EPA TO-15 Rev. 2 (1999)	1
Non-Potable Water		
Ethane	EPA RSK-175	1
Ethene	EPA RSK-175	1
Methane	EPA RSK-175	1

Accredited Parameter Note Detail

(1) Accreditation based in part on recognition of Perry Johnson Laboratory Accreditation, Inc. Department of Defense accreditation.(2) Accreditation based in part on recognition of the United States Department of the Navy Laboratory Accreditation.(3) Modified EPA Method 25C.



Authentication Signature
Alan D. Rue, Lab Accreditation Unit Supervisor

04/14/2017

Date

ATTACHMENT C

LABORATORY STANDARD OPERATING PROCEDURES

ALS Standard Operating Procedure

DOCUMENT TITLE:	ANALYSIS OF HYDROGEN AND HELIUM USING GAS CHROMATOGRAPHY WITH THERMAL CONDUCTIVITY DETECTION (TCD)
REFERENCED METHOD:	EPA 3C MODIFIED
SOP ID:	VOA-HHE
REV. NUMBER:	06.0
EFFECTIVE DATE:	09/20/2014

Proprietary - Uncontrolled Copy

STANDARD OPERATING PROCEDURE

ANALYSIS OF HYDROGEN AND HELIUM USING GAS CHROMATOGRAPHY WITH THERMAL CONDUCTIVITY DETECTION (TCD)

EPA 3C MODIFIED

SOP ID:	VOA-HHe	Rev. Number:	06.0	Effective Date:	09/20/2014

Approved By: _____ Date: 9/3/14
Department Supervisor - Wade Henton

Approved By: _____ Date: 9/3/14
QA Manager - Chaney Humphrey

Approved By: _____ Date: 9/5/14
Laboratory Director - Kelly Horiuchi

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ANALYSIS OF HYDROGEN AND HELIUM USING GAS CHROMATOGRAPHY WITH THERMAL CONDUCTIVITY DETECTION (TCD)

1) Scope and Applicability

1.1 This gas chromatographic method is used in the analysis of helium and hydrogen by a modification of EPA Method 3C.

1.2 This method applies to but is not limited to the following sample matrices: ambient air, soil vapor extraction, and source emissions. The range of this method for quantifying target analyte gases, depending on the concentration of the samples, is approximately 25ppm to percent values. The upper limit may be extended by diluting the sample with an inert gas or by using a smaller injection volume. The number of samples, which may be analyzed in one eight hour day, is approximately twenty.

1.3 The method detection and reporting limits covered by this document are listed in Attachment D. The method reporting limit for a compound is defined as the minimum reliably quantifiable concentration of that compound.

2) Summary of Procedure

2.1 Samples are collected in Tedlar bags, Mylar bags, glass bottles, Summa or specially prepared canisters and delivered to the laboratory for analysis (Tedlar bags are not recommended because of the relative porosity of Tedlar to molecular size of helium or hydrogen). An aliquot is drawn from the sampling container using a gastight syringe and injected onto a packed chromatographic column where the analytes are separated and measured using a thermal conductivity detector (TCD). Analytes are identified and quantified based on their retention time, which is compared with that of a known standard under identical conditions.

3) Definitions

3.1 Relative Standard Deviation (RSD) The RSD is the coefficient of variation (CV; ratio of the standard deviation to the mean) multiplied by 100 to convert the CV to a percentage of the mean.

3.2 Analytical Sequence The analytical sequence describes exactly how the field and QC samples in an analytical batch are to be analyzed.

3.3 Field Sample A sample collected and delivered to the laboratory for analysis.

3.4 Batch QC Batch QC refers to the QC samples that are analyzed in an analytical batch of field samples and includes the Method Blank (MB), Laboratory Control Sample (LCS) and Laboratory Duplicate (LD), etc.

3.5 Calibration Standard (Initial Calibration – ICAL) A calibration standard is a known concentration of desired analyte(s) prepared from a primary standard, which is, in turn, prepared from a stock standard material. A calibration standard is analyzed at varying concentrations and used to calibrate the response of the measurement system with respect to analyte concentration.

3.6 Initial Calibration Verification (ICV) Standard An initial calibration verification standard (ICV) is a standard that is prepared from materials obtained from a source other than the source for the calibration standards and is analyzed after the measurement system is calibrated, but prior to sample analysis in order to verify the calibration of the measurement system.

3.7 Continuing Calibration Verification (CCV) Standard A continuing calibration verification standard (CCV) is a midrange calibration standard that is analyzed periodically to verify the continuing calibration of the measurement system.

3.8 Method Blank (MB) The method blank (MB) for this method is ultra-pure nitrogen that is analyzed to verify the zero point of the analytical system and to verify freedom from carryover.

3.9 Laboratory Control Sample (LCS) For the purposes of this document, a laboratory control sample (LCS) shall be a calibration standard of known concentration. The percent recovery of the analyte(s) in the LCS is used to assess method performance.

3.10 Laboratory Duplicate An aliquot of a sample taken from the same container under similar laboratory conditions which are processed and analyzed independently.

3.11 Precision Precision of a method is how close results are to one another, and is usually expressed by measures such as standard deviation, which describe the spread of results.

3.12 Bias The bias of a method is an expression of how close the mean of a set of results (produced by the method) is to the true value.

3.13 Manual Integration This term applies to a data file in which setpoints have been changed and reintegration has occurred under the changed setpoints; baselines have been adjusted; peak integration start and stop "ticks" have been changed; peak area, or peak height, are changed after the time of data collection and data file generation.

4) Health and Safety Warnings

4.1 Each compound, mixture of compounds and standards, as well as samples, should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest level possible through the use of gloves (to minimize absorption through the skin) and hoods (to minimize inhalation). Refer to the laboratory's Environmental, Health and Safety Manual as it makes reference to the safe handling of chemicals, Safety Data Sheet location, as well as the laboratory waste management plan for the safe disposal of chemicals and samples.

4.2 Safety Data Sheets (SDS) Safety data sheets (SDS) are available in the conference room and should be reviewed as part of employee training.

4.3 Protective Clothing Personal protective clothing (safety glasses and gloves) should be used when preparing standards, handling standards in neat form or performing maintenance on pressurized systems.

4.4 Pressurized Gases The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp at all times. The regulator should not remain on size "D" cylinders when not in use. Sources of flammable gases (i.e. pressurized hydrogen) should be clearly labeled.

4.5 Syringes Care should be taken to avoid personal injury as a result of improper handling techniques.

4.6 Pollution Prevention and Waste Management All waste management must be carried out in accordance with the requirements detailed in the *SOP for Waste Disposal* as well as the Health and Safety Manual.

5) Cautions

5.1 A maintenance log shall be kept documenting maintenance performed on each analytical system and the instrument maintenance log must be kept current. The serial

numbers of each instrument shall be recorded in the front of the logbook. An entry shall be made in the appropriate log every time maintenance is performed (no matter the extent). The extent of the maintenance is not important, however, it is important that a notation be included for each maintenance activity such as changing a column. The entry in the log must include:

(a) The date of maintenance
(b) Who did the maintenance
(c) Description of the maintenance
(d) Proof that the maintenance activity was successful

A notation of a successful continuing calibration or initial calibration shall serve as proof that the maintenance is complete and the instrument is in working order.

5.2 Carrier Gas Purifier If in-line purifiers or scrubbers are in place, these purifiers must be changed as recommended by the supplier.

5.3 GC System

5.3.1 Column Performance should be monitored by observing peak shapes and column bleed. Over time, the column may exhibit a poor overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur depends on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced.

Declining performance can also be due to ineffective column ferrules, which should be replaced when a tight seal around the column is no longer possible. This can be detected with the use of a leak detector.

5.3.2 Injection Port Injection port maintenance includes changing the injection port liner and column ferrule as needed. Liners should be changed when recent sample analyses predict a problem in chromatographic performance.

5.3.3 Injector Septa Septa should be changed monthly or whenever there is a noticeable change in peak definition. For best results with air analyses, two septa are placed into the injector in order to eliminate loss during manual injections.

5.3.4 Detector See manual for TCD cleaning as needed.

6) Interferences

6.1 Column Conditioning Conditioning of the chromatographic column is required prior to use of the system. The column should be conditioned with a continuous flow of chromatographic grade nitrogen and temperature programmed from 35˚C to 200˚C at a rate of five degrees per minute. The column should be held at 200˚C for at least four hours.

6.2 Contamination in the Sample Care must be taken to prevent ambient air intrusion into the sample container during pressurization and laboratory analysis. When using adapters and fittings the dead volume must be evacuated and replaced with the sample gas prior to sampling from the container. The sampling loop shall then be flushed with the sample gas to remove residual ambient air. An aliquot greater than is needed is drawn, and the syringe plunger is adjusted to the appropriate volume *immediately* before injecting.

6.3 Carrier Gas Contamination To prevent system contamination, UHP/ZERO grade helium (99.999% purity) is used as the carrier gas. Also, a purifier and an oxygen trap are

incorporated into the analytical system as additional insurance against possible contamination.

6.4 Detector The TCD is a non-specific detector, and will produce a response for a wide range of compounds. It is important to be sure that the target peak is completely chromatographically resolved from compounds that elute immediately before or after.

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review and reporting per the corresponding standard operating procedures. Analysis and interpretation of results must be performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration shall be in accordance with the training program of the laboratory described in the *SOP for Training Policy*.

7.2 An initial demonstration of proficiency shall be performed prior to independent analyses of samples. In addition, a continuing demonstration must be performed annually. Both demonstrations consist of spiking Tedlar bags with an LCS standard and evaluating for both precision and accuracy. The criteria for approval are the same as the acceptance criteria for the LCS as specified in this document. See Attachment D.

7.3 The department supervisor/manager or designee shall perform final review and sign-off of the data.

8) Sample Collection, Handling, and Preservation

8.1 The samples are collected and delivered to the laboratory for analysis in either Tedlar bags, glass bottles (Bottle Vac. Entech Instruments), or specially prepared canisters.

8.2 Samples collected in bags must be analyzed within 72 hours after sample collection unless otherwise specified by the client.

8.3 Samples delivered in cleaned, evacuated summa or other specially prepared containers do not have specified holding times for atmospheric gases but should be analyzed within 30 days from the date of collection.

9) Equipment and Supplies

9.1 Gas Chromatograph HP 5890A, Series II or equivalent equipped with a thermal conductivity detector, and having a temperature programmable oven. The column shall be a molecular sieve 5A, 6 meter, 1/8" ID packed column.

9.2 Regulators Regulators are used on the gas cylinders supplying the GC and for preparing cylinder standards.

9.3 Data System A data system with the ability to collect data from the GC detector, integrate the peaks and perform the appropriate quantification calculations shall be used. ALS Environmental - Simi Valley currently uses HP Chemstation/Enviroquant GC software.

9.4 Syringes Gas tight syringes of the following volumes: 10mL, 2.5mL, 1.0mL and 0.5mL.

9.5 Tedlar Bags/Mylar Bags/Glass Bombs Glass "bombs" of volumes 125ml, 250ml, or 500ml are used for diluting very concentrated samples, which fall outside of the initial calibration range and for making working standards. In addition, Mylar or Tedlar bags can be used.

10) Standards and Reagents

10.1 All samples and standards must be stored separately. The concentration, preparation and expiration date as well as analyst's initials must be identified on the standard label. Each standard must also be uniquely identified with a laboratory ID number.

10.2 All certificates shall be maintained (turned in to quality assurance) and noted with the standard identification number, date received and initials of the receiving analyst. For additional information on these and other requirements, refer to the *SOP for Handling Consumable Materials.*

10.3 Carrier and Calibration Standard Balance Gas

10.3.1 Nitrogen - UHP/ZERO (99.999%) or higher in purity

10.4 Neat Standards These standards must be stored in accordance with the requirements described in the *SOP for Handling Consumable Materials*. These standards may be stored for a period of 2 years for air standards or as recommended by the manufacturer.

Compound	Purity
Hydrogen	99.999+%
Helium	99.999+%

10.5 Initial Calibration Standard / Working Standard Prepare the working standard by spiking the appropriate pure compound into a clean glass dilution bomb. Depending on the desired dynamic range of the initial calibration, various concentrations shall be made in the glass dilution bombs. Serial dilution should not be necessary.

The current concentration recommendation is two glass dilution bombs of 500ppm and 20000ppm for Helium. 500ppm is made by spiking 250ul of pure helium into a 0.5L glass bomb of nitrogen and 20000ppm is made by first removing 10mls of the nitrogen and then spiking in 10mls of pure helium into a 0.5L glass bomb. Record the calibration standard in accordance with the requirements described in the *SOP for Handling Consumable Materials.* The intermediate standard, along with all dilutions, must be stored at room temperature and expire 1 day after preparation.

11) Method Calibration

11.1 Initial Calibration

The instrument must be calibrated initially and whenever the laboratory performs corrective action (maintenance), which may change or affect the initial calibration criteria, or if the continuing calibration acceptance criteria have not been met. Introduce each initial calibration concentration standard (at least five levels, analyzed from low concentration to high concentration) by direct injection using a gas tight syringe. Perform all calibration runs according to the analytical portion of the sample analysis described in Section 12.7.

Note: The concentrations of the initial calibration may change as long as the low standard analyzed is the same as the reporting limit for each analyte.

Refer to Section 15.1 for the required calculations and Section 16.4 for the acceptance criteria and corrective action.

11.1.1 Initial Calibration Requirements

Once a set of ICAL standards is analyzed, the previous ICAL may no longer be used to analyze new samples and it must be archived. The only time an

archived ICAL can be used thereafter is to review or re-evaluate samples(s) previously processed using that ICAL.

1. A minimum of 5 concentrations must be used to calculate the calibration curve.
2. Highest concentration, together with the lowest concentration, defines the calibration curve.
3. Lowest concentration must be at method reporting limit.
4. A blank should be analyzed prior to beginning the analysis of the calibration standards.
5. The initial calibration event may not be interrupted by maintenance.
6. Only one value per concentration may be used.
7. Analyze calibration standards from low to high concentration.
8. All ICAL analyses must be completed within 48 hours.
9. If 5 calibration standards are in the ICAL, one standard may be re-analyzed. If 6 to 10 calibration standards are in the ICAL, two calibration standards may be re-analyzed.
10. Point dropping policy
    - Minimum of 5 consecutive concentrations must be used to calculate the calibration curve.
    - Lowest concentration must be at the MRL and may not be dropped unless the MRL is changed to the concentration of the remaining lowest standard.
    - Points at high end may be dropped, but doing so lowers the calibration curve.
    - Points may not be dropped from the interior of the curve unless an assignable cause (i.e., gross dilution or standard preparation error, or instrument malfunction) is accounted for and documented in a nonconformity and corrective action report (NCAR). In these instances, all the analytes in that calibration standard must be dropped from the calibration curve as the corrective action.
    - If a point or a calibration standard is dropped, the reason must be documented (and the results maintained with the documentation for the final ICAL).
    - A calibration standard may be re-analyzed if the first analysis of the standard has been dropped and other requirements in this policy are met (i.e., still within 48 hours).
    - Once the ICAL has been used to calculate and report sample results, it is not to be changed.

11.1.2 ICAL Update Procedure

1. Open most recent method
2. Save to new ICAL method ID. The date used in method ID is the date files were analyzed.
3. Clear all responses prior to update initiation and/or clear levels if different concentrations are to be used (Initial Calibration → Clear All Calibration Responses; Initial Calibration → Clear All Calibration Levels).
4. Quantitate standard
5. Review all peaks for retention time, integration, etc.
6. Update responses for standard
7. Repeat for all standards
8. If necessary load midpoint standard and update retention times
9. Save method

10. Verify Calibration Files listed on Response Factor Report are correct (Both Primary and Secondary Reviewer).
11. Verify responses of Page 3 of Edit Compounds are correct (Both Primary and Secondary Reviewer).
12. Verify file ID, acquisition time, quant time, update time, and last update information is correct on the Calibration Status Report (Both Primary and Secondary Reviewer).
13. Save Method. Confirm that no other copies of the method are open on other computer workstations.

Note: It is also acceptable to quantitate all standards and review all peaks before updating responses but steps 1-2 still must be completed initially. Step 3 also must be done prior to beginning ICAL update.

11.1.3 Initial Calibration Review

Analyst's calculations and assessment along with a peer review of all ICAL data and documentation as stated in Attachment B is required before the ICAL may be used to analyze samples. Sample results may only be reported if the ICAL is reviewed and found to be acceptable.

11.1.4 Initial Calibration File

An ICAL file is to be created for each initial calibration performed per instrument into which is placed the following ICAL documents. The file shall remain in the laboratory and be filed by instrument and date.

- ICAL Checklist filled out, reviewed and approved
- Blank analysis quantitation report
- Calibration status report (a.k.a. Calibration History)
- Relative Response Factor Report / Percent Relative Standard Deviation
- Quantitation report for each calibration standard (including manual integration documentation – before and after manual integration)
- ICV quantitation report and evaluate continuing calibration report (a.k.a. Percent Difference Report)

11.1.5 Initial Calibration Verification Verify the initial calibration by analyzing an independent calibration verification standard (ICV). Refer to Section 15.2 for the required calculations and Section 16.5 for the acceptance criteria and corrective action.

12) Sample Preparation/Analysis

12.1 Analytical Sequence and Data System Setup

12.1.1 Data System Load the appropriate acquisition method file for the gas chromatograph temperature program. Load the appropriate analytical sequence. Enter the analytical sequence information in the table window, including standard name, sample name and injection volume.

12.1.2 Analytical Sequence The analytical batch must be completed for the analysis of ≤20 field samples. Laboratory duplicates (LD), duplicate field samples and sample dilutions are considered samples. Batch QC samples may be analyzed anywhere in the analytical sequence, with the exception of the method blank which must be analyzed prior to sample analysis in order to demonstrate a contamination free system.

Analytical Sequence Guideline[1]

Sample Description (w/ICAL)	*Sample Description*
Calibration Stds.[2]	CCV[3]
ICV[4]	MB[5]
MB[5]	LCS[6]
LCS[6]	Samples 1-10
Samples 1-10	CCV[3]
CCV[3]	Samples 11-19
Samples 11-19	LD[7]
LD[7]	CCV[3]
CCV[3]	

[1] The batch QC may be analyzed in an order other than the one listed in this document; the analytical sequence specified below is a guideline.
[2] The initial calibration must be generated in accordance with the guidelines detailed in Section 11.1 of this document.
[3] In cases, where the ICAL is not performed the analytical sequence must begin with the analysis of a CCV standard. In addition, the analytical sequence shall end with an acceptable CCV.
[4] Every ICAL must be followed by a second source standard (ICV) which contains all of the target analytes.
[5] The method blank must be analyzed prior to any samples within the sequence.
[6] Every analytical sequence must include a laboratory control sample. A LCS shall be analyzed at a rate of one per twenty samples or fewer for each analyte.
[7] A laboratory duplicate must be analyzed at a frequency of 1 in 20 or fewer samples.

12.2 GC Configuration

12.2.1 Temperature Program The carrier gas flow rate and GC oven temperature programming must be set to completely elute the target analyte(s). The temperature program ramps up to a high temperature, not exceeding the maximum temperature rating of the column in use, and holds there to allow all heavier compounds to elute, in order to prevent carryover to the next injection.

The settings and system parameters are as follows:

HP 5890 GC Instrument Control Parameters

Sample Inlet: GC
Injection Source: Manual
Injection Location: Back
Run Time: 7.0 minutes

OVEN

Initial Temperature: 125ºC *Maximum Temperature:* 280ºC
Initial Time: 1.0min *Equilibration Time:* 0.0

Ramp 1 *Rate:* 25ºC/min
Final Temp: 200ºC
Final Time: 0min

INJECTOR

Mode: Packed Column
Temp: 100ºC
Pressure: 24psi at 55ºC oven temperature

STAINLESS STEEL PACKED COLUMN

Packing material: Molecular sieve 5A
Max. Temp.: 325ºC
Nominal Length 1: 6.0m
Nominal Diameter 1: 1/8" ID

DETECTOR

Reference Gas (N2): 2.5mL/min

12.3 Retention Time (RT) Windows

Retention time windows for each target analyte must be generated initially, when standard analyses result in analyte retention times outside the established window, and whenever there is a major change in instrument conditions including flow rates. The procedure for determining the retention time windows for this method is as follows. However, other approaches may be employed, providing that the analyst can demonstrate that they provide performance appropriate for the intended application. For example, the analyst may use the corresponding retention times from the initial calibration as they may show shifts in RTs due to analyte concentrations.

1. Make sure that the system is operating reliably and that the system conditions have been optimized for the target analytes in the sample matrix to be analyzed.
2. Make four injections of all applicable standard mixes over a 72 hour period. Make the injections cover the entire 72-hour period or the end result could be windows which are too tight.
3. Record the retention time for each single component analyte to three decimal places. Calculate the mean and standard deviation of the four absolute retention times for each single component analyte.
4. If the standard deviation of the retention times for the target compound is 0.000, then additional injections may be included or the use of a default standard deviation of 0.01 minutes.
5. The width of the retention time window for each analyte is defined as ±3 times the standard deviation of the mean absolute retention time established during the 72 hour period. If the default standard deviation of 0.01 is used, the width of the window will be 0.03 minutes.
6. Establish the center of the retention time window for each analyte by using the absolute retention time for each analyte from the continuing 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.
7. Retention time windows must be calculated for each analyte on each instrument. New retention time windows must be established when a new column is installed.

12.4 Continuing Calibration

A continuing calibration standard must be analyzed every ten field samples or every 12 hours, whichever is more frequent. The analytical batch must also begin and end with the analysis of a CCV standard and shall not exceed a 24 hour period. The

concentration of the calibration verification may be varied within the established calibration range. Refer to Section 15.3 for the required calculations and Section 16.6 for the acceptance criteria and corrective action.

12.5 Method Blank

The method blank shall be obtained using ultra high purity nitrogen directly injected in the same manner as the standards and samples. A method blank must be analyzed prior to analysis of samples. A method blank must also be analyzed if carryover contamination is suspected. Refer to Section 16.7 for the acceptance criteria and corrective action.

12.6 Laboratory Control Sample

The laboratory control sample shall be an injection of the continuing calibration or initial calibration verification standard. Make sure that all of the pertinent information is included on the quantitation report including the sample identification (LCS), concentration, standard used, and analyst. Refer to Section 15.4 for the required calculations and Section 16.8 for the acceptance criteria and corrective action.

12.7 Analysis

12.7.1 Container Pressurization Sample analysis must be made using the same instrument parameters as that of the calibration standards. Refer to the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters* for the procedure of how containers are to be pressurized prior to analysis. The analyst shall record the appropriate pressures on the Service Request form. This includes noting the difference between the initial (as received pressure) and the pressure prior to pressurization for which the appropriate corrective actions have been detailed and must be followed accordingly.

12.7.2 Sample Analysis Sample analysis shall be performed by a direct injection technique using gas tight syringes. When using adapters and fittings the dead volume must be evacuated and replaced with the sample gas prior to sampling from the container. The sampling syringe shall then be flushed with the sample gas to remove residual ambient air and vented into a waste bag. This procedure entails drawing an aliquot greater than is needed, and adjusting the syringe plunger to the appropriate volume of 1mL *immediately* before injecting. Refer to Section 15.5 for the required calculations and Section 16.9 for the acceptance criteria and corrective action.

Bottle Vacs use a proprietary quick connect fitting (Micro-QT, Entech Instruments). Each female Micro-QT fitting must be purged after use to remove any remaining sample residue and prevent contamination from subsequent usage. Connect a male Micro-QT fitting to a source of ultrapure or carbon-filtered gas. Adjust the pressure to about 10 psig using an inline regulator. Connect the female fitting for several seconds, then remove and place in an oven kept at 60ºC until the next use. Do not heat the fitting higher than 80ºC.

12.7.3 Sample Dilution If any target analyte results are above the highest level of the initial calibration, a smaller sample aliquot or a dilution in a glass dilution bomb must be analyzed. Guidance in performing dilutions and exceptions to this requirement are given below.

- Use results of the original analysis to determine the approximate dilution factor required getting the largest analyte peak within the initial calibration range.

- The dilution factor chosen should keep the response of the analyte peak for a reported target compound in the upper half of the initial calibration range of the instrument. Additional compounds may be reported as long as they are within the calibration range.
- Analysis involving dilution should be made with high purity nitrogen and must be reported with a dilution factor.

Glass bomb dilution:

- Calculate the sample amount for the given size of the glass dilution bomb needed to obtain the required dilution.
- Fill the glass dilution bomb with nitrogen.
- First remove the equivalent amount of nitrogen from the glass dilution bomb using the appropriate gas tight syringe.
- Add the calculated sample amount using a gas tight syringe.

12.8 Laboratory Duplicate

Analyze two separate aliquots from the same sample container. A laboratory duplicate must be analyzed at a frequency of 1 in 20 field samples. The laboratory duplicate should be rotated among clients, whenever possible. Refer to Section 15.6 for the required calculations and Section 16.10 for the acceptance criteria and corrective action.

12.9 Manual Integration

The integration(s) for each sample is checked to ensure that it has been integrated properly. Assuming an incorrect automatic integration the analyst shall conduct the manual integration in accordance with the *SOP for Manual Integration Policy* including all documentation and reviews associated with the process. The review should include the analyst and peer reviewer initialing and dating the manual integration as an indication of acceptability and approval.

12.10 Method Detection and Limits of Quantitation

An MDL study shall be performed in accordance with the procedure outlined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* Method detection limits must be determined annually and each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation is such that it affects the sensitivity of the analysis. The MDL study shall be performed on each instrument for which this method is performed. All supporting data must be approved and retained. Refer to Section 19.2 for additional information.

12.11 Cleaning Glass Dilution Bombs

Heat to 60ºC for 30 minutes and purge for ~30 seconds from a clean pressurized nitrogen source.

12.12 Provided that the requirements of this document are followed, the modifications of EPA Method 3C by Gas Chromatograph are:

- Analysis of Hydrogen and Helium, which is not analyzed by the documented method.
- The Sampling media may be bags (Mylar, Tedlar, etc.), Bottle Vacs, Summa or specially prepared canisters.
- The samples are not analyzed in duplicate.

Note: Tedlar bags are not recommended for this analysis.

13) Troubleshooting

13.1 Prepare new standards, check instrument maintenance, prepare a new curve as needed, etc. Refer to the corrective actions listed in Section 16 of this SOP for additional troubleshooting details.

14) Data Acquisition

14.1 Storing Electronic Data

The initial calibration data must be stored in a quantitation method (on the server) using a unique filename and may not be overwritten at any time in order to maintain an accurate audit trail. Files should be named with a two-character notation indicating the compound list and the date of the corresponding initial calibration. In addition, all data files including method blanks, continuing calibration verification, laboratory control samples and client submitted samples files shall be saved in a unique sub-directory on the server. An example of how the analyst should store analytical data is as follows:

Instrument Number/Data/Method ID/yr_month/*.d

* Injection (automatically assigned based on order of injection)

14.2 Sufficient raw data records must be retained of the analysis, instrument calibrations and method detection limit studies including: analysis/calibration date and time, test method, instrument, sample identification, each analyte name, analyst's initials, concentration and response, and standards used for the analysis and calibrations, any manual calculations including sample dilutions and manual integrations. All information entered and reported on the quantitation report and instrument run log must be complete and accurate. If manual integration is necessary the guidelines described in Section 12.9 shall be followed.

14.3 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date of analysis, time of analysis, instrument operating conditions/parameters (or reference to such data), analysis type, any manual calculations including dilutions and manual integrations, analyst's initials, sample preparation (pressure readings), standard and reagent origin, sample receipt, calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, and assessment and reporting conventions.

15) Calculation and Data Reduction Requirements

15.1 Initial Calibration

The initial calibration curve must be saved with a two-letter identification (HH) followed by the date of the analysis (mm,dd,yy). This file should be saved in the following directory: J:\"instrument ID"\Method\. No curve may be overwritten at any time to ensure a complete audit trail.

- Tabulate the peak area along with standard concentration injected to determine the response factor (RF) for each analyte at each concentration using equation number 1.
- Calculate the percent relative standard deviation (%RSD) of the mean RF (equation number 2) for each analyte over the range of each concentration of the calibration standards using equation numbers 4 and 5.
- Calculate the mean retention time of each analyte spanning the initial calibration range using equation number 10.

15.2 Initial Calibration Verification

- Calculate the concentration for each analyte using equation number 3.
- Calculate the percent difference (%D) between the calculated concentration (equation number 3) and the actual concentration using equation number 6.

15.3 Continuing Calibration Verification

- Calculate the concentration of each analyte using equation number 3.
- Calculate the percent difference (%D) between the calculated concentration (equation number 3) and the actual concentration using equation number 6.

15.4 Laboratory Control Sample

- Calculate the concentration of each analyte using equation number 3.
- Calculate the percent recovery (%R) for each analyte using equation number 8.

15.5 Sample Analysis

- Calculate the concentration of each analyte using equation number 3.
- Calculate the dilution factor if necessary using equation number 9.

15.6 Laboratory Duplicate

- Calculate the concentration of each analyte using equation number 3.
- Calculate the relative percent difference (RPD) using equation number 7.

15.7 Calculations

15.7.1 Equation Number 1

Response Factor (RF)

The response factor, for analyte *x* is given by:

$$RF = \frac{A_x}{C_x}$$

where:

A_x = Area of the analyte in the standard

C_x = Concentration of the analyte in the standard

15.7.2 Equation Number 2

Average (or Mean) RF

$$\overline{RF} = \frac{\sum_{i=1}^{N} RF_i}{N}$$

where:

RF_i are the individual RFs from each concentration level in the initial calibration curve
N is the number of calibration concentration levels

15.7.3 Equation Number 3

Concentration (C):

$$C = \frac{Area}{\overline{RF}} \times \frac{D_{inj}}{A_{inj}}$$

where:

Area is the area obtained from the chromatogram
$\overline{RF}$ Average (or Mean) RF of all concentration levels in the initial calibration curve
D_{inj} default injection volume (mL)
A_{inj} actual injection volume (mL)

15.7.4 Equation Number 4

Standard Deviation, SD:

$$SD = \sqrt{\sum_{i=1}^{N} \frac{\left(RF_i - \overline{RF}\right)^2}{N-1}}$$

where:

RF_i are the individual RFs from each concentration level in the initial calibration curve
$\overline{RF}$ Average (or Mean) RF of all concentration levels in the initial calibration curve
N total number of calibration concentration levels

15.7.5 Equation Number 5

Percent Relative Standard Deviation, %RSD:

$$\%RSD = \frac{SD}{\overline{RF}}(100)$$

where:

SD Standard Deviation calculated in equation number 3
$\overline{RF}$ Average or Mean RF

15.7.6 Equation Number 6

Percent Difference, %D,

The %D is used for evaluating ICV and CCV vs. the initial calibration

$$\%D = \frac{C_{CCVorICV} - C_{std}}{C_{std}}(100)$$

where, for any given analyte:

$C_{CCVorICV}$	is the concentration being evaluated
C_{std}	is the concentration from the current calibration curve

15.7.7 Equation Number 7

Relative Percent Difference (RPD)

$$\frac{|R_1 - R_2|}{\left(\frac{R_1 + R_2}{2}\right)} x100$$

where:

R_1	First measurement value
R_2	Second measurement value

15.7.8 Equation Number 8

Percent Recovery (%R):

$$\%R = \frac{C}{S} x100$$

where

C = Concentration of the analyte recovered
S = Spiked amount

15.7.9 Equation Number 9

Dilution Factor

$$DF = \frac{V_T}{V_S}$$

Where:

DF = dilution factor
V_S = volume of sample (mL) used
V_T = total volume of dilution (mL)

15.7.10 Equation Number 10

Mean Retention Time ($\overline{RT}$)

$$\frac{\sum RT}{n}$$

where:

$\sum$ = sum of
RT = absolute retention time for each concentration
n = total number of retention times

15.8 Data Review

The analyst must review data on a real time basis for all calibration and QC data. The QC data must be evaluated following the data review checklist in Attachment C. The data shall be reviewed and the sample results calculated and assessed by one analyst and reviewed by a second qualified analyst. The data review checklist shall be used to document the review process. Once it has been completed, the checklist must be initialed, dated and filed with each job file.

Initial calibrations must be reviewed in the same manner as QC data with all ICAL documentation retained in a separate file. Refer to the initial calibration checklist in Attachment B for the review guideline. The ICAL file must contain all the pertinent information stated in Section 11.1.4.

15.9 Reporting

The results of each test shall be reported clearly, unambiguously and objectively, and shall include all the information necessary for the interpretation of the test results. The analyst shall ensure that all of the requirements specified in this document and the *SOP for Data Review and Reporting* are followed.

15.10 Sample Preparation and Analysis Observations / Case Narrative Summary Form

This form, which is included in the *SOP for Laboratory Storage, Analysis and Tracking*, must be generated when there are any specific sample composition information, sample preparation, analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved.

This form is necessary as a means for documentation. This form, among other information, will be reviewed when compiling the final report and case narrative. All information regarding the job shall remain in the file, in order that sufficient

documentation is available to recreate the job from sample receipt through preparation, analysis, data reduction, and reporting.

16) Quality Control, Acceptance Criteria, and Corrective Action

16.1 This section of the standard operating procedure contains technical acceptance criteria and preferred corrective actions to data nonconformities. Corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action*, where appropriate.

16.2 To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.3 It must be determined if there are any instrumentation problems contributing to the occurrence of any out of control data. If it is decided that problems do exist then the analyst must determine if the effects have caused any modification in the data from client submitted samples. This being the case, all samples (including QC) that are affected by instrumentation problems must be re-analyzed following any necessary maintenance activity.

16.4 Initial Calibration

16.4.1 Acceptance Criteria

- The percent relative standard deviation (%RSD) of the analytes of each of the levels must be less than 15% for the calibration to be considered acceptable.
- The retention time of each analyte at each calibration level must be within 0.06 minutes of the mean retention time.

16.4.2 Corrective Action If the initial calibration technical acceptance criteria are not met, inspect the system for possible sources. It may be necessary to change the column or take other corrective actions. Also, check standards for a bad injection and re-analyze standard. If a bad injection is not evident, perform maintenance and attempt another initial calibration (make notation in maintenance logbook regarding any steps taken). A demonstration of an in-control system is required before proceeding with the analysis.

Note: No ICAL may be interrupted by any maintenance procedure. Therefore, all standards incorporated in a curve must be reanalyzed.

16.5 Initial Calibration Verification Standard (ICV)

16.5.1 Acceptance Criteria

- The percent difference (%D) for each calculated target analyte must be within ±15% of the actual concentration of the standard.
- The retention time of each target analyte must fall within the generated retention time window using the ICAL midpoint as the absolute retention time.

16.5.2 Corrective Action If the initial calibration verification fails to meet the acceptance criteria, it should be re-analyzed. A second failed ICV must initiate corrective action and two consecutive ICVs must pass in order for the ICAL to be deemed acceptable. It may be necessary to prepare either new ICAL or ICV standards or both, perform maintenance and reanalyze the initial calibration.

16.6 Continuing Calibration Verification (CCV)

16.6.1 Acceptance Criteria

- The percent difference (%D) for each calculated target analyte must be within ±15% of the actual concentration.
- The retention time of each target analyte must be within 0.33 minutes of the mean retention time of the ICAL.

16.6.2 Corrective Action If the criteria are not met, reanalyze (no more than two injections may be made before corrective action is initiated) or prepare a fresh CCV standard and reanalyze. If routine corrective action procedures fail to produce an acceptable calibration verification, a new initial calibration must be performed. However, sample data associated with an unacceptable calibration verification may be reported as qualified data only under the following special condition:

> *When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the sample affected by the unacceptable CCV shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.*

16.7 Method Blank

16.7.1 Acceptance Criteria The method blank result for any target analyte must not be greater than the method reporting limit.

16.7.2 Corrective Action If the analyte results in the blank do not meet the acceptance criteria the source of the problem must be investigated and measures taken to eliminate the source. Determine whether the contamination is from the instrument or due to contamination in the nitrogen, syringe or other source. Regardless, appropriate corrective measures must be taken and documented before further sample analysis proceeds. *If the results are the same, the blank along with all associated samples must be reported to the client with the appropriate qualifier as specified in Section 18.*

16.8 Laboratory Control Sample (LCS)

16.8.1 Acceptance Criteria The percent recovery for all compounds must be within 85-115% or laboratory generated limits if available.

16.8.2 Corrective Action If the LCS criteria are not met, determine whether the cause is instrumentation or the result of a poor injection. If the problem is instrumentation, perform maintenance and reanalyze the associated sample(s). If the problem is with the injection, reanalyze the LCS. If the results are still unacceptable and there does not appear to be any instrumentation problems refer to Section 18 for the appropriate reporting information.

16.9 Sample Analysis

Sample results must be quantitated from the current instrument initial calibration and may not be quantitated from any continuing calibration verification standard.

16.9.1 Acceptance Criteria

- The field samples must be analyzed along with a laboratory method blank that has met the blank criteria in Section 16.7.
- All target analyte peaks must be within the initial calibration range.

- The retention time of each target analyte must fall within the retention time window using the CCV as the absolute retention time.
- The retention time of each target analyte must be within 0.33 minutes of the retention time in the mean RT from the ICAL and within the retention time window using the most recent CCV.

16.9.2 Corrective Action To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measures shall be reported with the appropriate data qualifier(s) as detailed in this document and most current Quality Assurance Manual.

- When corrective actions are made, samples analyzed while the system was not functioning properly must be reanalyzed.
- Results not bracketed by initial instrument calibration standards (within calibration range) must be reported as having less certainty, e.g., defined qualifiers or flags.

16.10 Laboratory Duplicate

16.10.1 Acceptance Criteria The selected samples must be rotated among client samples so that various matrix problems may be noted and/or addressed. The results must meet all of the criteria stated in Section 16.9.1 as well as be ≤15% relative percent difference for all analytes of interest, provided that the concentration is greater than 10x the RL.

16.10.2 Corrective Action If the replicate results do not fall within the technical acceptance window, the sample should be re-analyzed. If the results are still unacceptable and there does not appear to be any matrix effects, interfering peaks, or instrument problems, the results for both injections shall be reported to the client with the appropriate qualifier as specified in Section 18.

16.11 Sample's Holding Time Expired

The client is to be notified (best attempt) that the sample's holding time was missed and the client is to decide if the sample analysis shall continue. The documentation of missed holding time and the client's decision to proceed must be included in the corresponding job file. A statement dictating all holding time occurrences must accompany the sample results in the final report.

17) Data Records Management

17.1 All data resubmittal forms and job documentation including Service Requests, Chain of Custody forms, Sample Acceptance Check forms and hardcopy electronic mail messages must be filed in the project file. Final reports, revised reports, and final invoices are stored electronically.

17.2 All laboratory and client documentation must be retained for a minimum of five years.

18) Contingencies for Handling Out of Control Data

18.1 When analysis quality control results (CCV, MB, LD, and LCS recoveries) are out-of-control If the associated samples are within holding time, re-analyze the sample. Alternatively, evaluate the effect on the sample results and report the results with qualifiers and/or discuss in the case narrative as detailed below.

18.1.1 CCV Refer to Section 16.6. The LCS should be in control in order for any results to be reported with an out of control CCV (biased high).

18.1.2 Method Blank If an analyte in the blank is found to be out of control and the analyte is also found in associated samples, those sample results shall be "flagged" in the report. If the analyte is found in the blank but not in the sample and all other quality control meets acceptance criteria then the results for the sample may be reported without a qualifier. However, if other QC is out of control then an evaluation must be made and the results reported accordingly.

18.1.3 Laboratory Control Sample If the samples are analyzed with an out of control LCS, then all reported analytical results must be "flagged" with the appropriate data qualifier and/or discussed in the case narrative.

18.1.4 Laboratory Duplicate The appropriate data qualifier must be included for results associated with an out-of-control laboratory duplicate and/or discussed in the case narrative.

18.2 When sample quality control results are out-of-control

Examine the sample results for matrix interference and for carryover. Reanalyze the sample(s) and/or reanalyze the sample(s) at a lower aliquot. If the out-of-control results are due to matrix interference, report the results with a matrix interference qualifier.

Holding time qualifiers must be included for those samples not analyzed within holding time.

19) Method Performance

19.1 An on-going assessment of method performance is conducted in order to ensure that the laboratory is capable of reporting results which are acceptable for its intended use. Validation of the method is confirmed by the examination and provision of objective evidence that these requirements are met.

19.2 Method Detection Limit (MDL)

The procedure used to determine the method detection limits are as stated in the *Code of Federal Regulations* (40 CFR 136 Appendix B) as defined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. MDLs can be obtained using standards at a concentration of about 25ppm to 100ppm and making at least seven replicate measurements of the compounds of interest, computing the standard deviation, and multiplying this value by the appropriate Student's t value for 99 percent confidence.

The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

19.3 Accuracy and Precision

Refer to Section 16.10 for information on replicate precision criteria for method performance. Single laboratory accuracy is presented as the second source initial calibration verification standard, which meets the method performance criteria of 15%.

20) Summary of Changes

Table 20.1			
Revision Number	Effective Date	Document Editor	Description of Changes
06.0	09/20/14	C. Humphrey	2.1 – Revised first sentence to include glass bottles
			4.1 – Updated MSDS to Safety Data Sheets
			4.2 – Updated Material Safety Data Sheets (MSDS) to Safety Data Sheets (SDS)
			6.2 – Changed 'canister pressurization' to 'pressurization'
			8.1 – Revised to include glass sampling bottles
			8.3 – Revised 'specially prepared canisters' to 'specially prepared containers'
			12.7.1 – Replaced canister with container to be inclusive of Bottle Vacs
			12.7.2 – Added second paragraph
			12.12 – Included Bottle Vacs to method modifications
			Attachment 4 – Updated limits in Table 2

21) References and Related Documents

21.1 *SOP for Handling Consumable Materials*, SOP ID ADM-CONSUM

21.2 *SOP for Training Policy*, SOP ID CE-QA003

21.3 *SOP for Laboratory Storage, Analysis, and Tracking*, SOP ID ADM-LabSAT

21.4 *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters*, SOP ID SMO-CanCert

21.5 *SOP for Manual Integration Policy*, SOP ID CE-QA002

21.6 *SOP for the Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*, SOP ID CE-QA011

21.7 *SOP for Nonconformance and Corrective Action*, SOP ID CE-QA008

21.8 *SOP for Data Review and Reporting*, SOP ID ADM-DATA_REV

21.9 EPA Method 3C, "Determination of Carbon Dioxide, Methane, Nitrogen, and Oxygen from Stationary Sources".

21.10 TNI Standard, Volume 1 – Modules 2 and 4, 2009.

22) Appendix

22.1 Attachments

Attachment 1 – Training Plan

Attachment 2 – Initial Calibration Checklist

Attachment 3 – Data Review Checklist

Attachment 4 – Target Analytes with Corresponding Method Detection and Reporting Limits and Control Limits

Attachment 1

Training Plan

Training Plan for Analysis of Hydrogen and Helium by GC/TCD

Trainee _____ Trainer _____ Instrument _____

1. Read SOP (VOA-HHe) Trainer ____ Trainee ____ Date _____
2. Read Method: EPA Method 3C Trainer ____ Trainee ____ Date _____
3. Demonstrated understanding of the scientific basis of the analysis

   Gas chromatography Thermal Conductivity Detector Trainer ____ Trainee ____ Date _____
4. Demonstrated familiarity with related SOPs Trainer ____ Trainee ____ Date _____
   - SOP for Batches and Sequences
   - SOP for Making Entries Onto Analytical Records
   - SOP for Manual Integration Policy
   - SOP for Significant Figures
   - SOP for Nonconformance and Corrective Action
   - SOP for Performing MDL Studies and Establishing Limits of Detection and Quantitation
5. Observe performance of SOP Trainer ____ Trainee ____ Date _____
   - ___sample preparation (gas-phase dilutions)
   - ___standard preparation
   - ___analytical sequence setup
   - ___initial calibration and initial calibration verification
   - ___continuing calibration verification
   - ___sample analysis
   - ___EnviroQuant introduction
   - ___data reduction and reporting
6. Perform SOP with supervision Trainer ____ Trainee ____ Date _____
   - ___sample preparation (gas-phase dilutions)
   - ___standard preparation
   - ___analytical sequence setup
   - ___initial calibration and initial calibration verification
   - ___continuing calibration verification
   - ___sample analysis
   - ___EnviroQuant use
   - ___data reduction and reporting
7. Independent performance of the SOP Trainer ____ Trainee ____ Date _____
   - ___sample preparation (gas-phase dilutions)
   - ___standard preparation
   - ___analytical sequence setup
   - ___initial calibration and continuing calibration verification
   - ___sample analysis
   - ___EnviroQuant proficiency
   - ___data reduction and reporting
   - ___initial demonstration of competency
     - ___ Four consecutive laboratory control samples
8. Instrument operation and maintenance Trainer ____ Trainee ____ Date _____
   - ___gas chromatograph and capillary column installation
   - ___detector (TCD) setup and maintenance
   - ___data system

Attachment 2

Initial Calibration Checklist

Analysis: Hydrogen and Helium

ICAL Date: _____

Instrument: ☐ GC8 ☐ GC_____

Initial Calibration Checklist

Analyst			Reviewer
☐	1.	Is the required documentation in the ICAL file?	☐
	☐	Sequence report	☐
	☐	Blank analysis Quantitation Report	☐
	☐	Calibration Status Report (aka Calibration History) – Initial	☐
	☐	Response Factor Report	☐
	☐	Quantitation Report for each calibration standard (including manual integration documentation – before and after printouts)	☐
	☐	ICV Quantitation Report and Evaluate Continuing Calibration Report (aka Percent Diff. report)	☐
☐	2.	Was the ICAL performed continuously (i.e., not interrupted for maintenance or sample analysis)?	☐
☐	3.	Was the ICAL performed within 24 hours?	☐
☐	4.	Were the standards analyzed from low concentration to high concentration?	☐
☐	5.	Are all the analytes in the blank analysis < MRL?	☐
☐	6.	Does each analyte's ICAL include a minimum of 5 consecutive concentrations?	☐
☐	7.	For each analyte, is there only one value used for each calibration level?	☐
☐	8.	If a point is dropped, is information noted in the ICAL explaining the reason?	☐
☐	9.	Does this follow the laboratory's point dropping policy (including re-analysis within 24 hrs)?	☐
☐	10.	For each analyte, is the lowest standard's concentration at or below the MRL?	☐
☐	11.	For each analyte, are there no levels skipped?	☐
☐	12.	Does the calibration curve give a %RSD of <15%?	☐
☐	13.	For the ICV analysis, is the percent recovery for each analyte 85-115%?	☐
☐	14.	Are all peak integrations including manual integrations (per *SOP for Manual Integration Policy*) acceptable? *If so, initial and date the appropriate pages.*	☐

COMMENTS:

Analyst _____ Secondary Reviewer _____

Date _____ Date _____

Attachment 3

Data Review Checklist

Hydrogen and Helium by Modified EPA Method 3C
Data Review Checklist

Analysis Date _____ Instrument _____
Client _____ QC level _____
Project # _____ Due Date _____

Analyst **Reviewer**

Initial Calibration

☐ 1. Referenced ICAL the most recent ICAL performed? ☐
☐ 2. Referenced ICAL peer reviewed and all associated documentation including the ICAL review checklist available for review? ☐
☐ 3. All associated requirements within the specified limits? ☐

Continuing Calibration

☐ 4. CCV raw data submitted? ☐
☐ 5. Was the %D for the CCV ±15% between the calculated and actual concentration? ☐
☐ 6. Was a CCV analyzed at the beginning of the sequence, every 10 samples and the end of the sequence? ☐
☐ 7. Does the RT for each analyte for the CCV fall within 0.33min of the mean RT of the ICAL? ☐

Sample Data

☐ 8. Is all sample data present and correct? ☐
___Sample raw data?
___All target analyte responses within calibration range?
___All peak integrations acceptable?
___All manual integrations flagged and properly documented?
If so, initial and date.
___Does the RT of each compound fall within the RT window using the CCV as the absolute RT and within 0.33 minutes of the most recent CCV?
___All calculations correct?
___First quantitation report initialed and dated by analyst?

QC Data

☐ 9. Duplicate sample analyzed 1 per 20 or fewer samples? ☐
☐ 10. Lab Dup - For analyte concentrations > 10x the MRL, is the RPD <15%? ☐
☐ 11. LCS – % recoveries within 85%-115% or within lab generated limits, if available? ☐
☐ 12. All the analytes in the MB <MRL? ☐

Reporting Information

☐ 13. Sample Prep and Analysis Observations/Case Narrative Summary completed if applicable? ☐
☐ 14. Appropriate flags indicated on a Sample Prep and Analysis Observations/Case Narrative Summary form if applicable? ☐

COMMENTS:

Analyst _____ Secondary Reviewer _____

Date _____ Date _____

Attachment 4

Target Analytes with Method Detection and Reporting Limits
and Control Limits

TABLE 1

Method Detection and Reporting Limits

ANALYTE	MDL (ppm)	MRL (ppm)
Hydrogen	4.4	25
Helium	3.8	25

Note: These values may change with each new MDL study performed.

TABLE 2

Control Limits

Analyte	LCS - LCL (%R)	LCS -UCL (%R)	LD (RPD)
Hydrogen	85*	115*	15*
Helium	63	131	18

* Fixed limits until enough points have been accumulated.

Note: Limits may change after the revision of this document; therefore, refer to the most recent limits.

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)
REFERENCED METHOD:	EPA TO-15
SOP ID:	VOA-TO15
REV. NUMBER:	24.0
EFFECTIVE DATE:	06/03/2017

Proprietary - Uncontrolled Copy

STANDARD OPERATING PROCEDURE

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

EPA TO-15

SOP ID:	VOA-TO15	Rev. Number:	24.0	Effective Date:	06/03/2017

Approved By: Wida Ang — Date: 5/25/17
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Approved By: [signature] — Date: 5/26/17
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Approved By: [signature] — Date: 5/31/17
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Approved By: [signature] — Date: 5/31/17
Laboratory Director - Kelly Horiuchi

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DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)

1) Scope and Applicability

1.1 This procedure is based on and incorporates the requirements detailed in EPA Compendium Methods TO-15 and TO-14A and is used to quantify a wide range of volatile organic compounds (VOCs) in gaseous matrices collected in gas collection bags (method modification) and specially prepared stainless steel canisters or glass bottles. This method typically applies to ambient concentrations of VOCs 0.50ug/m3 (down to 0.10ug/m3 for low level ambient analyses) and above for the SCAN mode and 0.010ug/m3 and above for the SIM mode; however, refer to Tables 3 and 3A for the specific laboratory initial calibration ranges for each target compound. The method requires VOC enrichment by concentrating up to one liter of a sample volume, with a virtually unlimited upper concentration range using dilutions from source level samples.

In this document, Tables 2 and 2A (see Note 1 below) list compounds that can be determined by this procedure along with their corresponding laboratory method reporting limits (MRLs) and method detection limits (MDLs). The reported MRL may be adjusted higher; however, the capability of achieving lower MRLs for specific project requirements must be thoroughly demonstrated (by an acceptable initial calibration and method reporting limit check standard) and documented as long as the MRL is higher than the current method detection limit for each compound. Additional compounds may be analyzed according to this procedure as described in the referenced methods as long as the requirements of this document are adhered to; however, if a compound is not listed in the TO-15 method, refer to Note 1 below. The number of samples that may be analyzed in a 24-hour period is about twenty. The number of sample results that may be reduced in an eight-hour day is approximately twenty.

2) Summary of Procedure

2.1 The analytical method involves using a high-resolution gas chromatograph (GC) coupled to a mass spectrometer (MS). The GC/MS utilizes a linear quadrupole system, which allows for it to be operated by either continuously scanning a wide range of mass to charge ratios (SCAN mode) or by Select Ion Monitoring mode (SIM), which consists of monitoring a small number of ions from a specified compound list.

An aliquot of an air sample is concentrated on a solid adsorbent trap (either cryogenically or fan cooled glass beads or stronger adsorbents at higher temperatures) to collect the analytes of interest. To remove co-collected water vapor, the concentrated sample then goes through a water removal (dry purge) step. After the sample is pre-concentrated on a trap, the trap is heated and the VOCs are thermally desorbed onto a refocusing cold trap. The VOCs are then thermally desorbed onto the head of a capillary column once the cold trap is heated. The oven temperature (programmed) increases and the VOCs elute and are detected by the mass spectrometer.

Mass spectra for individual peaks in the total ion chromatogram are examined with respect to the fragmentation pattern of ions corresponding to various VOCs including the intensity of primary and secondary ions. The fragmentation pattern is compared with stored spectra taken under similar conditions, in order to identify the compound. For any given compound, the intensity of the primary fragment is compared with the system response to the primary fragment for known amounts of the compound. This method

utilizes the internal standard calibration technique; refer to Section 3.16 for a complete definition.

3) Definitions

3.1 Cryogen A refrigerant used to obtain sub-ambient temperatures in the VOC concentrator and/or on front of the analytical column. Liquid nitrogen (cryogen) is used for this purpose and it has a boiling point of -195.8°C.

3.2 Gauge Pressure Pressure measure with reference to the surrounding atmospheric (barometric) pressure, usually expressed in units of psig. Zero gauge pressure is equal to atmospheric pressure.

3.3 MS-SCAN Mass spectrometric mode of operation in which the gas chromatograph (GC) is coupled to a mass spectrometer (MS) programmed to SCAN all ions repeatedly over a specified mass range.

3.4 MS-SIM Mass spectrometric mode of operation in which the GC is coupled to a MS that is programmed to scan a selected number of ions repeatedly [i.e., selected ion monitoring (SIM) mode].

3.5 Analytical Sequence The analytical sequence describes exactly how the field and QC samples in an analytical batch are to be analyzed.

3.6 Neat Stock Standard A purchased, single component assayed reference material having a stated purity used to prepare working calibration standards.

3.7 Stock Standards Solution A concentrated solution of one or more target analytes at a known concentration purchased from a reputable commercial vendor. Stock standard solutions are used to prepare working calibration standards.

3.8 Intermediate Calibration Standard A solution of one or more target analytes at a known concentration prepared either from one or more neat stock standards or from one or more stock standards solutions.

3.9 Working Calibration Standard A solution of all the target analytes at a known concentration prepared either from one or more intermediate calibration standards and/or from one or more stock standard solutions.

3.10 Calibration or Standard Curve A calibration or standard curve is a graph which plots the concentration of a compound (or an analyte) versus the instrument response to the compound.

3.11 Initial Calibration Verification (ICV) Standard A solution prepared in the laboratory containing known concentration(s) of analytes of interest. The solution is prepared from neat stock standards and/or stock standards solutions which are from a different source than the standards used to prepare the working calibration standards.

3.12 Continuing Calibration Verification (CCV) Standard A working calibration standard which is analyzed at specific intervals in order to verify that the instrument continues to meet the calibration criteria.

3.13 Field Sample A sample collected and delivered to the laboratory for analysis.

3.14 Manual Integration This term applies to a data file in which setpoints have been changed and reintegration has occurred under the changed setpoints; baselines have been adjusted; peak integration start and stop "ticks" have been changed; peak area, or peak height, are changed after the time of data collection and data file generation.

3.15 Batch Quality Control (QC) Batch QC refers to the QC samples that are analyzed in an analytical batch of field samples and includes the Method Blank (MB), Laboratory Control Sample (LCS) and Laboratory Duplicate (LD).

3.16 Internal Standard Calibration Compares the instrument responses from the target compound in the sample to the responses of specific standards (called internal standards), which are added to the sample or sample preparation prior to analysis. The ratio of the peak area (or height) of the target compound in the sample or sample preparation is compared to a similar ratio derived for each calibration standard.

3.17 May This action, activity, or procedural step is neither required nor prohibited.

3.18 Must This action, activity, or procedural step is required.

3.19 Shall This action, activity, or procedural step is required.

3.20 Should This action, activity, or procedural step is suggested, but not required.

3.21 SOP Standard Operating Procedure

3.22 Service Request A form generated, at the time of sample receipt, which details pertinent information such as client name, address, contact, client and laboratory sample identifications, sampling and receipt dates and times, requested analyses, sample type, canister pressures (initial and final), and the service request number (unique number for each submitted job) and serves as an inter-laboratory "custody" form which accompanies all samples throughout the laboratory.

3.23 Selectivity Selectivity of a method refers to the extent to which it can determine particular analyte(s) in a complex mixture without interference from other components in a mixture. Another definition is the extent to which a particular method can be used to determine analytes under given conditions in the presence of other components of similar behavior.

3.24 Limit of Detection (LOD) The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%. (DoD Clarification). For consistency purposes, the LOD may be referred to as the MDL once it is reported; however, full verification will be on file in the laboratory per the procedures detailed in this document.

3.25 Limit of Quantitation (LOQ) The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard. (DoD Clarification). For consistency purposes and since the LOQ and MRL are equivalent with regards to laboratory procedure, the LOQ will be referred to as the MRL in this document and once it is reported. Full verification will be on file in the laboratory per the procedures detailed in the document.

3.26 Detection Limit (DL) / Method Detection Limit (MDL) The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type 1 error) is 1%. (DoD Clarification). For consistency purposes, the DL may be referred to as MDL. Also, as far as reporting is concerned the MDL will be raised up (where necessary) to the verified LOD per the procedures defined in this document and reported accordingly.

4) Health and Safety Warnings

4.1 Refer to the laboratory's Environmental, Health and Safety Manual as it makes reference to the safe handling of chemicals, Safety Data Sheet (SDS) location, and the laboratory waste management plan for the safe disposal of chemicals and samples.

4.2 Pollution Prevention and Waste Management

All waste disposals shall be carried out in accordance with the requirements detailed in the *SOP for Waste Disposal.* In addition, canisters must be cleaned in accordance with the requirements detailed in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters.*

4.3 This procedure may include CHEMICAL, OPERATIONAL and/or EQUIPMENT hazards. Employees must review and understand the following hazards and their preventive measures prior to proceeding with this activity. Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

HAZARD ASSESSMENT		
Job Task 1	**Hazards**	**Preventative Measures**
Standard and Sample Preparation. Compounds, mixtures of compounds, standards, surrogates, and samples.	Exposure to potential health hazards through absorption through skin. Inhalation hazards.	Reduce exposure through the use of gloves and fume hoods. Safety glasses must be worn when working in the prep lab. Care should be taken when handling standard material in a neat or highly concentrated form. Personal protective clothing (safety glasses, gloves, and lab coat) are required when handling standard material in neat form. Consult Safety Data Sheets (SDS) for compounds being handled in this procedure. Be familiar with proper safety precautions.
Job Task 2	**Hazards**	**Preventative Measures**
Working with Liquid Nitrogen: Turning valves and handling tubing and fittings that have been in contact with cryogen.	Can cause serious tissue damage (frostbite) with only a few seconds of contact.	Wear neoprene or leather gloves. Valves on cryogen dewars should be opened slowly so leaky fitting can be identified.
Job Task 3	**Hazards**	**Preventative Measures**
Working with Pressurized Gases: Using and moving compressed gas cylinders.	Gas leak, fire, and explosion. Personal injury due to falling during transport.	All cylinders must be secured in an upright position to a wall or immovable counter with a chain or a cylinder clamp when not in use. Keep safety caps on when cylinders are not in use. A handcart must be used when transporting cylinders. The cylinder must be secured to the handcart with a chain or belt. The regulator should never remain on small "D" size cylinders following use. Full cylinders must be kept separate from empty cylinders. Flammable gases (i.e. pressurized hydrogen) must be clearly labeled. Flammables and oxidizers must be separated by a ½-hour fire wall or by at least twenty feet.
Job Task 4	**Hazards**	**Preventative Measures**
Glass syringe use	Skin lacerations and punctures.	Proper use of syringes should be part of employee training for this SOP. Care should be taken to avoid personal injury as a result or improper handling techniques.

5) Cautions

5.1 A maintenance log will be kept documenting maintenance performed on each analytical system. The serial numbers of each instrument shall be recorded, and each log entry must include a description of the maintenance performed and be initialed by the analyst performing or observing/authorizing maintenance by an outside contractor.

The instrument maintenance log must be kept current. An entry shall be made in the appropriate log every time maintenance is performed (no matter the extent). The entry in the log must include.

(a) The date of maintenance
(b) Who did the maintenance
(c) Description of the maintenance
(d) Proof that the maintenance activity was successful

A notation of a successful tune and continuing calibration or initial calibration and the file number that accompanies the data will serve as proof that the maintenance is complete and the instrument is in working order.

The extent of the maintenance is not important, however, it is important that a notation be included for each maintenance activity such as changing a column, tuning the instrument, changing the pump oil, cleaning the source, ordering a part. In addition, a notation should be made in the logbook stating that no samples were analyzed during the days that the instrument was down and no active maintenance was being conducted (i.e., where no other notation was made in the logbook for those days).

5.2 Concentrating Trap

Routine maintenance includes periodic solvent cleaning of the Silco steel lines in the valve oven if contamination is suspected. Also, periodic replacement of the multi-sorbent or partial replacement of the trap if analyte specific deterioration is detected is required. See Attachment 5 for trap packing instructions. For specific trap information refer to the instrument maintenance logbook and electronic method manual.

After repacking, the trap should be baked at 265°C for a minimum of three hours (or until a clean blank is generated) and a partial repacking requires baking (at 265°C) the trap for a minimum of 20 minutes (or until a clean blank is generated).

5.3 GC System

Column performance is monitored by observing both peak shapes and column bleed. Over time, the column will exhibit a poor overall performance, as contaminated sample matrices are analyzed. The length of time for this to occur will depend on the samples analyzed. When a noticeable decrease in column performance is evident and other maintenance options do not result in improvement, the column should be replaced (see Section 9.5). Whenever GC maintenance is performed, care should be taken to minimize the introduction of air or oxygen into the column.

Clipping off a small portion of the head of the column often improves chromatographic performance. When cutting off any portion of the column, make sure the cut is straight and “clean” (uniform, without fragmentation) by using the proper column-cutting tool. When removing any major portion of the column, which will affect the retention times and elution characteristics, a change in instrument conditions may be required to facilitate nominal analytical activity.

Declining performance can also be due to ineffective column ferrules, which should be replaced when a tight seal around the column is no longer possible. This can be detected with the use of a leak detector.

5.4 Mass Spectrometer

The Mass Selective Detector (MSD) ion source requires periodic cleaning to maintain proper performance. Symptoms of a dirty ion source include difficulty keeping the MSD in tune and fluctuating internal standard areas. The vacuum system should be serviced every six months, including changing the pump oil and checking the molecular sieve in the back-streaming trap.

5.5 Instrument Tuning

The instrument is tuned with guidance from the procedure described in the HP Operations Manual, when necessary.

5.6 Computer Troubleshooting

Computer care and troubleshooting is conducted by the IT department. Refer to Section 9.6 for the computer hardware and software requirements.

Computers are selected to meet or exceed operating system and or acquisition software requirements. Periodic upgrades of memory are performed to maintain or improve system performance and reliability. Upgrades may be performed on systems until instrument hardware configurations become the limiting factor.

Basic Troubleshooting Outline:

1) Document occurrence and severity in IT Log
2) Interview user(s)
3) Investigate any available logs (Event Logs, Acquisition Logs, etc.)
4) Determine if problem is isolated (single user or acquisition) or widespread (multi user or network).
5) If multiple possibilities exist for cause, then eliminate in systematic manner.
6) Hardware issues are addressed with component replacement (beginning with most suspect portion).
7) Software issues are addressed first with internet investigation (user blogs, software source updates/findings).
8) Network issues are investigated from the Server, to Switch, to Network Card; utilizing all available managed devices to help discover possible failure points.
9) In some cases, system corruption may require reload or complete system replacement.
10) Finalize documentation in IT Log with actions taken
11) Perform periodic follow-up with User and review any log found to have suspect events that suggested source of issue.

6) Interferences

6.1 Summa Canisters

Canisters shall be stored in a contaminant free location and shall be capped tightly during shipment to prevent leakage and minimize any compromise of the sample. The pressure/vacuum is checked prior to shipment and upon receipt from the field. Any problems with the sample from the field are noted and the Project Manager contacted.

Also, canisters must be cleaned and certified to be free from target analytes before being shipped to the field for sample collection. The procedure is described in detail in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* (refer to this procedure as well as Section 16.7 for the acceptance criteria).

Current laboratory practice entails the segregation of 6L canisters into ambient (low) level and source levels. All the ambient canisters are used for low level (indoor air, ambient

air) projects and not intentionally for soil gas, SVE monitoring, or other higher level applications. It may be necessary to "retire" an ambient canister and re-assign for source level use if high concentrations are encountered. This decision will be made by management based on analytical concentrations and what compounds were encountered at these levels. If the level of any analyte is detected above 5,000ug/m*3* in the ambient can, then the supervisor/team leader must be contacted to determine if the canister(s) is to be retired. If retirement is decided upon, make a notation on the sample tag (or other color coded tag) of each canister in question. The notation must contain the analyte, threshold levels and retirement from ambient use (initial and date notation) so that the canister conditioning/management department may properly execute the retirement.

6.2 Analytical System

The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running humidified zero air blanks. The use of non-chromatographic grade stainless steel tubing, non-PTFE thread sealants, or flow controllers with buna-N rubber components must be avoided.

6.3 Carbon Dioxide

Excessive levels of carbon dioxide present in a sample may interfere with analysis by freezing up the cryogenic trap. A smaller aliquot must be analyzed to eliminate this problem, or the sample should be analyzed using the higher temperature multi-adsorbent trapping technique which allows carbon dioxide to pass.

6.4 Gas Collection Bags

This procedure covers the use of gas collection vessels such as Tedlar® or Mylar® bags. However, due to the nature of these types of bags it is not recommended that clients use this option for ambient air samples. Sample collection bags made out of ®Tedlar have contaminants that are inherent to the manufacturing process. The two main contaminants are phenol and N,N-Dimethylacetamide. However, this only becomes a problem when the concentration levels in the sample are low ppbv such as ambient air monitoring samples where more of the sample usually has to be concentrated and analyzed. To minimize the loss of sample integrity, a 72-hour hold time has been incorporated into the procedure.

6.5 Glassware

Interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware results in discrete artifacts and/or elevated baselines in the detector profiles should be minimized. All glassware associated with this method must be scrupulously cleaned to avoid possible contamination. The cleaning shall be performed in accordance with the procedure outlined in the *SOP for Glassware Cleaning*. The use of high purity water, reagents, and solvents helps to minimize these problems.

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP may perform analysis, interpretation and peer review of the results. Data reduction and/or peer review may be performed by another qualified employee. This employee must be familiar with the analytical technique and have completed a data review training plan to ensure familiarity with specific analysis and requirements.

7.2 The supervisor/manager must ensure that method proficiency is documented initially and whenever significant changes in the instrument type, personnel, and matrix or test method are made.

7.3 The department supervisor/manager or designee shall perform final review and sign-off of the data.

7.4 Demonstration of Capability

All analysts must be trained in accordance with the guidelines detailed in the *SOP for Training Policy*. Demonstrations shall also be performed in accordance with the 2009 TNI Standards (Volume 1 Module 4 Section 1.6) and DoD Quality Systems Manual. Attachment 1 shall be used to document the training plan for new analysts' initial demonstration. Additionally, these demonstrations are performed anytime there is a change in instrument type, personnel or method.

Once performance is found to be acceptable, a required certification statement must be completed by the QA Manager and either the immediate supervisor or Laboratory Manager and retained on file as a demonstration of compliance.

7.4.1 Quarterly Demonstration A demonstration of method sensitivity must be performed *quarterly on each instrument* performing this method.

1) A spike at the current LOD must be analyzed.

2) Verification of precision and bias at the LOQ must be performed.

Refer to Section 11.1.4.2 (LOQ) and 12.14.1 (LOD) for additional information on how these demonstrations are to be performed as well as the acceptance criteria.

7.4.2 Annual Demonstration Each analyst must perform a demonstration of capability initially and annually. For the initial demonstration analyze four LCS standards at 1-4x the MRL (LOQ) either concurrently or over a period of days as a verification of precision and bias of the quantitation range. The standard deviation (n-1) and average percent recovery of the four replicates are compared against the method requirement for precision (±25%) and current laboratory control limits for bias/LCS.

7.4.3 Change in Personnel, Instruments, Method and/or Matrix The requirements in Sections 7.4.1 and 7.4.2 must be performed per the schedule noted and when there is a change in personnel, instruments, method or matrix. "Change" refers to any change in personnel, instrument, test method, or sample matrix that potentially affects the precision and bias, sensitivity, or selectivity of the output (e.g., a change in the detector, column type, matrix, or other components of the sample analytical system, or a method revision).

All completed attempts at this demonstration must be completed and turned into the QA department for retention.

8) Sample Collection, Handling, and Preservation

8.1 Air samples are collected in the field and delivered to the laboratory and shall be collected in either a specially prepared, leak-free, stainless steel pressure vessel (with valve) of desired volume (e.g., 6L), a glass sampling bottle (Bottle Vac, Entech Inntruments) or a sample collection bag (Tedlar). Canister samples may either be grab or time integrated (using a variable flow controller, refer to the *SOP for Flow Controllers and Critical Orifices*) utilizing the canister vacuum to draw the sample. Bags require the use of an upstream pump or a "lung machine."

8.2 There are no special preservation requirements for either canisters, Bottle Vacs or bags. However, bags should be stored in an environment free from puncture or deterioration sources (by hanging them from clips), labeled with the specific service request number, in accordance with the *SOP for Laboratory Storage, Analysis and Tracking*. Canisters and bottles should be stored on the appropriate shelves until they are to be analyzed.

8.3 Sample collection bags must be analyzed within 72 hours from the confirmed time of sampling. Samples received by the laboratory shall be analyzed within 30 days of sampling or sooner if project specific requirements dictate. Programs, which have shorter recommended or required hold times, include the Department of Toxic Substances Control (DTSC), which advises a 72 hour hold time. The Minnesota Pollutions Control Agency (MPCA) and EPA Region 9 both require a 14 days hold time. Additionally, the MPCA does not allow the use of Tedlar bags for sampling or sample dilution. The DTSC requirement is an advisory notice, but the laboratory shall make every effort to comply. However, the following statement shall be added to each report where sample analyses do not meet the 72 hour hold time and the client project is intended to comply with DTSC requirements. "The recommended 72-hour hold time for the analysis of TO-15 was exceeded per the DTSC and LARWQCB Advisory – Active Soil Gas Investigations document dated January 28, 2003; however, this specific hold time statement is advisory and not considered as regulation. In addition, the samples were analyzed within the EPA Method TO-15 stated requirement of 30 days."

9) Equipment and Supplies

9.1 Additional instruments and/or differing models may be utilized as long as they are equivalent and meet the minimum requirements of this document.

9.2 Gas Chromatograph (GC)

An instrument capable of temperature programming, with a column oven that may be cooled to sub-ambient temperature at the start of the gas chromatographic run to result in the resolution of the VOCs.

Hewlett Packard 5890 Series II Plus
Hewlett Packard 6890 Series
Hewlett Packard 6890A Series
Agilent 6890N Series
Agilent 7890A Series
Agilent 7890B Series

9.3 Autosampler

Tekmar-Dohrmann AUTOCan Autosampler:	14-ACAN-074
Markes Autosampler:	UNITY 2/CIA Advantage
Concentrating Trap (cryogenic trap, built-in):	14-6938-020
Cryofocusing Module w/split valve:	14-6520-A00
GAST Vacuum Pump:	DOA-P104-AA or equivalent

9.4 Mass Spectrometer (MS)

A MS capable of scanning from 34 to 350 amu every second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Bromofluorobenzene (BFB) which meets all of the criteria when 50ng or less of BFB is injected onto the GC/MS system.

Hewlett Packard 5972 Series
Hewlett Packard 5973 Series
Agilent 5973N
Agilent 5973 *inert*
Agilent 5975B *inert*
Agilent 5975C *inert*
Agilent 5977A

9.4.1 Ionization Gauge Controller

- Agilent: 59864B
- Granville-Phillips 330 Ionization Gauge Controller: 330001/2/3
- Hewlett Packard Ionization Gauge Controller: 59864B

9.5 Analytical Column

Any analytical column capable of separating the compounds of interest may be used. The capillary column should be directly coupled to the source of the mass spectrometer. The following are suggested columns; an alternative column may be used as long as sufficient peak resolution and separation is achieved.

- Restek Rxi-1ms Fused Silica Capillary Column; 30m x 0.25mm ID 1.0µm film thickness

  OR

- Restek Rxi-1ms Fused Silica Capillary Column; 60m x 0.25mm ID 1.0µm film thickness

9.6 Data Systems

IBM-compatible PC with Windows 95/98/NT/XP/7 (Microsoft Office EXCEL version 2003 or newer) and Hewlett Packard Chemstation software including EnviroQuant with Extracted Ion Current Profile (EICP), National Institute of Standards and Technology (NIST) library (2011 version or newer) or equivalent.

9.7 Canister Pressurization Station

Vacuum/Pressure Gauge [0 to –30 inHg; 0-90 or 100 psig]

9.8 Canister Sampling Devices

Refer to the *SOP for Flow Controllers and Critical Orifices* for specific calibration and other pertinent information.

- VICI Condyne Model 300 Flow Controller
- Critical Orifices (Laboratory manufactured)

9.9 Gas Collection Devices

- Lab Commerce, Aerosphere Model S6L, 6.0L Summa Passivated Canisters or equivalent
- Lab Commerce, Stabilizer Model 22.4L, 2.4L Canisters or equivalent
- Restek Corporation, #24203, 3.0L Silco Canisters or equivalent
- Tedlar bags – 0.5L, 1L, 3L, 5L, 10L, 25L, and 40L (other sizes are available; however, the volumes that are listed encompass the majority of the bags supplied and the samples submitted to the laboratory).

9.10 Dynamic Dilution System

- Entech Dynamic Diluter Model 4620A
- Toshiba laptop computer Model 2210CDT/6.0 and Software NT460

10) Standards and Reagents

10.1 Reagents and Equipment

10.1.1 UHP Grade Helium (99.999%) (GC carrier gas, preconcentrator purge/sweep gas, pressurization gas)

10.1.2 Cryogen - Liquid nitrogen from bulk tank or 50 psig dewars (used to cool preconcentrator traps)

10.1.3 UHP/Zero Grade Air (canister pressurization)

10.1.4 ASTM Type II Water, DI water or equivalent

10.1.5 UHP Grade Nitrogen (99.999%) (additional pressurization gas, based on other methods requested – modification to method)

10.2 Standards

Standards are prepared for both SCAN and Selective Ion Monitoring (SIM) modes according to the procedures detailed in this section. The preparation of standards for the analysis of air samples is carried out by following the procedure, "Preparation of Gas Phase Standards for Ambient Air Analysis", Application Note, Spring 96, Vol. 6.5, *Tekmar*-DOHRMANN AutoCan User's Manual. Neat standards that are used for making trace gas standards must be of high purity; generally a purity of 98 percent or better is commercially available.

10.2.1 Instrument Performance Check, Internal Standard and Surrogate Spiking Mixture
Prepare a standard solution of p-Bromofluorobenzene (BFB-used as both a tune check and surrogate compound), bromochloromethane, chlorobenzene-d5, and 1,4-difluorobenzene, 1,2-dichloroethane-d4(surrogate), and toluene-d8(surrogate) at 500μg/m^3 each in humidified zero air (Section 9.2.1.2). Prepare this standard according to the procedure outlined in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note. This standard may also be prepared from a neat cocktail as in Section 10.2.2.2.1 or as stated in Section 10.2.1.3.

10.2.1.1 An intermediate standard is prepared from neat compounds in a glass static dilution bottle (SDB). After the volume of the SDB is determined, calculate the mass of each compound to be spiked to achieve a final concentration of 5.0μg/ml. Then use the density of each neat compound to calculate the microliter amount to be spiked into the SDB. The SDB is then heated for a minimum of one hour at ~60˚C to completely volatilize all components.

Concentration of the intermediate standard prepared in a SDB is 5.0μg/mL. The amount required to achieve this concentration is determined through the use of the following equation.

$$A = \frac{(C)(V)}{D} \qquad \text{(Equation 1)}$$

Where:

A Amount of each compound required to achieve the desired concentration of the standard in the SDB (µL)
C Desired concentration of SDB (µg/mL)
V Actual volume of the SDB (mL)
D Density of the compound in question (µg/µL)

Example:

Calculate the amount of neat bromochloromethane needed to achieve the final concentration of 5.0µg/mL of that compound in the SDB.

V = 2010mL
D = 1934.4µg/µL
C = 5.0µg/mL

$$A = \frac{\left(5.0\,\frac{\mu g}{mL}\right)2010\,mL}{1934.4\,\frac{\mu g}{\mu L}} = 5.2\mu L$$

Density (µg/µL)	Compound
1934.4	Bromochloromethane
1170.1	1,4-Difluorobenzene
1157	Chlorobenzene-d5
1307	1,2-Dichloroethane-d4
943	Toluene-d8
1593	BFB

10.2.1.2 The Working standard is prepared in a Summa canister by spiking an aliquot of the stock SDB standard (Section 10.2.1.1) using a heated gastight syringe. Connect a cleaned, evacuated Summa canister to a source of pure diluent gas (humidified zero air) using a Teflon line with a stainless steel tee directly above the canister valve. One port of the tee is fitted with a septum. Spike the SDB stock and following removal of syringe a small flow of diluent gas to flush the spike into the can. Pressurize the can to positive 83.3 psig with humid zero air, and allow the contents to equilibrate for approximately 24 hours before using.

Concentration of the working standard prepared in a Summa canister is 500ng/L. The final pressure of the canister is 83.3psig; therefore, the pressurized volume is 40L, which is obtained through the use of the following equation.

PV = PDF(V) (Equation 2)

Where:

PV Pressurized canister volume (L)

PDF Pressure Dilution Factor, where PF = $\frac{P_{atm} + P_f}{P_{atm} + P_i}$

P_f Final Canister Pressure

P_i Initial Canister Pressure

V Volume of canister at 1atm

P_{atm} Atmospheric Pressure = 14.7psig

Example:

$$\frac{14.7 + 83.3}{14.7 + 0}(6L) = 40L$$

In order to prepare the canister with a concentration of 500ng/L, it must be determined how much of the intermediate standard is required. This is achieved through the use of the following equation.

$$A = \frac{(F)(V)}{(C)\left(1000\frac{ng}{\mu g}\right)} \qquad \text{(Equation 3)}$$

Where:

F Desired concentration of working standard (ng/L)
V Pressurized Volume of Canister (L)
C Concentration of prepared SDB (µg/mL)
A Amount of standard (mL) of the SDB required to obtain the desired working standard concentration

Example:

$$A = \frac{500\frac{ng}{L}(40L)}{\left(5.0\frac{\mu g}{mL}\right)\left(1000\frac{ng}{\mu g}\right)} = 4mL$$

10.2.1.3 Currently the working standard is purchased in a cylinder at a certified concentration of 500ng/L (prepared by Linde SPECTRA Environmental Gases, Alpha, NJ).

The internal standard (IS) cylinder comes from the vendor with a one year expiration date. These compounds should be stable in the high-pressure cylinder for five years or longer so the laboratory will extend the expiration date to two years from the date of preparation. The working standards are Summa canisters filled directly from the main cylinder and are given a two month expiration period. The method utilized relative response factors for target analyte quantitation so the IS concentrations are factored out since they appear in the numerator and denominator of the final calculation.

A quantitation report with chromatogram of a TO-15 blank run will be printed as soon as a new IS cylinder is put into use and again after one year. The latter will be checked for any unexpected peaks to look for possible degradation of the IS compounds in the cylinder. These shall be kept on file with the original certificate of analysis.

10.2.1.3.1 For SCAN analyses, the working standard is filled directly into a summa canister to a pressure of 70 to 80 psig.

10.2.1.3.2 For SIM analyses, the working standard is diluted and pressurized with humid zero air to the desired concentration using Equation 2 in Section 10.2.1.2. Typical concentrations will be 20ng/L, 40ng/L or 50ng/L.

10.2.2 Initial Calibration (ICAL) Standard Prepare the primary source calibration standards in Summa canisters with nominal concentrations of 1ng/L (optional), 20ng/L and 200ng/L for analyses in SCAN mode and 0.1ng/L, 5.0ng/L, and 200ng/L for analyses in Selective Ion Monitoring (SIM) mode for each of the target analytes. Differing injection volumes will create the standard concentrations listed in Tables 3 (SCAN) and 3A (SIM) of this document. The full list of analytes which are analyzed according to this method can also be found in Tables 2 (SCAN) and 2A (SIM).

Standards are prepared by diluting the stock standard with humid zero air into a Summa canister. The stock standard is a certified custom-blended cylinder (prepared by Linde SPECTRA Environmental Gases, Alpha, NJ). Refer to Tables 3 and 3A for the list of analytes and certified concentrations in the purchased cylinder.

10.2.2.1 Working standards are prepared into Summa canisters using the Entech Dynamic Diluter. Turn on the power to the diluter one hour prior to using to allow for the components to come to thermal equilibrium. Connect the computer and start the software. Connect a Zero Air source to the humidification chamber (flow controller #1). Connect stock standard cylinder#1 to flow controller #2 inlet. Open the cylinder valves. Adjust the inlet pressures to 50 to 60psig.

Standard Concentration Selection: The concentration of the three working standards prepared in Summa canisters should be 200ng/L, 20ng/L and 1ng/L (depending on the dynamic range of the initial calibration include 1ng/L if a 0.08ng and 0.4ng on column standard is desired or this standard may be used for the 0.5ng/L concentration as well) for SCAN and 0.2ng/L, 4.0ng/L, and 200ng/L for SIM.

Position 1 – Total Air Flow (Zero Air)
Position 2 – Standard Flow (Purchased Standard One)
Position 3 – Standard Flow (Purchased Standard Two if Applicable)
Position 4 – Total Air Flow (Zero Air) (utilized if preparing a two dilution standard)
Position 5 – Diluted Standard Flow (utilized if preparing a two dilution standard)

Step1: Determine the required flow rate of the stock standards (positions #2 and #3). The range must be from 5 to 50sccm (standard cubic centimeters per minute, same as ml/min). The flows listed below are guidelines to be used for the default standard flow (based on the desired standard concentration) and were chosen based on the ultimate final

dilution required and limitations of the Dynamic Diluter (flows must be from 150 to 2000ml/min.).

Desired Standard Conc.	Default Standard Flow
200ng/L	50ml/min
100ng/L	50ml/min
20ng/L	20ml/min
5.0ng/L	10ml/min
4.0ng/L	8ml/min
1ng/L	50ml/min; 20ml/min (See Note 1 below)
0.2ng/L	10ml/min; 20ml/min (See Note 1 below)

Note 1: For the 1ng/L and 0.2ng/L standards (or any standard requiring more than a 400X dilution of the stock), a slightly different procedure is performed. In order to prepare these standards, a double dilution must be performed which involves taking the primary dilution flow and making a secondary dilution of that using the diluent gas. Unscrew the cover of the dilutor and connect the first mass flow controller as well as the tubing to re-route the first dilution output from the final standard Summa canister to the 2nd dilution chamber. Refer to example 2 for the calculation guidelines to prepare a two dilution standard.

Example 1: Prepare a 200ng/L working standard. The concentration of each stock standard is 1000ng/L.

Step 2: Determine the required dilution factor for each stock.
Dilution factor = Stock Conc. (ng/L) / Desired Standard Conc. (ng/L)
Dilution Factor = 1000ng/L / 200ng/L = 5

Step 3: Calculate Total Flow
Total Flow= (stock std. flow-see table above)*(Dilution Factor)
Total Flow=50ml/min*5 = 250ml/min

Step 4: Calculate Diluent Air Flow
Air Flow=Total Flow-(Sum of stock std. flows-purchased cylinders)
Air Flow=250ml/min-(50+50)ml/min = 150ml/min

Example 2: Prepare a 0.2ng/L working standard. The concentration of each stock standard is 1000ng/L.

Step 2: Determine the required total dilution factor for the 0.2ng/L standard.
Dilution factor = Stock Conc. (ng/L) / Desired Standard Conc. (ng/L)
Dilution Factor = 1000ng/L / 0.2ng/L = 5,000

The two dilutions must be performed which total the dilution factor calculated above. Since the flow for the Diluter is restricted to a maximum of 2000ml/min, the total flow (as calculated in Step 3 below) cannot exceed 2000ml/min; therefore, the dilutions must be chosen accordingly.

Step 3: Calculate Total Flow
Total Flow = (stock std. flow-see table above)*(Dilution Factor)
Total Flow (Dilution 1) = 10ml/min*200 = 2000ml/min

For the 2nd dilution take the stock standard flow selected for dilution 1 for the two purchased cylinders (10ml/min each based on the desired final concentration) and add them together (10ml/min + 10ml/min for 20ml/min) to get the stock standard flow for the 2nd dilution.

2nd Dilution Factor Needed = Total Dilution/1st Dilution
2nd Dilution Factor = 10000/200(1st dilution) = 50
Total Flow (Dilution 2) = 20ml/min*50 = 1000ml/min

Step 4: Calculate Diluent Air Flow
Air Flow=Total Flow-(Sum of stock std. flows-purchased cylinders)
Air Flow=2000ml/min-(10+10)ml/min = 1980ml/min (Dilution 1)
Air Flow=1000ml/min-20ml/min = 980ml/min (Dilution 2)

Position 1 = 1980ml/min
Position 2 = 10ml/min
Position 3 = 10ml/min
Position 4 = 980ml/min
Position 5 = 20ml/min

Step 5: Enter flow rates in the appropriate fields in the Entech software. Start flows by clicking the "GO" button in the top right of the window. Allow flows to equilibrate for at least fifteen minutes, then attach an empty canister to the outlet port and open the valve. The outlet pressure will be displayed in the lower right of the window, in units of psia. Close the canister valve when the pressure reaches 30psia. There is a relief valve on the diluter that will open when the pressure reaches 35psia, so the canister will still be usable if the valve is not closed in time.

10.2.2.2 When analysis of additional (extra) compounds are requested which are not in the purchased stock cylinders, the following preparation instructions should be used. In addition, the internal standard / surrogate standard may also be prepared in this manner (Sections 10.2.2.2.1 – 10.2.2.2.2) as mentioned in Section 10.2.1.

10.2.2.2.1 *Equi-mass "soup"* (contains compounds in equal mass amounts) or *cocktail* prepared from the neat compounds for a large number of components. If additional SIM compounds are requested, the same cocktail may be used.

Cocktail Preparation:

Step 1: This cocktail is prepared by combining 25mg of each neat compound into a small glass vial. Use a microliter syringe to transfer each compound, cleaning with solvents in between. Put the vial in the freezer between aliquots to minimize volatilization. Take the density of each compound into account to determine the actual amount of each compound to spike into the cocktail by using the following equation.

$$S = \frac{A}{D} \qquad \text{(Equation 4)}$$

Where:

S Actual spike amount (µL)
A Desired amount for each compound (mg)
D Density (mg/µL); refer to Table 2 for the density

Example: The actual volume of acrolein to add to the cocktail is calculated by the following.

$$S(\text{Acrolein}) = \frac{25\,mg}{\left(0.840\,\frac{mg}{\mu l}\right)} = 29.8\mu L$$

Step 2: The concentration of each compound in the cocktail is determined by the following equation.

$$C = \frac{A}{V}\left(1000\,\frac{\mu g}{mg}\right) \qquad \text{(Equation 5)}$$

Where:

C Concentration of cocktail (µg/µL)
A Amount of each compound (mg)
V Final volume of cocktail (total spike volumes of each compound) (µL)

Example:

$$C = \frac{25\,mg}{631.8\,\mu L}\left(1000\,\frac{\mu g}{mg}\right) = 39.569\mu g/\mu L$$

10.2.2.2.2 *An intermediate standard* is prepared from neat compounds by spiking individual compounds into a glass static dilution bottle (SDB) as described in Section 10.2.1.1 or spiking an aliquot of a cocktail into the SDB. The spike amount of a cocktail is determined by using the following equation.

$$S = \frac{C_1 V}{C_2} \qquad \text{(Equation 6)}$$

Where:

S Spike amount required in order to obtain the desired concentration (µL)
C_1 Desired concentration of SDB (µg/mL)
C_2 Concentration of cocktail (µg/µL)
V Volume of SDB (L)

Example: Determine the spike amount of the cocktail required to achieve the desired intermediate standard concentration.

$$S = \frac{\left(1\frac{\mu g}{ml}\right)(2010ml)}{27.81\frac{\mu g}{\mu L}} = 72.28\mu L$$

10.2.2.2.3 *Intermediate Standard Preparation (Gaseous Compounds)* As an alternative to the glass SDB method, if the extra compounds needed to be analyzed are gases at room temperature, use a gastight syringe to prepare an intermediate standard in a 1L Tedlar bag filled with humidified zero-grade air. Use the molecular weight of the compound to calculate the microliter amount to be spiked into the bag to achieve desired concentration. The spike amount is determined by using the following equation.

$$S = \frac{C * V * 24.46}{M * \left(1000\frac{ng}{\mu l}\right)}$$

S	Spike amount required in order to obtain the desired concentration (µl)
C	Desired concentration (ng/L)
V	Volume of the Tedlar Bag (1L)
M	Molecular Weight of the compound
24.46	Molar Volume of gas at 25°C, 1atm

Example:

Make a 100,000ng/L intermediate standard of Chloro-difluoromethane (Freon22) in a Tedlar Bag, where M=86

$$S = \frac{100{,}000\frac{ng}{L} * 1L * 24.46}{86 * \left(1000\frac{ng}{\mu l}\right)} = 28.44\mu l$$

10.2.2.2.4 *The Working standard* for extra compounds is prepared in a Summa canister by spiking an aliquot of the intermediate standard (glass SDB or Tedlar bag) using a heated gastight syringe. The preparation of these standards shall follow the instructions detailed in Section 10.2.1.2. The concentrations for working standards are usually 20 and 200ng/L, however different concentrations can be chosen which work best for a particular project.

10.2.3 Initial Calibration Verification (ICV) - (Laboratory Control Sample - LCS) Prepare a secondary source standard (either a different manufacturer or different lot from the same manufacturer as the initial calibration standard) using the same procedures as the primary source. The ICV/LCS working standard should contain each target analyte present in the calibration working standard. Prepare the ICV/LCS working standard at a concentration of 200ng/L. Differing injection volumes account for the allowed concentrations listed in Table 4 for SCAN and 4A for SIM. The preparation of this standard shall follow the instructions detailed in Section 10.2.2, using the certified second-source standard cylinder.

10.2.4 Continuing Calibration Verification (CCV) Standard The CCV is the same as the initial calibration working standards detailed in Section 10.2.2.

10.2.5 Screening Standards Recommended procedure: Prepare a 0.5ug/mL and/or a 3.0ug/mL concentration standard so that the GC may be calibrated utilizing a few levels (may include approximately 0.5ng, 150ng and 600ng). However, other concentrations can be prepared depending on the desired range.

Any of the desired standard concentrations (primary and secondary) may change as long as the equations and the appropriate densities remain the same.

10.3 Storage and Expiration Dates

- All standards that are to be stored in a freezer shall be stored at ≤-10˚C for DoD projects.
- Neat Stock Liquids are stored at < -10˚C (-10˚C to -20˚C) as specified by the manufacturer or for a period of five years.
- Equi-Mass Primary Stock Standard is a cocktail or soup of neat compounds (containing compounds in equal mass amounts) used to in preparing intermediate gas phase standards and shall be stored in the freezer at < -10˚C (-10˚C to -20˚C) for up to six months. This is assuming that the soup is sealed with a septum-containing screw cap or Mininert™ valve. The selection of the compounds for the soup should be performed in accordance with the guidelines in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.
- Purchased Stock Standards Cylinders must be stored at laboratory temperature for a period of 2 years or as specified by the manufacturer before vendor re-certification or purchase of new standards. Expiration dates of the cylinders must be entered into the yearly wall calendar located next to the cylinders. Analysts must verify that the assigned expiration dates of prepared standard canisters do not exceed the parent standard expiration date.
- Intermediate Calibration Standards prepared by static dilution must be stored in an oven at a temperature of approximately 60°C to ensure analyte vaporization. Every time a standard is prepared from the static dilution bottle (SDB), the concentration changes. To increase the useful lifetime of an SDB standard, remove volumes of 25mL or less. The volume removed can be manipulated by increasing the SDB concentration or by adjusting the canister final volume/pressure. Depending upon the volume removed, an SDB intermediate standard is stable for approximately two months as long as new working standards made from this standard continue to meet acceptance criteria. These bottles must be in the oven for a minimum of one hour prior to use in preparing working standards. The guidelines for the storage and expiration date for the intermediate calibration standards are stated in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.
- Prepared Stock / Intermediate Calibration Standards prepared in Summa canisters (1000ng/L) may be stored at laboratory conditions for up to three months in an

atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

- Calibration or Working Calibration Standards prepared in canisters may be stored at laboratory conditions for one month in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

11) Method Calibration

11.1 Initial Calibration

The initial calibration is performed to determine instrument sensitivity and the linearity of the GC/MS response for the target compounds.

Initial calibration requirements are as follows:

1. A minimum of 5 concentrations must be used to calculate the calibration curve.
2. An initial calibration must be performed at a minimum initially per instrument, annually thereafter or whenever the continuing calibration verification standard does not meet the acceptance criteria.
3. Highest concentration, together with the lowest concentration, defines the calibration range.
4. The method reporting limit for any reported analyte must be at >/= the lowest calibration point.
5. The initial calibration event may not be interrupted by maintenance.
6. Only one value per concentration may be used.
7. Analyze calibration standards from lowest to highest concentration.
8. All ICAL analyses must be completed within the 24-hour tune window.
9. If 5 calibration standards are in the ICAL, one standard may be re-analyzed. If 6 to 10 calibration standards are in the ICAL, two calibration standards may be re-analyzed.
10. One of the calibration points from the initial calibration curve must be at the same concentration as the continuing calibration verification standard.
11. The upper end of the calibration range must not exhibit any peak saturation for any analyte or the range must be lowered accordingly.
12. The initial calibration model must be linear calibration using average of response factors and cannot be changed for any reason.
13. Point dropping policy
    - Minimum of 5 consecutive concentrations must be used to calculate the calibration curve.
    - Lowest concentration must be at or below the MRL (LOQ) and may not be dropped unless the MRL is changed to the concentration of the remaining lowest standard.
    - Points at the high end may be dropped, but doing so lowers the calibration range.
    - Points may not be dropped from the interior of the curve unless an assignable cause (i.e., gross dilution error, missing internal standards, purge malfunction, standard preparation error, or instrument malfunction) is accounted for and documented. In these instances, all the analytes in that calibration standard must be dropped from the calibration curve as the corrective action (the reason must be documented and the results maintained with the documentation for the final ICAL).

- Dropping individual compound points from the upper or lower end of the calibration range to improve linearity is not considered an error correction. The reason for dropping these points does not need to be documented but the ICAL documentation must state the revised calibration range if the MRL must be adjusted or the calibration range is lowered for a particular compound. This must be documented on the ICAL Review Checklist.

  When an individual compound point is dropped from an ICAL both the response and concentration fields in the compound database of the method must be cleared. This ensures the average ICAL RRF calculates correctly when executing the CCV check routine.

- A calibration standard may be re-analyzed if the first analysis of the standard has been dropped and other requirements in this policy are met (i.e., still within 24 hours).
- Once the ICAL has been used to calculate and report sample results it MUST not to be changed for any reason.
- It is recommended that if an analyte has a higher MRL than the lowest concentration analyzed that the low standard be automatically dropped from the curve (i.e., acetone MRL is 5, drop at least the 0.4ng point).

11.1.1 Calibration Points Analyze the calibration standards (analyze low to high) that span the monitoring range of interest of the samples. For SCAN, the range is typically 0.4ng-100ng on column; however, 0.08ng on column may be added if low level analyses are requested. For SIM, the range is 10pg on column to 50,000pg on column. The dynamic range is dependent on the sensitivity of a particular instrument as well as the required reporting limit for a given project and may be adjusted accordingly. Refer to Table 3 (SCAN) and Table 3A (SIM) for the concentrations of the compounds of interest in the initial calibration at each particular calibration concentration level.

Note: Refer to the EXCEL TO-15 Standard Concentration templates, located on the network at Q:\\TO15 Std. Concentrations\Std. Conc. Templates for both the SIM and SCAN templates. These templates must be utilized for the documentation of the standard canister concentration selection, final ICAL level concentrations and the determination of the correct injection volumes for the selected standard canister concentrations. If the primary or secondary stock standard cylinder concentrations are revised (upon re-certification or new purchases), the EXCEL spreadsheet templates, injection amounts and the ICAL concentrations in each instrument method must be adjusted accordingly. Other templates may be employed as long as they are validated and provide at least the same information.

SCAN

1. Determine if the lower end of the calibration range is to be 0.08ng or 0.4ng on column. If the low end is 0.08ng, then the 1ng/L standard must be utilized.
2. Determine if the 1ng/L or 20ng/L standard canister is to be used for the 0.4ng on column point.
3. Follow the instructions in the spreadsheet and save the file under the correct instrument folder and the initial calibration method identification.
4. Print the final ICAL concentration sheets and place into the corresponding ICAL folder

11.1.2 Recalibration Each GC/MS system must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument, if the continuing calibration verification acceptance criteria are not

met and at least annually. The following procedure must be followed when updating an initial calibration method.

1. Open the most recent method.
2. Save the method with the new ICAL method ID using the "Save Method As" option. Date used in the method ID must be the date files were analyzed.
3. Quantitate midpoint standard and check retention times and integrations. Update retention times if necessary using QEdit or Easy ID (Tools → Easy ID). Requant if any changes are made and verify all peaks are identified correctly. Print.
   a. While midpoint standard is loaded update reference spectra (Continuing Calibration → Update Reference Spectra).
   b. With midpoint standard loaded update qualifier ion ratios and retention times (Initial Calibration → Update Levels → Select Update Level and then select Retention Times (Replace) and Replace Qualifier Ion Relative Responses).
   c. If necessary adjust integration parameters prior to processing remaining ICAL points.
4. Quantitate remaining ICAL standards. Review each peak for retention time, integration, and print. Review low level standards for acceptable signal to noise ratios and high level standards for saturation.
5. All responses must be cleared from ICAL before updating (Initial Calibration → Clear All Calibration Responses).
6. Update responses for each standard level (Initial Calibration → Update Levels) or (Initial Calibration → Quick Levels Update). If Quick Levels Update is used do not requant datafiles.
7. Save method.
8. Check Response Factor Report and evaluate whether any points should be dropped following the criteria outlined in this SOP.
9. Save method if any changes are made.
10. Verify calibration files listed on Response Factor Report are correct.
11. Verify file ID, acquisition time, quant time, update time, and last update information is correct on the Calibration Status Report.

11.1.3 Analytical Window If time remains in the tune window after meeting the acceptance criteria for the initial calibration, samples may be analyzed according to the procedure described in this document (see Section 12.3.2). If time does not remain in the analytical window, a new sequence shall commence with the analysis of the instrument performance check compound (BFB) and the continuing calibration verification standard.

11.1.4 Procedure The system should be operated using temperature and flow rate parameters equivalent to those in Section 12.4. Use the standard prepared in accordance with Section 10.2.2 of this SOP. Attach the calibration standard and internal standard/surrogate canisters to the designated inlets on the preconcentrator and open the canister valves. Analyzing different volume aliquots of the calibration standards produces differing concentrations.

Analyte responses (target ion areas) are tabulated and recorded using the Enviroquant program. Quantitation ions for the target compounds are shown in Table 2 and 2A and the primary ion should be used unless interferences are present, in which case the secondary ion may be used, but the reason documented in the initial calibration file and all subsequent quantitations utilizing that ICAL must be performed using the same ion selections. Refer to Section 15.2 for the required calculations and Section 16.4 for the acceptance criteria.

11.1.4.1 Additional Requirements The procedure for performing and generating a new initial calibration method must follow a few additional requirements.

1. If any analyte lacks the appropriate sensitivity (3 to 1 signal to noise ratio) at the low end of the calibration range, this point must be dropped from the curve and the MRL/LOQ raised accordingly.
2. No detector saturation may occur for any compound; the upper calibration level must produce no saturated peaks. Exhibited by:
   - The flattening of the response for the higher concentration standards as shown on the plot;
   - The presence of a reverse tail or rise on the front part of the peak;
   - The observed actual percent ratio of the secondary ion presence is lower than the expected percent ratio; or
   - The presence of a flat topped peak and again by the decline or saturation of the secondary ion compared with the expected % recovery.

11.1.4.2 LOQ Establishment, Verification and Acceptance Criteria

1. The LOQ must be set within the calibration range (≥ low std. of the current passing ICAL) prior to sample analysis.
2. The LOQ is verified by analyzing an LOQ verification QC sample containing the analyte at 1-2 times the claimed LOQ.
2. The LOQ for each analyte must be > the analyte's LOD.
3. The verification is acceptable if:
   a. The S/N ratio is at least 3:1 for each analyte.
   b. All ion abundances are acceptable per the requirements in this document.
   c. The % recovery for each analyte is within the laboratory generated control limits or 70-130% recovery for the annual Navy LOQ verification.
4. Using from 2 to 4 LOQ verification points, calculate the ongoing %RSD to demonstrate precision at the LOQ.
5. If the LOQ verification check fails, determine and document the cause. Additional LOQ verification checks must be performed at a higher level to set a higher LOQ.
6. Turn in all LOQ verification data (quantitation reports and software reports/checks) to QA regardless of pass or fail.
7. Verify the LOQ on each instrument quarterly. Navy accreditation requires an annual LOQ verification.

11.1.5 Initial Calibration Review Analyst's calculation and assessment along with a peer review of all ICAL data and documentation as stated in Attachment 2 is required before the ICAL may be used to analyze samples. In the case where samples are placed on the autosampler and allowed to run overnight, the sample results may only be reported if the ICAL is reviewed and found to be acceptable. The ICAL checklist in Attachment 2 must be used to document the review and approval process.

Perform a review of specific aspects of the calibration which might compromise data quality such as inappropriate extension of the calibration range with detector saturation and/or a lack of sensitivity for any analyte. Analyte concentrations which do not meet the signal to noise ratio or exhibit saturation are not to be

reported and must be eliminated from the initial calibration. These instances should be followed by a short explanation regarding the reason for the omission.

11.1.6 Initial Calibration File An ICAL file is to be created for each initial calibration performed per instrument into which is placed the following ICAL documents. The file shall remain in the laboratory and be filed by instrument and date.

- ICAL Checklist filled out, reviewed and approved
- BFB tune analysis report
- Calibration status report (aka Calibration History)
- Relative Response Factor Report / Percent Relative Standard Deviation
- Quantitation report for each calibration standard (including manual integration documentation – before and after manual integration)
- ICV quantitation report and % recovery report.
- TO-15 Standard Concentration Spreadsheet (exact ICAL level concentrations and ICV concentrations)
- Any manual integration documentation

11.2 Initial Calibration Verification Standard

Verify the initial calibration by analyzing an initial calibration verification standard (ICV). This standard shall be obtained or prepared from materials acquired from a different manufacturer or lot from that of the initial calibration and prepared according to Section 10.2.3.

Analyze 50ng or less (refer to Table 4 for the secondary source standard concentrations) of the ICV standard depending on the dynamic range of a given instrument and refer to Section 15.4 for the required calculations.

12) Sample Preparation/Analysis

12.1 Sample Preparation

The pressure/vacuum is checked and the canister pressurized upon receipt by the laboratory, as needed. When necessary, canisters shall be pressurized with humidified zero grade air. However, if the samples are to be analyzed in accordance with EPA Method 3C then the samples must be pressurized with UHP Helium (refer to Section 12.9 for additional information). The client must be made aware of this in advance and given the option of either submitting two canisters for analysis or receiving a report with qualified results (TO-15 Modified).

Depending on the size of the canister and location of sampling and as specified in the SOP below, samples may be pressurized to approximately 1.0psig to 3.5psig. Additional information may be found in the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters*. Initial and final pressures are recorded in LIMS and should be repeated on the back of the sample tag. The dilution factor created by filling the sample canister is calculated using equation number 12 in Section 15.7.

12.2 Screening

The analyst must screen a sample or subset of samples if the source is of unknown origin. Typically, if the source is known to be indoor or ambient outdoor air, no screening is necessary. However, if screening is required make sure that the instrument is calibrated. A single point calibration is sufficient; however, the instrument may be calibrated utilizing a two point calibration. The ICAL points are recommended to be at approximately 0.5ng, 150ng and/or 600ng spanning the desired dynamic range. Refer to Section 10.2.5 for additional information.

Inject a 1mL or smaller aliquot of each sample into a GC/flame ionization detector (FID) system that has been calibrated with a standard containing a subset of the target analytes. This subset represents the most commonly found compounds in air samples, such as acetone, trichloroethylene, and toluene. Use the results to determine the maximum volume of sample to be analyzed by TO-15 by utilizing the following equation. Dilutions may be prepared as necessary according to Section 12.9.1.

$$I = \frac{C}{H}$$

Where:

I Injection volume (mL)
C Maximum calibration level (ng on column)
H Compound screening concentration (ng/mL)

Example: Select the compound with the highest concentration (toluene = 1.0ng/mL). If the upper calibration level is 100ng on column, then the following calculation determines the maximum injection volume to analyze.

$$\frac{100ng}{1.0ng/mL} = 100mL \text{ maximum injection volume}$$

12.3 Analytical Sequence and Data System Setup

12.3.1 Data System For the Tekmar AUTOCAN, fill in the sequence log of the Teklink program with the appropriate information. Refer to the Section 12.4.1 for the operating parameters.

For HP Chemstation, load the appropriate acquisition method for the GC/MS in the top window of the Chemstation program. Suggested GC/MS operating parameters are given in Section 12.4.2.

12.3.2 Analytical Sequence The analytical sequence must be completed for the analysis of ≤20 (19 samples including dilutions with one laboratory duplicate) field samples. A method blank (MB) shall be run to monitor for laboratory introduced contamination. There must be at a minimum a laboratory duplicate (LD) analyzed in each batch to access batch precision. The following generalized analytical sequence is to be followed:

Analytical Sequence Guideline

With Calibration	Tune Check[1] Calibration Standards (5 Standards Minimum) ICV Standard[2] (Acts as the ICV and LCS) QC Canister Checks[6] MB[7] Sample(s) – 1-19 Laboratory Duplicate[4]
With Continuing	Tune Check[1] CCV Standard[5] QC Canister Checks[6]

MB[7]
LCS[3]
MRL Check Standard[8]
Sample(s) – 1-19
Laboratory Duplicate[4]

[1] The instrument performance check solution must be analyzed initially and once per 24 hour (or as specified by the project) time period (sequence / tune window) of operation. All analyses for a sequence must be initiated (injected) prior to the expiration of the tune window.
[2] In this scenario, the ICV may also be evaluated as the LCS (differing acceptance criteria).
[3] An LCS shall be analyzed at a rate of 1 in 20 or fewer samples. The LCS is the second source calibration check standard analyzed at the lower end of the calibration curve (below the midpoint).
[4] A laboratory duplicate must be analyzed at a rate of 1 per 20 or fewer samples. The duplicate must be rotated among clients, whenever possible. Also, a duplicate laboratory control sample may be analyzed to assess precision to meet project requirements or due to sample matrix effects.
[5] A CCV must be analyzed at the beginning of every analytical sequence.
[6] Any number of QC check canisters may be analyzed in the sequence to determine a canister cleaning batch or batches acceptability.
[7] Any of the QC Check Canisters may serve as the method blank as long as the minimum requirements detailed in this document are met. A method blank shall be analyzed at a rate of 1 in 20 or fewer samples.
[8] A MRL check standard may be analyzed with each batch of 20 or fewer samples (when an initial calibration is not analyzed within the same batch). Additional information is included in Section 12.15.

Note: Client project batch specifications may require certain modifications to the analytical sequence; however, a batch may not be more lenient than that which is specified in this document.

12.4 Conditions

12.4.1 Sample Collection Conditions The suggested settings and system parameters are as follows:

Adsorbent Trap

Set Point:	35˚
Sample Volume:	up to 1L
Dry Purge:	300mL
Sampling Rate:	100mL/min (utilize for a sample injection volume of >100mL); 40mL/min (utilize for a sample injection volume of 25-100mL)
Desorb Temp.:	200˚C to 230˚C
Desorb Flow Rate:	8-10mL/min He, measured at refocuser split vent
Desorb Time:	3.0 minutes

Refocusing Trap

Temperature:	-180˚C
Injection Temp.:	160˚C

Injection Time: 1.0 min

Adsorbent Trap Reconditioning Conditions

Temperature: 265°C
Initial Bakeout: 3 hours or until clean blank is obtained
After each run: 5-8 minutes

Sample Run Time

Each analytical run is approximately 20 minutes long; the total cycle time is about 30 minutes between injections.

12.4.2 GC/MS System

Optimize GC conditions for compound separation and sensitivity.

Item	Condition
Carrier Gas	Helium
Flow Rate	1.0-1.6mL/minute
Temperature Program	Initial Temperature: ~20˚C Initial Hold Temperature: 3 minutes Ramp Rate: 5˚C/min to 80˚C 2nd Ramp: 10˚C/min to 160˚C 3rd Ramp: 20˚C/min to 240˚C for 5 min hold
Detector B (MSD *Interface)*	260˚C
Electron Energy	70 Volts (nominal)
Mass Range (Scan mode)	34 to 280 amu
Mass Range (SIM mode)	Scan masses corresponding to the target analytes
Scan Time	To give at least 10 scans per peak, not to exceed 1 second per scan.

Note: The instrument may be operated in Selective Ion Monitoring (SIM) mode if requested by the client.

12.5 Instrument Performance Check

Since the BFB tuning compound is included in the internal standard and surrogate standard canister and an autosampler is used, it is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to the reduction and approval of any data collection. The 24-hour time period for GC/MS instrument performance check and standards calibration (initial calibration or continuing calibration verification criteria) begins at the injection of the BFB, which shall be documented in laboratory records. Upon completion of the successful BFB tune, the tune report must be printed and retained on file for future reference.

The mass spectrum of BFB must be acquired in the following manner.

- Inject 50ng or less (on column)
- Three scans (peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.
- Background subtraction is conducted using a single scan prior to the elution of BFB.
- All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

- The ion abundance criteria must not be changed from the requirement stated in this document (TO-15 or TO-14A, as requested).

All subsequent standards, samples and QC samples associated with a BFB analysis must use identical instrument conditions.

12.6 Continuing Calibration Verification Standard

Verify the calibration each working day, where necessary (e.g., an ICAL was not analyzed or the tune window has closed) by analyzing a continuing calibration verification (CCV) standard from the initial calibration standard canister. The concentration of the calibration verification may be varied between the low calibration standard and the midpoint of the calibration range; however, the concentration must be at one of the levels analyzed in the initial calibration. Refer to Table 3 for the standard concentrations. Refer to Section 15.3 for the required calculations.

DoD QSM 5.1 Requirement: A CCV standard must be analyzed daily before sample analysis; after every 24 hours of analysis time; and at the end of the analytical batch run.

12.7 Canister Quality Control Check and Method Blank

The method blank must be a sample of a matrix similar to the batch of associated samples that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure, and in which no target or interferences are present at concentrations that impact the analytical results for sample analyses. Prepare a canister that has not left the building by pressuring with humidified zero air. Analyze an aliquot of one liter along with the same volume of internal standard and surrogate as standards and samples. Additionally, a blank must be analyzed whenever a high concentration sample is encountered and carryover is suspected. For all method blanks the unique laboratory barcode for the canister must be included in the sample analysis identification.

A Quality Control (QC) check canister pressurized with humidified zero air may serve as a method blank as long as the analyte concentration requirements stated in the canister quality control check section (Sections 16.7 and 16.8) and other requirements (refer to Section 16.12 for internal standard requirements) are met. Assuming continuing failure, another QC canister or a new canister must be prepared and analyzed in order to verify that no system contamination exists. For tracking purposes the unique laboratory barcode given to a canister shall be the information included in the sample analysis identification.

12.7.1 Sampling Systems Section 7.1 and 8.4 of Method TO-15 describe the setup and certification procedure for a specific sampling apparatus that has been used by the EPA for several of its large air monitoring programs. These systems are rarely used for the types of projects that make up the bulk of the laboratory's work. The vast majority of samples analyzed by the laboratory are taken into Summa canisters either as grab samples or using a simple time integrated sampling device (flow controller), as in Section 8.2.1 of the method, so these procedures are not part of the typical protocol for providing sampling materials to clients. The laboratory has developed an SOP for the cleaning and certification of the materials it provides its clients for obtaining air samples to be analyzed by method TO-15. Refer to the *SOP for Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters* for additional information.

It is this laboratory's interpretation that the sampler system certification procedure described in Section 8.4.4 of the TO-15 method applies to the specific sampling apparatus described in the method and not to the sampling procedures used by our clients. The laboratory does not maintain a dynamic calibration manifold or canister sampler apparatus as described in the method and thus

performance of the relative accuracy certification procedure described in section 8.4.4 is not possible.

12.8 Laboratory Control Sample

The laboratory control sample is a sample matrix, which is free from the analytes of interest and spiked with a standard containing known amounts of analytes. The laboratory control sample is an injection of the initial calibration verification standard. Inject the LCS (ICV) at concentrations below the midpoint of the calibration curve. Make sure that all of the pertinent information is included on the quantitation report including the sample identification (LCS), concentration, standard used, and analyst.

12.9 Sample Analysis

Prior to analysis, all sample containers (canisters and bags) should be at temperature equilibrium with the laboratory.

- Attach sample canisters to Tekmar AUTOCan using a 9/16" wrench. Bottle Vacs use a proprietary quick connect fitting (Micro-QT, Entech Instruments). Tedlar bags can be connected using soft silicone tubing or a 3/16" fitting with a reusable ferrule.
- Before opening the valve, check for leaking fittings by running the leak check program in the Teklink software. Quick connect fittings must be leak checked before connecting the sample container.
- If system is leak tight, open the canister valves and start the automated preconcentration procedure. Make sure the Chemstation data acquisition software has been readied.
- Maintain the trap at an elevated temperature until the beginning of the next analysis.

Check all target compounds using the QEdit routine in Enviroquant, making sure all extracted ion chromatogram peaks are integrated properly (see Section 12.13).

Note 1: The secondary ion quantitation is only allowed if there is sample matrix interference with the primary ion. If the secondary ion quantitation is performed, document the reasons in the instrument run logbook and/or on the quantitation report (initial and date any notation).

Note 2: Each female Micro-QT fitting must be purged after use to remove any remaining sample residue and prevent contamination from subsequent usage. Connect a male Micro-QT fitting to a source of ultrapure or carbon-filtered gas. Adjust the pressure to about 10 psig using an inline regulator. Connect the female fitting for several seconds, then remove and place in an oven kept at 60ºC until the next use. Do not heat the fitting higher than 80ºC.

SCAN Mode - The instrument is normally operated in the SCAN mode, where the following procedure may be followed.

- Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic range from 34 to 270 amu. At least ten scans per eluting chromatographic peak should be acquired. Scanning allows identification of unknown compounds in the sample through searching of library spectra. See operating conditions in Section 12.4.
- Generate a quantitation report for each run.
- If reporting Tentatively Identified Compounds (TICs), refer to Section 12.9.2 for identification criteria.

SIM Mode - When the client requests SIM mode, select SIM instead of SCAN mode and identify a minimum of two ions per analyte of interest. Also, a minimum of two ions for each internal standard and surrogate compound should be selected.

Helium Pressurization – If a canister is pressurized with helium, a correction factor is applied to sample volumes extracted from the canister via auto sampler. This is due to the difference in thermal properties between helium and air. A correction factor worksheet has been generated to determine the exact volume taken from a canister and may be found at J:\\A-GCMS\Helium Pressurization. Save file, print the sheet and include with the data. Refer to the instruction page in the template for all of the instructions and calculations including backfilled canisters.

AutoCAN Leak Checks – Canisters should be put on at least two different AutoCAN positions to confirm a "leak". In addition, the valve threads should be inspected for defects which may prevent a good seal with the AutoCAN. Once a canister has "failed" the leak check it must be tagged, an NCAR initiated, and the PM notified. Regardless of what the client or PM specifies as the fate of the sample, the canister must be put on maintenance hold to complete a full 24-hour leak check. A yellow sheet is to be completed in addition to, but not in lieu of an NCAR. This is a fixed QA procedure with no allowance for deviation.

12.9.1 Sample Dilution If any target analyte results are above the highest level of the initial calibration, a smaller sample aliquot should be analyzed. The dynamic range of volume aliquots for the automatic cryogenic concentrator is 15ml to 1L. If a volume smaller than 15ml is to be analyzed, a dilution should be made in a Tedlar bag, or the sample directly injected using a gastight syringe. Guidance in performing dilutions and exceptions to this requirement are given below.

- Refer to Section 12.4.1 (Adsorbent Trap Sampling Rate) for the required sampling rate if less than 100mL is to be analyzed.
- Use results of the original analysis to determine the approximate dilution factor required and get the largest analyte peak within the initial calibration range.
- The dilution factor must be documented (and included in the final report) and chosen in such a way as to keep the response of the analyte peak for a reported target compound in the upper half of the initial calibration range of the instrument.

Tedlar bag dilution:

- Make a dilution by filling a Tedlar bag with 1.0 liter of humidified zero air using a one-liter gas syringe.
- Calculate the volume of balance gas needed to obtain the required dilution.
- Remove the difference in the balance gas using a syringe.
- Add the calculated sample amount using a gastight syringe.

Direct injection:

- Make a direct injection by attaching a clean, humidified zero air filled Summa canister to the preconcentrator autosampler using 1/4" stainless steel or teflon tubing with a "tee" septum port. This canister should be the same canister that may be used as the method blank.
- Inject the sample through the septum while the preconcentrator withdraws a 200cc aliquot from the canister.

12.9.2 Tentatively Identified Compounds When requested, a mass spectral library search may be made for the purpose of tentatively identifying sample components not

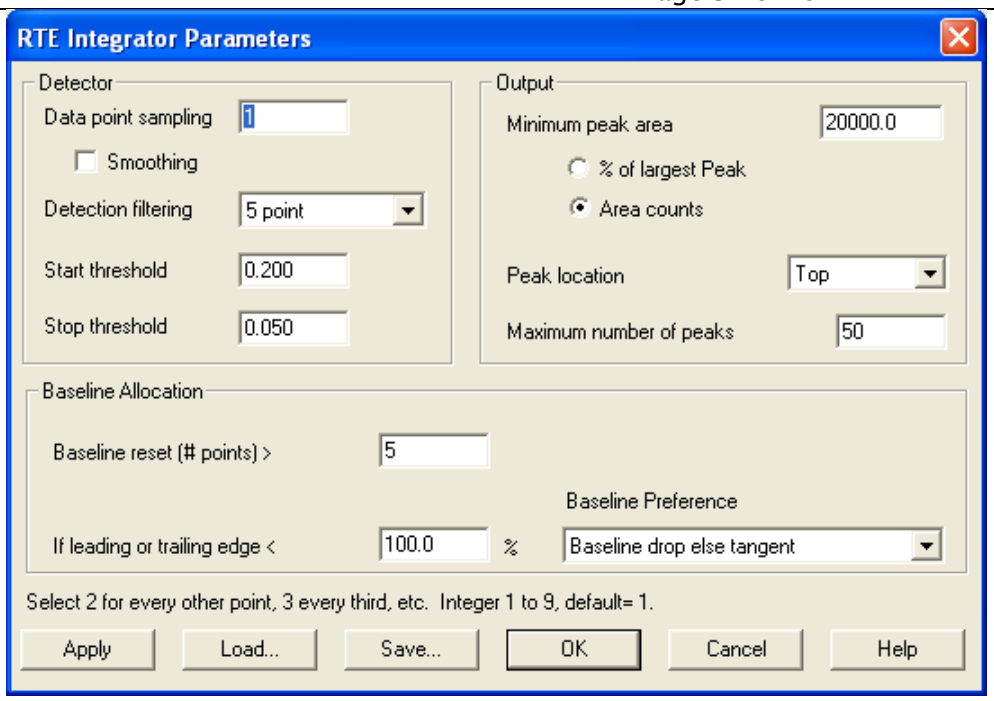
associated with the calibration standards. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system mass spectral library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Certain programs may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. The following guidelines are used for making tentative identifications.

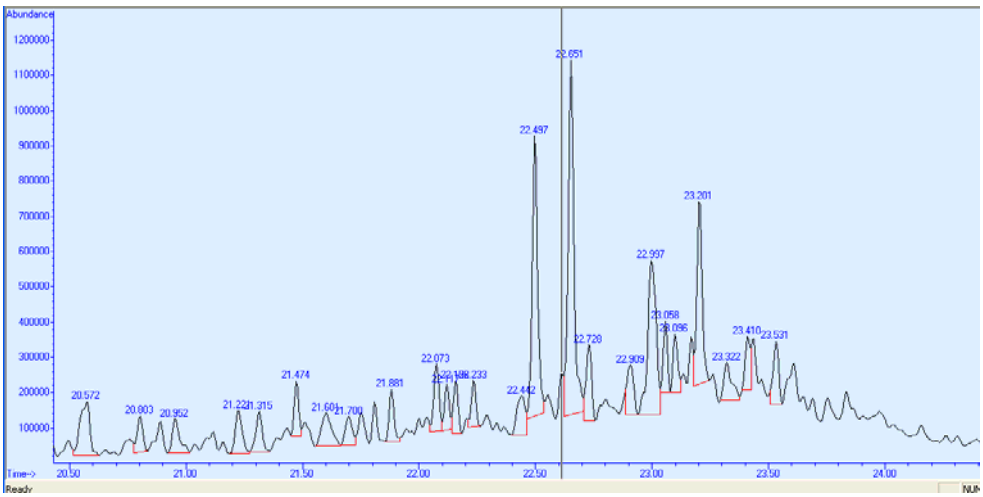
- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20%. For example, for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30 and 70%.
- Molecular ions present in the reference spectrum should be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.
- The concentration of the tentatively identified compound is estimated by assuming a response factor of 1.0 and comparing the response of the tentatively identified compound to the response of the nearest internal standard.
- If non-target analytes are not Q-deleted from the quant report, the analyst must evaluate whether these compounds should be reported as TICS.

<u>Procedure for Reporting Tentatively Identified Compounds (TICs) for samples and associated Method Blanks</u>

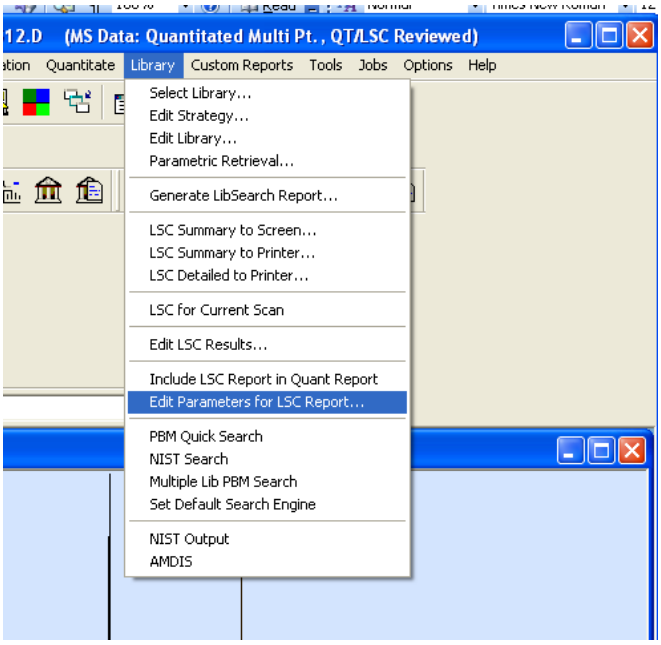
1. Load the datafile in the main Enviroquant window.
2. Load the TIC integration parameters (LSCINT.p). Typical setpoints are as shown below.



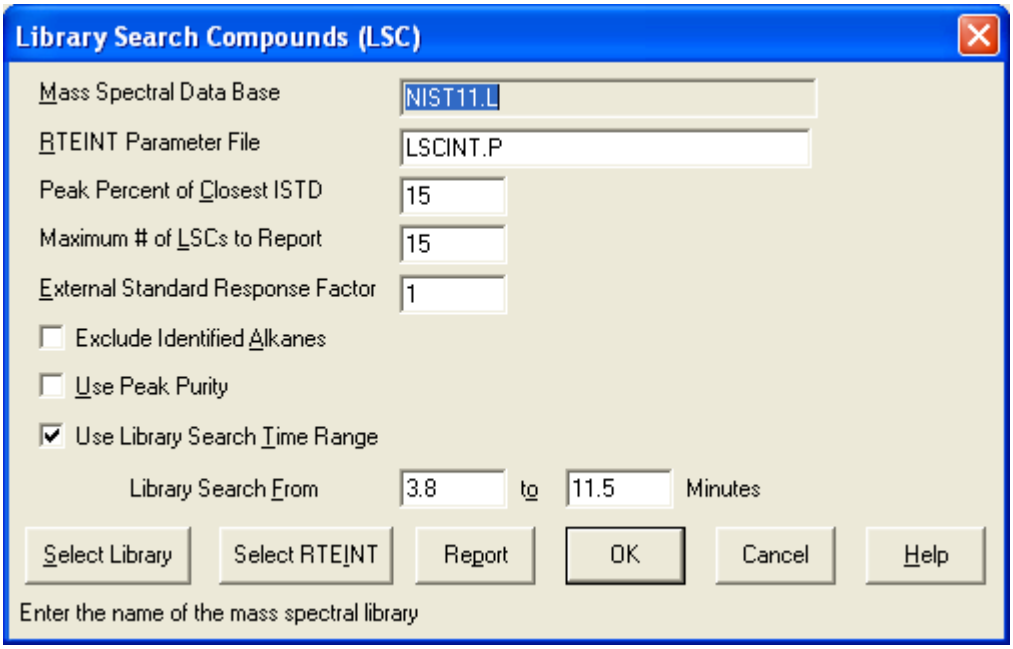
3. Integrate the chromatogram and inspect the peak integrations. Adjust the parameters as needed to achieve integration that will:
   - Resolve closely-eluting peaks that only have a small valley separating them.
   - Not include excess area below the peak in a complex matrix with an elevated baseline.
   - Include peak tailing when necessary.
   - Yield a sufficient number of peaks that will ensure that the internal standards are included.



4. Edit the parameters to be used in generation the library search report:



Select the most current mass spectral library database available, the correct integration parameters file, the area threshold (as a percent of IS area), number of peaks to report, and a time range of the chromatogram to search (set to start after the CO2 peak).



5. Run the LSC routine from the Library menu. You may choose 'LSC Summary to Screen' (Calculate/Generate Report) to get a quick view of the results and then proceed if they seem acceptable. Set the default printer to 'Adobe PDF' and then choose 'LSC Detailed to Printer'.
6. Open the pdf file and inspect the LSC summary (last page). Check the internal standard areas and confirm that they are correct. If any IS area is biased high due to a coeluting peak use the 'Edit LSC Results' routine to switch all associated TICs to use a different IS. If all three IS peaks have coelutions substitute the areas from the daily method blank in the calculations.
7. Use the LSC Summary as a guide and inspect the chromatogram in the data analysis window. Integrate the chromatogram from the Integrate menu and look for peaks that may have been missed by the LSC routine. Possible reasons for missed peaks are excessive tailing (organic acids), RT close to a target compound, coeluting peaks with no valley between them. These will need to be added manually.
8. Use the DOSCAN routine from the Tools menu to search individual missed peaks one by one. This will add them to the LSC list.
9. Go back into the Edit LSC Results routine and make any necessary changes to compound names and/or the internal standard used for quantitation.
10. Run the macro "QT '0,0,C' by clicking the Custom Tool 1 button. This will update the LSC list to the quant.csv file.
11. Run the LSC Detailed to Printer routine from the Library menu (Generate Report *only*). This will print the file to pdf.
12. Excel Reporting
    1. In Excel, open the TIC reporting template (I:\A-GCMS\TICS\System\StarLIMS_TICQ).
    2. Enter the service request number and click ok.
    3. Click the Get Samples button. Select the samples to be reported. Delete any samples that are not to be reported (right click/delete row).
    4. Click the Update PEF button.
    5. Click the Get TICs from CSV button. Enter the date analyzed and select the instrument ID.
    6. Click the Apply to all Samples button. Change the date for any sample that was analyzed on a different date.
    7. Click the Apply Instrument to all Samples button.
    8. Enter file number in column E (i.e. enter 07 for file 12301507.d).
    9. Click the Copy Data button. This copies the TIC info to the report sheets.

12.10 Duplicate

A duplicate must be analyzed to assess laboratory precision and samples selected for duplicate analysis shall be rotated among client samples, where applicable. Some projects or sample matrix issues may require the analysis of a duplicate laboratory control sample (DLCS).

12.11 Internal Standard (IS)

The concentration of internal standard added to each standard, field sample and QC sample must be consistent from that of each current ICAL standard.

12.12 Surrogates

Internal standards/surrogates must be added at the same volume for every standard, sample and QC sample. Surrogate compound recoveries are requested by a number of clients, but are more appropriately used as system monitoring compounds. This is due to the fact that the compounds are introduced directly into the analytical system and not

into the canisters or bags. It is for this reason that they are not considered to be true surrogates and a fixed window is applied. Additionally, surrogates are not included in the ICAL because they are not required by the method and are only system monitoring compounds.

12.13 Manual Integration and Q Deletion

A list of abbreviations (codes) that may be used to give a reason for performing either of these procedures are listed in the *SOP for Data Review and Reporting*.

12.13.1 Manual Integration The integration for each peak must be legally defensible and shall be checked to ensure that it has been integrated properly and consistently between samples, standards and QC samples. All peak reviews and manual integrations must follow the requirements specified in the *SOP for Manual Integration Policy* and the *SOP for Laboratory Ethics and Data Integrity.* The requirements in the above stated procedure include when manual integrations are performed, raw data records shall include a complete audit trail for those manipulations (i.e., chromatograms showing both the integration prior to any manual integrations and those depicting the corresponding manually integrated peaks), and notation of rationale, date, and initials of person performing the manual integration operation. In addition, manual integrations must be reviewed and approved by a second reviewer and the manual integrations maintained in the appropriate job file.

Reporting Requirements Certain project requirements including samples which are submitted under the Department of Defense (DoD) QSM require that the case narrative include an identification of samples and analytes for which manual integration is required. Refer to project requirements to determine if this is necessary.

12.13.2 Q Deletion Q deleting may be performed to either delete a false positive or delete non-target compounds.

12.14 Detection Limits and Limits of Detection

The MDL shall be performed in accordance with the procedure outlined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* The detection limit shall be used to determine the LOD for each analyte.

12.14.1 Performance and Acceptance Criteria

1. The MDL must be <0.5ppbV for each analyte (Method 11.11.1).
2. Following the MDL study perform a Limit of Detection (LOD) verification on all instruments (performing this method). Spike the LOD at 2-4x the MDL; the spike level establishes the LOD.
3. LOD Acceptance
   - Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio).
   - It is specific to each combination of analyte, matrix, method and instrument configuration.
   - The LOD must be verified quarterly on each instrument (spiked at LOD) using the criteria listed above.
4. If the LOD verification fails (per #3), repeat the detection limit determination and LOD verification at a higher concentration or perform and pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration.

5. The laboratory shall maintain documentation for all detection limit determinations and LOD verifications (regardless of pass or fail).

12.15 Method Reporting Limit Check Standard

It is recommended to analyze a MRL check standard at the current MRL or required MRL for the batch (per client requirements) of twenty or fewer samples if the CCV fails low for any target compound. A MRL check standard may also be required per client specifications.

This check standard can also serve as the LOQ verification if it meets the specific requirements listed in Section 11.1.4.2. Apply the requirements and retain all documentation accordingly. Refer to Attachment 4 for Minnesota specified MRL check standard criteria.

12.16 Method Modifications

Method modifications are not allowed under TNI standards; therefore, a statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAP certificate of approval. In addition, the following items are considered to be method modifications and must be reported accordingly.

- Sample collection in gas collection bags
- The pressurization of canisters with nitrogen or helium (if EPA Method 3C is requested) refer to Section 12.9.

13) Troubleshooting

13.1 Prepare new standards, check instrument maintenance, prepare a new curve as needed, etc. Refer to the corrective actions listed in Section 16 of this SOP for additional troubleshooting details.

14) Data Acquisition

14.1 Storing Electronic Data

The initial calibration data must be stored in a quantitation method (on the server) using a unique filename and may not be overwritten at any time in order to maintain an accurate audit trail. There are multiple quantitation methods, which are subsets of the compound list in Table 2. Therefore, files will be named with an eight-character notation indicating the compound list and the date of the corresponding initial calibration. In addition, all data files including method blanks, continuing calibration verification, laboratory control samples and client submitted samples files are saved in a unique sub-directory on the server.

14.2 Sufficient raw data records must be retained on file of all laboratory analyses described in this document including passing QC canister checks, tune checks, instrument calibrations, verifications, sample analyses and dilutions, QC checks, and method detection limit studies. The information that is required includes: analysis/calibration date and time, test method, instrument, sample identification, analyte identification, analyst's initials, concentrations and responses, as well as standards used for the analysis and calibrations, all manual calculations including sample dilutions and manual integrations to permit reconstruction of analyses. Information entered and reported on the quantitation report and instrument run log must be complete and accurate. All data shall be obtained following defensible and ethical practices in accordance with the most recent Quality Assurance Manual and the *SOP for Laboratory Ethics and Data Integrity*.

Note: All data records must explicitly connect data to the initial instrument calibration.

This includes all samples, continuing calibrations and QC samples.

14.3 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date and time (if the holding time is 72 hours) of analysis, instrument operating conditions/parameters (or reference to such data), analysis type, all manual calculations including dilutions and manual integrations, analyst's initials, sample preparation (pressure readings and balance gas if pressurized with helium), standard and reagent origin, receipt, preparation, and use, as well as calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions.

15) Calculation and Data Reduction Requirements

15.1 This method has specific requirements including the use of canisters; any modification must be reported accordingly. All reports that fall under the laboratory's certificate of approval (in accordance with TNI standards) must include a statement(s) clarifying any deviations from the scope of this certification. Refer to Section 15.10 for additional information and specific items, which require this clarification.

15.2 Initial Calibration

Tabulate each of the following:

15.2.1 Equation Number 1 - **Relative Response Factor (RRF):**

$$RRF = \frac{A_x C_{is}}{A_{is} C_x}$$ where:

A_x is the area response of the analyte quantitation ion.
A_{is} is the area response of the corresponding internal standard quantitation ion.
C_{is} Internal standard concentration, ng.
C_x Analyte concentration, ng.

Note: The equation above is valid under the condition that the volume of internal standard spiking mixture added in all field and QC samples is the same from run to run.

15.2.2 Equation Number 2 - **Average (or Mean) RRF:**

$$\overline{RRF} = \frac{\sum_{i=1}^{N} RRF_i}{N}$$ where:

RRF_i are the individual RRFs from each concentration level in the initial calibration curve.

N is the number of calibration concentration levels.

15.2.3 Equation Number 3 - **Standard Deviation, SD:**

SD = $\sqrt{\sum_{i=1}^{N} \frac{\left(RRF_i - \overline{RRF}\right)^2}{N-1}}$ where:

RRF_i are the individual RRFs from each concentration level in the initial calibration curve.

$\overline{RRF}$ Average (or Mean) RRF of all concentration levels in the initial calibration curve.

N total number of calibration concentration levels

15.2.4 Equation Number 4 - **Percent Relative Standard Deviation, %RSD:**

%RSD = $\frac{SD}{\overline{RRF}}(100)$ where:

SD Standard Deviation calculated in equation number 3

$\overline{RRF}$ Average or Mean RRF

15.2.5 Equation Number 5 - **Relative Retention Time (RRT):**

$RRT = \frac{RT_C}{RT_{is}}$ where:

RT_C Retention time of the target compound, seconds.

RT_{is} Retention time of the internal standard, seconds.

15.2.6 Equation Number 6 - **Mean Relative Retention Time ($\overline{RRT}$):**

$\overline{RRT} = \sum_{i=1}^{n} \frac{RRT_i}{n}$ where:

$\overline{RRT}$ Mean relative retention time (seconds) for the target compound for all initial calibration levels.

RRT_i Relative retention time for the target compound in level i.

n Number of calibration levels

15.2.7 Equation Number 7 - **Mean Area Response ($\overline{Y}$):**

$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n}$ where:

Y_i Area response for the primary quantitation ion for the internal standard for each initial calibration standard.

n number of calibration concentration levels

15.2.8 Equation Number 8 - **Mean Retention Times ($\overline{RT}$):**

$$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n}$$ where:

$\overline{RT}$ Mean retention time, seconds

RT_i Retention time for the internal standard for each initial calibration standard, seconds.

n number of initial calibration levels

15.3 Continuing Calibration Verification

- Calculate the (RRF) of each target compound using equation number 1.

15.3.1 Equation Number 9 - **Percent Difference, %D:**

$$\%D = \frac{RRFx - \overline{RRF}}{\overline{RRF}}(100)$$ where, for any given analyte:

RRF_x is the RRF from the CCV being evaluated.

$\overline{RRF}$ is the mean RRF from the current calibration curve.

15.4 Percent Recovery – ICV, LCS, Surrogates, MRL Check Standard

15.4.1 Equation Number 10 - **Percent Recovery (%R):**

%R = X/TV x 100

where
X = Concentration of the analyte recovered
TV = True value of amount spiked

15.5 Duplicate Analysis

15.5.1 Equation Number 11 - **Relative Percent Difference (RPD):**

$$\frac{x_1 - x_2}{\bar{x}}(100)$$ where:

x_1 First measurement value

x_2 Second measurement value

$\bar{x}$ Average of the two values

15.6 Internal Standards (IS)

- Calculate the mean area response $\overline{Y}$ for each internal standard using equation number 7.
- Calculate the mean of the retention times for each internal standard using equation number 8.

15.7 Pressure Dilution Factor (PDF)

15.7.1 Equation Number 12 - **PDF**, for samples collected in Summa canisters:

$$PDF = \frac{P_{atm} + P_f}{P_{atm} + P_i}$$ where:

P_{atm} is the ambient atmospheric pressure, 14.7 psi at sea level.
P_f is the final sample canister pressure, in psig.
P_i is the initial sample canister pressure, in psig. This will most often be a negative value (sub-ambient initial pressure).

15.8 Results

If a canister has been pressurized with Helium and the Tekmar AutoCan was utilized, refer to Section 12.9.

15.8.1 Equation Number 13 - For calculating analyte concentrations in a sample, the starting point is the nanogram amount generated by the HP Enviroquant software, which appears on the quantitation report.

$$ng_x = \frac{A_x ng_{is}}{A_{is}\overline{RRF}}$$ where:

ng_x is the nanogram amount of analyte *x*.
A_x is the area response of the analyte's quantitation ion.
A_{is} is the area response of the corresponding internal standard's quantitation ion.
ng_{is} is the internal standard amount, in nanograms.
$\overline{RRF}$ is the average or mean RRFs

15.8.2 Equation Number 14 - The final analyte concentration, C_x, in units of micrograms per cubic meter (µg/m³), is then calculated from the following:

$$C_x = \left(\frac{ng_x PDF}{V}\right)\left(\frac{1\mu g}{1000ng}\right)\left(\frac{1000l}{1m^3}\right)$$ where:

V is the sample volume analyzed, in liters.
PDF is the sample canister pressure dilution factor.

15.8.3 Equation Number 15 - To convert to units of parts per billion volume (ppbv):

$$ppbv = \frac{\mu g/m^3}{MW} x24.46 \qquad \mu g/m^3 = \frac{ppbv}{24.46} xMW$$ where:

MW is the molecular weight (Table 2) of the analyte, in g/mole.

24.46 is the molar volume of an ideal gas at 298 K (25 °C) and 760 mmHg (1 atm), in liters per mole (l/mol).

C_x the final analyte concentration in micrograms per cubic meter.

15.8.4 Equation Number 16 – Helium Pressurization (Injection Amount)

Applicable to canisters pressurized with helium and injected utilizing the mass flow controller of the AutoCAN. For full instructions and calculations, refer to the 1st tab of the template located at: J:\A-GCMS\Helium Pressurization\System\HE Pressurization Template.

15.9 Data Review

The analyst must review data on a real time basis for all calibration and QC data. The QC data must be evaluated by analytical sequence following the Daily QC review checklist (Attachment 3). The data shall be reviewed and the sample results calculated and assessed by one analyst and reviewed by a second qualified analyst. The Sample Review checklist (Attachment 3) is used to document sample review per service request and once completed, initialed and dated must be filed with each job file.

Initial calibrations must be reviewed in the same manner as QC data with all ICAL documentation retained in a separate file organized by instrument and date. Refer to the initial calibration checklist in Attachment 2 for the review guideline. The ICAL file must contain all the pertinent information stated in Section 11.1.6.

15.10 Reporting

The results of each test shall be reported clearly, unambiguously and objectively, and shall include all the information necessary for the interpretation of the test results and information required by this laboratory's policy, TNI standards, DoD Manual (applicable version, see reference section), client projects, and the TO-15 method including modifications, observances, data qualifiers, and certification information.

If the project requires that results be reported below the MRL (LOQ), but above the LOD all of the requirements specified for normal reporting apply (3:1 S/N ratio and ion abundance). This is regardless of the fact that the results will be qualified as estimated.

15.10.1 Analysis Observations / Case Narrative Summary Form

This form, which is included in the *SOP for Laboratory Storage, Analysis and Tracking*, may be generated when there are specific sample composition information or analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved. All data qualifiers and flags should follow those listed in the most recent Quality Assurance Manual or as defined in any client requirements.

This form may be used as a means for documentation. This form, among other information, will be reviewed when compiling the final report and case narrative. Alternatively, information may be included on the Daily QC and Sample Review Checklists (Attachment 3). All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through analysis, data reduction, and reporting.

15.10.2 NELAP\TNI Requirements

The following items do not comply with TNI standard requirements and must be reported accordingly. A statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAP certificate of approval.

- Reporting any compound which is not included in the second source standard (ICV or LCS) does not meet NELAP requirements.
- In addition, a report that contains a compound not included on the NELAP certificate of approval must also include the statement listed above.

15.10.2.1 Modifications

Method modifications are also not allowed under TNI standards; therefore, a statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAP certificate of approval. In addition, the following items are considered to be method modifications and must be reported accordingly.

- Sample collection in gas collection bags
- The pressurization of canisters with nitrogen or helium (if EPA Method 3C is requested) refer to Section 12.9.

15.10.3 Surrogates

Only report surrogates at the request of the client. If any surrogate is out of control, all samples results (with surrogates requested) associated with the surrogate must be reported with the appropriate data qualifier.

15.10.4 DoD Requirements

Report results with the appropriate data qualifiers, if samples cannot be reanalyzed for any reason. In addition and at a minimum, the following situations are to be noted in the case narrative: manual integrations, CCV out of control, and results exceeding the calibration range.

16) Quality Control, Acceptance Criteria, and Corrective Action

16.1 To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.2 Corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action*, where appropriate. Any maintenance which may alter instrument sensitivity or linearity must result in the re-analysis of the entire sequence including the tune compound, ICAL or CCV or any batch QC.

16.3 Instrument Performance Check

16.3.1 Acceptance Criteria

Refer to Tables 1 and 1A for the required ion abundance criteria.

16.3.2 Corrective Action Perform auto tune or manual tune and then re-analyze BFB. If the BFB acceptance criteria are still not met, the MS must be retuned according to the procedure outlined in the instrument user's manual. Perform necessary maintenance and make notations in the instrument maintenance logbook. It may

be necessary to clean the ion source, or quadrupole, or take other necessary actions to achieve the acceptance criteria. An acceptable tune is required for sample results to be calculated and reported.

16.4 Initial Calibration

16.4.1 Acceptance Criteria Refer to the following acceptance criteria for the initial calibration.

- The RRT for each target compound at each calibration level must be within 0.06RRT units of the mean RRT for the compound.
- The calculated %RSD for the RRF for each compound in the calibration standard must be less than 30% with at most two exceptions up to a limit of 40% (this may not be true for all projects).

  DoD QSM 5.1/Navy Requirement: The two exceptions of %RSD up to 40%, allowed by the method, are not allowed.

- For each Internal Standard the area response (Y) at each calibration level must be within 40% of the mean area response $\overline{Y}$ over the initial calibration range.
- The retention time shift for each of the internal standards at each calibration level must be within 20s of the mean retention time over the initial calibration range for each internal standard.
- All of the following information must be retained to permit reconstruction of the initial instrument calibration: calibration date, test method, instrument, analysis date, analyte identification, analyst's initials, concentration and responses, and response factors.
- All initial instrument calibrations must be verified with an acceptable ICV.

16.4.2 Corrective Action Follow the initial calibration requirements detailed in Section 11.1 for information on re-analyzing or dropping points and the restriction of maintenance performed during the analysis of the initial calibration standards.

If the initial calibration results are outside the established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed, if reanalysis of the samples is not possible, data associated with an unacceptable initial calibration shall be reported as estimated with the appropriate data qualifiers.

16.5 Initial Calibration Verification Standard (ICV)

16.5.1 Acceptance Criteria The percent recovery for each compound in the ICV must be between 70%-130% for all analytes except vinyl acetate, which must be within 50-150%. Exceptions to this allowance for the vinyl acetate recovery are project specific requirements and any DoD type project, which shall adhere to the 70-130% requirement for all target compounds.

16.5.2 Corrective Action If the initial calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column. Perform a new initial calibration if any performed maintenance has altered instrument linearity and/or sensitivity. Perform another initial calibration or if reanalysis is not possible, data associated with an unacceptable ICAL/ICV shall be reported as estimated with the appropriate data qualifiers.

16.6 Continuing Calibration Verification (CCV)

16.6.1 Acceptance Criteria All compounds must be evaluated prior to rounding. The percent difference for each target analyte must be within plus or minus 30% of the initial calibration average RRFs.

16.6.2 Corrective Action If the continuing calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources of the problem and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column.

If any corrective action and/or reanalysis fails to produce continuing calibration verification within acceptance criteria (analyzed immediately following the initial failure), then either two consecutive successful verifications must be performed following corrective action or a new initial calibration must be performed; however, refer to 16.6.2.1 below.

DOD Requirement: If a CCV fails, the laboratory must immediately analyze two additional consecutive CCVs (The two consecutive CCVs must be analyzed within one hour).

- Both of these CCVs must meet acceptance criteria in order for samples to be reported without reanalysis.
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- Corrective action(s) and recalibration must occur if the above scenario fails.
- Flagging data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

16.6.2.1 Method Reporting Limit Check Standard

If a per batch MRL check standard is analyzed due to a failing CCV or client requirement and is unacceptable for any compound (sensitivity; ratio or %D), reanalyze at the same or higher level within the same batch and report data with the CCV flag and case narrative notes accordingly. Reporting data with these conditions must be acceptable per project and client requirements otherwise corrective action must be initiated and samples reanalyzed.

Refer to Section 11.1.4.2 for annual (NELAP and Navy) and quarterly (DoD) LOQ verification requirements.

16.7 Canister Quality Control Check

The actual cleaning procedure, number of cans to select for analysis (to release a cleaning batch) and corrective actions are covered in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* and are not covered in this section. However, the procedure for analyzing and certifying a cleaning batch is included. If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, TNI Standards, and Department of Defense Quality Systems Manual – DoD QSM, etc.).

16.7.1 Scan Analyses A canister is considered "clean" for normal SCAN analyses if the analysis shows <0.2ppbv of any target analyte (analyte exceptions listed in table

below). If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, TNI Standards, and Department of Defense Quality Systems Manual - DoD QSM, etc.).

Low Level SCAN Analyses For those analytes with a MRL of 0.1ug/m3, the QC criteria of <MRL is acceptable; otherwise, <0.2ppbV is required (analyte exceptions listed in table below).

SIM Analyses Results <MRL will be acceptable as this complies with the <0.2ppbV method requirement.

DoD QSM 5.1 Requirement Each canister must be individually certified. A canister is considered clean if no reported analytes are detected at >1/2 the LOQ.

ANALYTE EXCEPTION LIST					
Compounds	ppbV	On Column (ng)	Compounds	ppbV	On Column (ng)
Target Analytes	0.2	0.50	Acrylonitrile	0.2	0.43
Chloromethane	0.2	0.41	Acetone	1.5	3.5
1,3-Butadiene	0.2	0.44	Ethanol	1.9	3.5
Acetonitrile	0.2	0.33	Vinyl acetate	0.99	3.5
Acrolein	0.65	1.5	1-Butanol	0.23	0.70
Isopropanol	0.28	0.70	Carbon Disulfide	1.1	3.5
2-Butanone	1.2	3.5			

Document the status of the check in LIMS and return the canister to the canister conditioning room. Additionally, if the check was found to be acceptable, the quantitation report must be kept on file for future reference

16.7.2 Tentatively Identified Compounds (TIC) If the batch of canisters are to be used for tentatively identified compounds (TIC) analysis, any non-target peaks present in the QC check canister analysis must be evaluated and determined to be less than the TIC reporting limit (10% of the internal standard). The concentration is estimated by assuming a RRF of 1.0 and comparing the response of the TIC to the response of the nearest internal standard.

16.8 Method Blank

16.8.1 Acceptance Criteria

- The concentration of a targeted analyte in the blank cannot be at or above the MRL, AND be greater than 1/10 of the amount measured in any associated sample. For any project that requires reported results less than the MRL, all associated measurements found in the MB should result in a qualifier; however, project requirements may differ and must be followed. Refer to DoD requirements listed below.
- The method blank should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.
- For DoD samples, the method blank will be considered to be contaminated if:

  1. The concentration of any target analyte in the blank exceeds 1/2 the reporting limit or is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
  2. The concentration of any common laboratory contaminant (acetone, ethanol, carbon disulfide, and methylene chloride) in the blank exceeds

the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); or

3. The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives.

The laboratory shall evaluate whether reprocessing of the samples is necessary based on the above criteria.

16.8.2 Corrective Action If the analyte concentration results in the blank do not meet the acceptance criteria repeat analysis with remaining QC canisters until results are acceptable or prepare a canister per Section 12.7. If the analyte results in the blank still do not meet the acceptance criteria the source of the problem must be investigated and measures taken to eliminate the source. Each method blank must be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch. Determine whether the contamination is from the instrument or due to contamination in the blank container (if results from the new can are not acceptable then the system is probably contaminated). In all cases, the corrective action (reprocessing or data qualifying codes) must be documented. However, the specific corrective action depends on the type of project the blank is utilized for; therefore, refer (below) to the reporting/reprocessing requirements.

DEPARTMENT OF DEFENSE (DoD) QSM PROJECT: Any sample associated with a blank that fails the criteria shall be reprocessed in the same or subsequent analytical batch, except when the sample analysis resulted in a non-detect. If reanalysis is not performed, the results shall be reported with appropriate data qualifier.

OTHER PROJECT TYPE: Appropriate corrective measures must be taken and documented before sample analysis proceeds. However, if this is not a possibility and the results must be reported follow the reporting requirements stated in Section 18.4.

16.9 Laboratory Control Sample (LCS)

16.9.1 Acceptance Criteria Round all results to the nearest whole number prior to determining if the acceptance criteria have been met. The percent recoveries must be within the laboratory-generated limits and are referenced in the electronic TO-15 Method Manual. However, Arizona requires the percent recovery for each compound in the LCS to be 70%-130% (to match the ICV requirement). Therefore, the ICV exception for vinyl acetate stated in Section 16.5 requires the percent recovery for AZ samples to be 50-150%.

Note: Client project requirements, AFCEE and DoD requirements shall take precedence over the AZ requirement for AZ samples. Meaning if a sample is collected for a DoD project in AZ, DoD requirements specified in this document and the project specific QAPP (if supplied) are to be followed.

DoD Requirement: In the absence of client specified LCS reporting criteria, the LCS control limits outlined in the DoD QSM Appendix C tables shall be used when reporting data for DoD projects.

16.9.2 Corrective Action If the LCS criteria are not met, determine whether the cause is instrumentation or the result of a poor injection. If the problem is instrumentation, perform maintenance and if the problem is with the injection re-analyze the LCS. DoD considers the same analyte exceeding the LCS control limits two out of three consecutive LCS to be indicative of non-random behavior;

therefore, this trend should be monitored and the appropriate corrective action taken when it occurs.

16.10 Sample Results

16.10.1 Acceptance Criteria

- Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.
- The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, initial calibration verification technical acceptance criteria described in this document.
- All target analyte peaks must be within the initial calibration range, diluted or reported with the appropriate data qualifier.

16.10.2 Corrective Action

- If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis as needed.
- If the area for any internal standard changes by more than ±40 percent between the sample and the most recent calibration, check for possible matrix interferences and re-analyze at a greater dilution. If the requirement is still not met and matrix interference is not detected the GC/MS system must be inspected for malfunction and maintenance made where necessary.
- When corrective actions are made, samples analyzed while the instrument was not functioning properly must be re-analyzed or the appropriate data qualifiers must be attached to the results.

  To the extent possible, samples shall be reported only if all of the quality control measures are acceptable. If a quality control measure is found to be out of control, and the data must be reported, all samples associated with the out of control quality control measure shall be reported with the appropriate data qualifier(s).

16.11 Laboratory Duplicate

16.11.1 Acceptance Criteria The relative percent difference must fall within ±25%. This RPD criterion also applies to duplicate laboratory control samples (DLCS).

16.11.2 Corrective Action If the duplicate results do not meet the technical acceptance criteria, perform another duplicate analysis. If the results are still unacceptable and the associated samples are not reanalyzed then all of the sample results in the associated batch must be flagged accordingly.

16.12 Internal Standards

16.12.1 Acceptance Criteria The following acceptance criteria must be applied to each run (except the ICAL – see Section 16.4).

- The area response for each internal standard in the blank must be within ±40 percent of the area response for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).

- The retention time for each internal standard must be within ±0.33 minutes of the retention time for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).

16.12.2 Corrective Action

- Internal Standard Responses If the problem is with the instrument, perform maintenance. If the problem is with a sample, check for interferences. If the response is high, it is likely that interference is present. In this case, lower the volume or aliquot of the sample and re-analyze. If the problem persists, report the results with the best quality and qualify the results. If the problem is corrected with the lower volume analysis, report those results.
- Internal Standard Retention Times If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis where required.

16.13 Surrogates

16.13.1 Acceptance Criteria Since the matrix precludes the use of true surrogates and there is no established method criterion, acceptable surrogate recoveries are based on a fixed window of 70 - 130%. This is the typical requirement from clients. Additionally, these limits are referenced in SW-846 for use as guidance in evaluating recoveries. These limits are sufficient for evaluating the effect indicated for the individual sample results.

16.13.2 Corrective Action Poor surrogate recovery should be followed by re-analyzing a smaller aliquot to mitigate any matrix interferences. Evaluate the out of control surrogate for the effect on individual sample results.

16.14 Method Reporting Limit Check Standard

16.14.1 Acceptance Criteria Per client requirements or if the CCV is biased low for any compound, then evaluate the MRL check standard. Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio). A percent difference +/-50% is recommended but program and client specific requirements must be followed if applicable.

16.15 Sample Holding Time Expired

The customer is to be notified that the sample's holding time was missed and the customer is to decide if the sample analysis is to continue. The documentation of missed holding time and the client's decision to proceed must be included in the corresponding job file. A statement dictating all holding time occurrences must accompany the sample results in the final report.

17) Data Records Management

17.1 All data resubmittal forms and job documentation including Service Requests, Chain of Custody forms, Sample Acceptance Check forms and hardcopy electronic mail messages must be filed in the project file. Final reports, revised reports, and final invoices are stored electronically.

17.2 All laboratory and client documentation must be retained for a minimum of five years.

18) Contingencies for Handling Out of Control Data

18.1 The following is specific information on how to report unacceptable data. If the data requires a data qualifier flag, as specified in this SOP, refer to Appendix D of the most recent version of the Quality Assurance Manual for the appropriate data qualifier.

18.2 Initial Calibration and/or Initial Calibration Verification

All results reported with an unacceptable ICAL must be reported as estimated and all data shall be reported using defined qualifiers or flags or explained in the case narrative accordingly.

18.3 Continuing Calibration Verification

All results associated with an unacceptable CCV (other than #1 below) must be reported with the appropriate data qualifier, flag and/or explained in the case narrative.

1. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported without a qualifier.
2. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples with detects, then those detects must be reported with a qualifier, flag and/or explained in the case narrative.
3. If however, the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, and there are associated samples that are non-detects, then those non-detects must be reported with qualifiers, flags and/or explained in the case narrative as having less certainty. However, along with the data qualifiers, the case narrative may include information stating the fact that the results were not significantly affected if:
   a. *An MRL check standard was analyzed and found to be acceptable. The MRL must be the same as that analyzed in the MRL check standard for those analytes that were biased low in the CCV. Adjust MRLs (if required), flag data and state the certainty in the case narrative where the sensitivity of the instrument was demonstrated at the MRL; therefore, results were not significantly affected.*
   b. *With the reporting limit adjusted to the next level in the calibration curve (typically 5 times higher) to prove the nonexistence of a false negative and note procedure in case narrative.*
4. If the acceptance criteria was exceeded (biased high) for the CCV and there were detectable results in a sample, the results may be "qualified" if the results exceeded the regulatory/decision limit (this is to be stated in the case narrative along with the data qualifiers or flags).
5. Data associated with a biased low CCV may be fully useable if the results reported exceed a maximum regulatory limit/decision level.

18.4 Method Blank

- If an analyte in the blank is found to be out of control and the analyte is also found in associated samples, those sample results shall be "flagged" in the report and the method blank results reported.
- If the analyte is found in the blank but not in the sample then the results for the sample may be reported without a qualifier.

18.5 Laboratory Control Sample

All results associated with an out of control laboratory control sample must be reported with the appropriate data qualifier. An indication of whether the LCS was out high or low should also be included.

18.6 Surrogate

Report sample results with the appropriate data qualifier.

18.7 Laboratory Duplicate

All batch sample results associated with an out of control laboratory duplicate must be flagged with the appropriate data qualifier.

18.8 Internal Standard

All target analytes associated with an out of control internal standard must be flagged with the appropriate data qualifier.

18.9 Estimated Sample Results

18.9.1 Sample Hold Time All occurrences of missed holding times must be included on the final report including those samples received and/or analyzed outside of the specified hold times detailed in this SOP.

18.9.2 Matrix Interference Sample data associated with matrix interference must be flagged with the appropriate data qualifier.

18.9.3 Results Outside Initial Calibration Range All sample results not bracketed by initial calibration standards (within calibration range) must be reported as having less certainty by reporting with the appropriate data qualifier.

19) Method Performance

19.1 An on-going assessment of method performance is conducted in order to ensure that the laboratory is capable of reporting results which are acceptable for its intended use. Validation of the method is confirmed by the examination and provision of objective evidence that these requirements are met.

19.2 Method Detection Limit (MDL)

The procedure used to determine the method detection limits are as stated in the *Code of Federal Regulations* (40 CFR 136 Appendix B) as defined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations are listed in Tables 2 and 2A for both SCAN and SIM modes and were obtained using spiked canisters prepared with humidified zero air, making at least seven replicate measurements of the compounds of interest, computing the standard deviation, and multiplying this value by the appropriate Student's t value for 99 percent confidence. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. All MDLs, regardless of the mode of operation, meet the method performance criteria of <0.5ppbV.

19.3 Accuracy and Precision

Refer to Section 11.4 in the referenced method for information on replicate precision criteria for method performance. Single laboratory accuracy is presented as the second source initial calibration verification standard, which meets the method performance criteria of 30%. Additionally, laboratory generated control limit data for LCSs are presented for the analytes of interest and may be referenced in the electronic TO-15 Method Manual. Refer to Section 11.1.4.2 for the accuracy and precision requirements for concentrations at the LOQ/MRL.

19.4 Selectivity

Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multidetector arrangement of these (see discussion in Compendium Method TO-14A). The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification.

It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least ten scans per eluting chromatographic peak must be acquired. Scanning also allows identification of unknown compounds in the sample by searching through library spectra.

The sample analysis using the GC/MS is based in part on a combination of retention times and relative abundances of selected ions. The retention time of each chromatographic peak should be ±0.10 minutes of the library/reference retention time of the compound. The acceptance level for relative abundance should be set at ±20% of the expected abundance. The data should be manually examined by the analyst to determine the reason for the # flag [(#) = qualifier out of range], if present and whether the compound should be reported as found or if there is matrix interference. A background subtraction may aid in this determination. Manual inspection of the qualitative results should also be performed to verify concentrations outside the expected range.

Specific selectivity information is provided in this section and document (such as relative retention time) as well as in the referenced method. Refer to the method for additional information on selectivity.

- Use NIST Library 2011 or newer version
- The *reference spectra updates* must be performed with every new ICAL utilizing the mid-level standard (minimum). If needed, the reference spectra may be updated sooner with the continuing calibration standard.
- *Retention time updates* must be performed using EasyID and not by updating to the method (InitCal \ Update Calibration). Refer to the Help selection of the software.

19.5 Demonstration of Capability

This laboratory has continuously performed this method since before July 1999. Therefore, ongoing demonstration of capable shall be performed and documented; however, the initial demonstration of method capability is not required.

19.6 Proficiency Testing (PT) Program

The laboratory shall participate in an air and emissions PT study for TO-15. The testing shall be performed in accordance with this document and meet the frequency and proficiency requirements detailed in the DoD QSM.

Proficiency testing samples including all accredited compounds are not available. Therefore, in addition to third party PT samples, intra laboratory comparisons must be performed biannually to meet the DoD QSM proficiency testing requirements. Eight QC analyses from various analysts and instruments shall be compiled and statistical validity evaluated using a Z-score.

20) Summary of Changes

Table 20.1			
Revision Number	Effective Date	Document Editor	Description of Changes
24.0	06/03/17	C. Humphrey	5.2 – Included reference to Attachment 5; changed bake time to three hours
		C. Humphrey	7.4 – Removed DoD QSM version number
		C. Humphrey	7.4.2 – Minor wording revision
		C. Parnell	9.6 – Updated NIST Library to 2011
		C. Parnell	12.4.1 – Added information to Desorb Flow Rate; changed bake time to 3 hours under Adsorbent Trap Reconditioning Conditions
		C. Humphrey	12.6 – Added DoD QSM 5.1 requirement
		C. Humphrey	12.14 – Revised to align with current procedure and SOP CE-QA011
		C. Humphrey	12.14.1 – Revised to align with current procedure and SOP CE-QA011
		C. Humphrey	15.8.4 – Updated file path
		C. Humphrey	15.10.1 – Revised to align with current procedure
		C. Humphrey	16.4.1 – Added DoD QSM 5.1/Navy requirement
		C. Humphrey	16.7.1 – Added DoD QSM 5.1 requirement
		C. Humphrey	16.8.1 – #1 changed "and" to "or" to align with DoD QSM version 5.1
		C. Humphrey	16.9.1 – Removed DoD QSM version number
		C. Parnell	19.4 – Updated NIST Library to 2011
		C. Humphrey	19.6 – Revised section
		C. Humphrey	21.7 – Updated reference
		C. Humphrey	21.8 – Updated reference
		C. Humphrey	22.2 – Included Attachment 5
		C. Humphrey	Updated Tables 2A, 3, 3A, 4, 4A
		C. Humphrey	Attachment 2 – Added #15 and renumbered; #17 revised wording
		C. Humphrey	Attachment 3 – Added #5 and renumbered; Added #12
		C. Parnell	Attachment 5 - New

21) References and Related Documents

21.1 EPA Method TO-14A, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.2 EPA Method TO-15, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.3 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, January 1999.

21.4 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Addendum, January 17, 2002.

21.5 2009 TNI Standards

21.6 *Preparation of Gas Phase Standards for Ambient Air Analysis,* Tekmar-DOHRMANN Application Note, Spring 96, Vol. 6.5.

21.7 DoD/DoE Quality Systems Manual Version 5.0, 2013; and Version 5.1, 2017.

21.8 Arizona Administrative Code, Title 9. Health Services, Chapter 14. Department of Health Services Laboratories, October 1, 2016.

21.9 Florida Department of Environmental Protection, Chapter 62-160.

21.10 Minnesota Department of Health, 4740.2065, *Standard Operating Procedures*, Statutory Authority: MS s 144.97; 144.98; History: 31 SR 446, Posted: October 09, 2006, Revised April 16, 2010.

22) Appendix

22.1 Tables

Table 1: Instrument Tune Check Ion Abundance Criteria (TO-15)

Table 1A: Instrument Tune Check Ion Abundance Criteria (TO-14A)

Table 2: Volatile Organic Compounds, EPA Compendium Method TO-15 (SCAN)

Table 2A: Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)

Table 3: Standard Concentrations (SCAN) (Primary Sources)

Table 3A: Standard Concentrations (SIM) (Primary Sources)

Table 4: Standard Concentrations (SCAN) (Secondary Sources)

Table 4A: Standard Concentrations (SIM) (Secondary Sources)

22.2 Attachments

Attachment 1 – Training Plan

Attachment 2 – Initial Calibration Checklist

Attachment 3 – Daily QC and Sample Review Checklists

Attachment 4 – State and Project Specific Requirements

Attachment 5 – Tekmar AutoCan Trap Packing Instructions

TABLE 1

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-15

Mass	Ion Abundance Criteria[1]
50	8.0 to 40.0 percent of m/e 95
75	30.0 to 66.0 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5.0 to 9.0 Percent of m/e 95
173	Less than 2.0 Percent of m/e 174
174	50.0 to 120.0 Percent of m/e 95
175	4.0 to 9.0 Percent of m/e 174
176	93.0 to 101.0 Percent of m/e 174
177	5.0 to 9.0 Percent of m/e 176

[1] All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

TABLE 1A

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-14A

Mass	Ion Abundance Criteria
50	15 to 40 percent of m/e 95
75	30 to 60 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5 to 9 Percent of m/e 95
173	Less than 2 Percent of m/e 174
174	>50 Percent of m/e 95
175	5 to 9 Percent of m/e 174
176	>95 and <101 Percent of m/e 174
177	5 to 9 Percent of m/e 176

Note: The criteria listed in Tables 1 and 1A shall be met or exceeded in order for EPA Compendium Methods TO-15 or TO-14A to be referenced.

TABLE 2 - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Bromochloromethane (IS1)	74-97-5	-	-	130	128, 132	-	-	-
Propene	115-07-1	42.08	NA	42	39,41	0.50	0.14	IS1
Dichlorodifluoromethane (CFC 12)	75-71-8	120.9	1.329	85	87, 101, 103	0.50	0.17	IS1
Chloromethane	74-87-3	50.49	0.911	50	52	0.50	0.15	IS1
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	76-14-2	170.9	1.455	135	137	0.50	0.19	IS1
Vinyl Chloride	75-01-4	62.50	0.9106	62	64	0.50	0.17	IS1
1,3-Butadiene	106-99-0	54.09	0.6149	54	39, 53	0.50	0.22	IS1
Bromomethane	74-83-9	94.94	1.6755	94	96	0.50	0.19	IS1
Chloroethane	75-00-3	64.52	0.8902	64	66	0.50	0.17	IS1
Ethanol	64-17-5	46.07	0.7893	45	46	5.0	0.80	IS1
Acetonitrile	75-05-8	41.05	0.7857	41	40	0.50	0.18	IS1
Acrolein	107-02-8	56.06	0.840	56	55	2.0	0.17	IS1
Acetone	67-64-1	58.08	0.7845	58	43	5.0	0.77	IS1
Trichlorofluoromethane	75-69-4	137.4	NA	101	103	0.50	0.17	IS1
Isopropyl Alcohol	67-63-0	60.10	0.7809	45	43	5.0	0.42	IS1
Acrylonitrile	107-13-1	53.06	0.8060	53	52	0.50	0.17	IS1
1,1-Dichloroethene	75-35-4	96.94	1.213	96	61	0.50	0.17	IS1
tert-Butanol	75-65-0	74.12	0.7887	59	57,41,43	1.0	0.33	IS1
Methylene Chloride	75-09-2	84.94	1.3266	84	49	0.50	0.17	IS1
Allyl Chloride	107-05-1	76.53	0.9376	41	76	0.50	0.16	IS1
Trichlorotrifluoroethane	76-13-1	187.38	1.5635	151	101	0.50	0.17	IS1

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Carbon Disulfide	75-15-0	76.14	1.2632	76	78	5.0	0.15	IS1
trans-1,2-Dichloroethene	156-60-5	96.94	1.2565	61	96	0.50	0.19	IS1
1,1-Dichloroethane	75-34-3	98.96	1.1757	63	65	0.50	0.16	IS1
Methyl tert-Butyl Ether	1634-04-4	88.15	0.7402	73	57	0.50	0.17	IS1
Vinyl Acetate	108-05-4	86.09	0.9317	86	43	5.0	0.65	IS1
2-Butanone (MEK)	78-93-3	72.11	0.7999	72	43	5.0	0.21	IS1
cis-1,2-Dichloroethene	156-59-2	96.94	1.2837	61	96	0.50	0.16	IS1
Diisopropyl Ether	108-20-3	102.18	0.7241	87	45,59,43	0.50	0.19	IS1
Ethyl Acetate	141-78-6	88.106	0.9003	61	70	1.0	0.35	IS1
n-Hexane	110-54-3	86.18	0.6548	57	86	0.50	0.15	IS1
Chloroform	67-66-3	119.4	1.4832	83	85	0.50	0.17	IS1
1,2-Dichloroethane-d4(S)	17060-07-0	-	-	65	67	-	-	IS1
Tetrahydrofuran	109-99-9	72.11	0.8892	72	71,42	0.50	0.20	IS1
Ethyl tert-Butyl Ether	637-92-3	102.176	0.7519	87	59,57	0.50	0.18	IS1
1,2-Dichloroethane	107-06-2	98.96	1.2351	62	64	0.50	0.16	IS1
1,4-Difluorobenzene(IS2)	540-36-3	-	-	114	88	-	-	-
1,1,1-Trichloroethane	71-55-6	133.4	1.3390	97	99, 61	0.50	0.17	IS2
Isopropyl acetate	108-21-4	102.13	0.8718	61	87,43	1.0	0.32	IS2
1-Butanol	71-36-3	74.1224	0.8098	56	41	1.0	0.48	IS2
Benzene	71-43-2	78.11	0.8765	78	77	0.50	0.16	IS2
Carbon Tetrachloride	56-23-5	153.8	1.5940	117	119	0.50	0.15	IS2

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Cyclohexane	110-82-7	84.16	0.7739	84	69,56	1.0	0.29	IS2
tert-Amyl Methyl Ether	994-05-8	102.176	0.7703	73	87,55,43	0.50	0.15	IS2
1,2-Dichloropropane	78-87-5	113	1.1560	63	62	0.50	0.16	IS2
Bromodichloromethane	75-27-4	163.8	1.980	83	85	0.50	0.15	IS2
Trichloroethene	79-01-6	131.4	1.4642	130	132	0.50	0.14	IS2
1,4-Dioxane	123-91-1	88.11	1.0337	88	58	0.50	0.16	IS2
Isooctane	540-84-1	114.23	0.6877	57	41	0.50	0.15	IS2
Methyl Methacrylate	80-62-6	100.12	0.944	100	69	1.0	0.31	IS2
n-Heptane	142-82-5	100.2	0.6837	71	57,100	0.50	0.17	IS2
cis-1,3-Dichloropropene	10061-01-5	111	1.224	75	77	0.50	0.14	IS2
4-Methyl-2-Pentanone	108-10-1	100.2	0.7965	58	85	0.50	0.16	IS2
trans-1,3-Dichloropropene	10061-02-6	111	1.217	75	77	0.50	0.16	IS2
1,1,2-Trichloroethane	79-00-5	133.4	1.4397	97	83	0.50	0.16	IS2
Chlorobenzene-d5(IS3)	3114-55-4	-	-	82	117	-	-	-
Toluene-d8(S)	2037-26-5	-	-	98	100	-	-	IS3
Toluene	108-88-3	92.14	0.8669	91	92	0.50	0.17	IS3
2-Hexanone	591-78-6	100.16	0.8113	43	58	0.50	0.16	IS3
Dibromochloromethane	124-48-1	208.3	2.451	129	127	0.50	0.16	IS3
1,2-Dibromoethane	106-93-4	187.9	2.1791	107	109	0.50	0.16	IS3
n-Butyl Acetate	123-86-4	116.16	0.8825	43	56, 73	0.50	0.16	IS3
n-Octane	111-65-9	114.23	0.6986	57	114	0.50	0.18	IS3

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Tetrachloroethene	127-18-4	165.8	1.6227	166	164	0.50	0.14	IS3
Chlorobenzene	108-90-7	112.6	1.1058	112	114	0.50	0.16	IS3
Ethylbenzene	100-41-4	106.2	0.8670	91	106	0.50	0.16	IS3
m-, p-Xylenes	179601-23-1	106.2	0.8642, 0.8611	91	106	1.0	0.30	IS3
Bromoform	75-25-2	252.8	2.899	173	175	0.50	0.15	IS3
Styrene	100-42-5	104.1	0.9060	104	78, 103	0.50	0.15	IS3
o-Xylene	95-47-6	106.2	0.8802	91	106	0.50	0.15	IS3
n-Nonane	111-84-2	128.26	0.7176	43	57, 85	0.50	0.15	IS3
1,1,2,2-Tetrachloroethane	79-34-5	167.9	1.5953	83	85	0.50	0.15	IS3
4-Bromofluorobenzene(S)	460-00-4	-	-	174	176	-	-	IS3
Cumene	98-82-8	120.2	0.8618	105	120	0.50	0.15	IS3
alpha-Pinene	80-56-8	136.24	0.8582	93	77	0.50	0.14	IS3
n-Propylbenzene	103-65-1	120.1938	0.8670	91	120,65	0.50	0.16	IS3
3-Ethyltoluene	620-14-4	120.2	0.8645	105	120	0.50	0.15	IS3
4-Ethyltoluene	622-96-8	120.2	0.8614	105	120	0.50	0.16	IS3
1,3,5-Trimethylbenzene	108-67-8	120.2	0.8652	105	120	0.50	0.16	IS3
alpha-Methylstyrene	98-83-9	118.19	0.9106	118	103,117	0.50	0.15	IS3
2-Ethyltoluene	611-14-3	120.2	0.8807	105	120	0.50	0.15	IS3
1,2,4-Trimethylbenzene	95-63-6	120.2	0.8758	105	120	0.50	0.15	IS3
n-Decane	124-18-5	142.28	0.7300	57	71,85	0.50	0.16	IS3
Benzyl Chloride	100-44-7	126.59	1.1004	91	126	0.50	0.11	IS3

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	CAS Number	Molecular Weight	Density	Primary Ion[2]	Secondary Ion(s)[2]	MRL[3] (µg/m³)	MDL[3] (µg/m³)	IS[4]
1,3-Dichlorobenzene	541-73-1	147	1.2884	146	148	0.50	0.15	IS3
1,4-Dichlorobenzene	106-46-7	147	1.2475	146	148	0.50	0.14	IS3
sec-Butylbenzene	135-98-8	134.2206	0.8601	105	134,91	0.50	0.16	IS3
p-Isopropyltoluene	99-87-6	134.2206	0.8573	119	134,91	0.50	0.15	IS3
1,2,3-Trimethylbenzene	526-73-8	120.1938	0.8944	105	120	0.50	0.15	IS3
1,2-Dichlorobenzene	95-50-1	147	1.3059	146	148	0.50	0.15	IS3
d-Limonene	5989-27-5	136.24	0.8402	68	93	0.50	0.14	IS3
1,2,Dibromo-3-Chloropropane	96-12-8	236.33	2.093	157	75, 39	0.50	0.099	IS3
n-Undecane	1120-21-4	156.31	0.7402	57	71, 85	0.50	0.15	IS3
1,2,4-Trichlorobenzene	120-82-1	181.5	1.459	180	182, 184	0.50	0.16	IS3
Naphthalene	91-20-3	128.17	1.0253	128	129	0.50	0.18	IS3
n-Dodecane	112-40-3	170.34	0.7487	57	71,85	0.50	0.13	IS3
Hexachlorobutadiene	87-68-3	260.8	1.556	225	227	0.50	0.14	IS3
Cyclohexanone	108-94-1	98.14	0.9478	55	42, 98	0.50	0.12	IS3
tert-Butylbenzene	98-06-6	134.22	0.867	119	134	0.50	0.15	IS3
n-Butylbenzene	104-51-8	134.22	0.867	91	134	0.50	0.17	IS3

(S) = Surrogate (IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3
NA = Not Available

Note 1: Additional compounds may be reported as long as the minimum requirements of this document are met. The compounds listed in this table are reported using TO-15 SCAN. The Selected Ion Monitoring (SIM) compounds are a subset of this list and are included in Table 2A.

Note 2: These are suggested primary and secondary ions. However, any ions in the analyte spectra that are sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 3: The laboratory performs three concentration level analyses (SIM, SCAN and Low Level SCAN). The method reporting limit listed is the standard SCAN limit (at or above lowest concentration in the initial calibration curve), but may change with each new initial calibration performed. Therefore, current reporting limits for the three analysis levels, MRLs in ppbv, and those from the Low Level SCAN should be reviewed in the electronic TO-15 Method Manual.

Note 4: The listing of the internal standard by which the compounds are quantitated is for TO-15 SCAN only. SIM compounds (SCAN subset) and their corresponding ions and internal standards are listed in Table 2A.

Note 5: m/e 101 is ~10% or less of m/e 85 (the base peak) and may not be present for low level results. Retention times must be carefully verified.

Table 2A – Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)					
Compound	**Primary Ion[1]**	**Secondary Ion[1]**	**MRL[2] (ug/m3)**	**MDL[2] (ug/m3)**	**IS**
Dichlorodifluoromethane	85	87	0.050	0.017	IS1
Chloromethane	52	50	0.050	0.019	IS1
Vinyl Chloride	62	64	0.025	0.0076	IS1
1,3-Butadiene	54	39	0.050	0.014	IS1
Bromomethane	94	96	0.025	0.0093	IS1
Chloroethane	64	66	0.025	0.0085	IS1
Acrolein	56	55	0.20	0.039	IS1
Acetone	58	43	2.5	0.056	IS1
Freon 11	101	103	0.050	0.015	IS1
1,1-Dichloroethene	96	98,61	0.025	0.0086	IS1
Methylene Chloride	84	49	0.10	0.013	IS1
Trichlorotrifluoroethane	151	153	0.025	0.0089	IS1
trans-1,2-Dichloroethene	96	98,61	0.025	0.0073	IS1
1,1-Dichloroethane	63	65	0.025	0.0061	IS1
Methyl tert-Butyl Ether	73	57	0.025	0.0093	IS1
cis-1,2-Dichloroethene	96	98,61	0.025	0.0092	IS1
Chloroform	83	85	0.10	0.018	IS1
1,2-Dichloroethane	62	64	0.025	0.0084	IS1
1,1,1-Trichloroethane	97	99	0.025	0.0059	IS1
Benzene	78	77	0.075	0.020	IS1
Carbon Tetrachloride	117	119	0.025	0.012	IS1
1,2-Dichloropropane	63	62,76	0.025	0.0073	IS2
Bromodichloromethane	83	85	0.025	0.0069	IS2
Trichloroethene	130	132	0.025	0.0085	IS2
1,4-Dioxane	88	58	0.10	0.0085	IS2
cis-1,3-Dichloropropene	75	77,39	0.025	0.0062	IS2
trans-1,3-Dichloropropene	75	77,39	0.025	0.0055	IS2
1,1,2-Trichloroethane	83	97,61	0.10	0.0079	IS2
Toluene	91	92	0.10	0.011	IS2
Dibromochloromethane	129	127	0.025	0.0088	IS3
1,2-Dibromoethane	107	109	0.025	0.0079	IS2
Tetrachloroethene	166	164	0.025	0.0082	IS2
Chlorobenzene	112	114	0.10	0.0092	IS3
Ethylbenzene	91	106	0.10	0.0097	IS3
m-&-p-Xylene	91	106	0.10	0.019	IS3
Styrene	104	103	0.10	0.0074	IS3
o-Xylene	91	106	0.10	0.0089	IS3
1,1,2,2-Tetrachloroethane	83	85	0.025	0.0072	IS3
1,3,5-Trimethylbenzene	105	120	0.10	0.0073	IS3
1,2,4-Trimethylbenzene	105	120	0.10	0.0083	IS3
1,3-Dichlorobenzene	146	148	0.025	0.0085	IS3
1,4-Dichlorobenzene	146	148	0.025	0.0081	IS3
1,2-Dichlorobenzene	146	148	0.025	0.0083	IS3
1,2-Dibromo-3-chloropropane	157	75	0.10	0.0095	IS3
1,2,4-Trichlorobenzene	182	184	0.025	0.013	IS3
Naphthalene	128	129	0.10	0.016	IS3
Hexachlorobutadiene	225	227	0.10	0.0092	IS3

NA = Not Available (IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3

Note 1: These are suggested primary and secondary ions. However, any ions in the analyte spectra that is sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 2: The method reporting limit listed is the standard SIM limit (lowest concentration in the initial calibration curve; must be higher than MDL), but may change with each new initial calibration performed. Therefore, current reporting limits should be reviewed. MDLs in ppbV may be reviewed in the electronic TO-15 Method Manual.

Table 3
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
Bromochloromethane (IS1)	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**
Propene	0.08288	0.2072	0.4144	1.036	5.180	25.900	51.80	103.6
Dichlorodifluoromethane (CFC 12)	0.08376	0.2094	0.4188	1.047	5.235	26.175	52.35	104.7
Chloromethane	0.08040	0.2010	0.4020	1.005	5.025	25.125	50.25	100.5
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	0.08040	0.2010	0.4020	1.005	5.025	25.125	50.25	100.5
Vinyl Chloride	0.08184	0.2046	0.4092	1.023	5.115	25.575	51.15	102.3
1,3-Butadiene	0.08456	0.2114	0.4228	1.057	5.285	26.425	52.85	105.7
Bromomethane	0.07944	0.1986	0.3972	0.993	4.965	24.825	49.65	99.3
Chloroethane	0.08072	0.2018	0.4036	1.009	5.045	25.225	50.45	100.9
Ethanol	0.41656	1.0414	2.0828	5.207	26.035	130.175	260.35	520.7
Acetonitrile	0.08368	0.2092	0.4184	1.046	5.230	26.150	52.30	104.6
Acrolein	0.08328	0.2082	0.4164	1.041	5.205	26.025	52.05	104.1
Acetone	0.42504	1.0626	2.1252	5.313	26.565	132.825	265.65	531.3
Trichlorofluoromethane	0.08392	0.2098	0.4196	1.049	5.245	26.225	52.45	104.9
Isopropyl Alcohol	0.16840	0.4210	0.8420	2.105	10.525	52.625	105.25	210.5
Acrylonitrile	0.08440	0.2110	0.4220	1.055	5.275	26.375	52.75	105.5
1,1-Dichloroethene	0.08472	0.2118	0.4236	1.059	5.295	26.475	52.95	105.9
tert-Butanol	0.16912	0.4228	0.8456	2.114	10.570	52.850	105.70	211.4
Methylene Chloride	0.08456	0.2114	0.4228	1.057	5.285	26.425	52.85	105.7
Allyl Chloride	0.08416	0.2104	0.4208	1.052	5.260	26.300	52.60	105.2
Trichlorotrifluoroethane	0.08392	0.2098	0.4196	1.049	5.245	26.225	52.45	104.9
Carbon Disulfide	0.08488	0.2122	0.4244	1.061	5.305	26.525	53.05	106.1
trans-1,2-Dichloroethene	0.08536	0.2134	0.4268	1.067	5.335	26.675	53.35	106.7
1,1-Dichloroethane	0.08160	0.2040	0.4080	1.020	5.100	25.500	51.00	102.0
Methyl tert-Butyl Ether	0.08528	0.2132	0.4264	1.066	5.330	26.650	53.30	106.6
Vinyl Acetate	0.42120	1.0530	2.1060	5.265	26.325	131.625	263.25	526.5
2-Butanone (MEK)	0.08392	0.2098	0.4196	1.049	5.245	26.225	52.45	104.9
cis-1,2-Dichloroethene	0.08512	0.2128	0.4256	1.064	5.320	26.600	53.20	106.4
Diisopropyl Ether	0.08496	0.2124	0.4248	1.062	5.310	26.550	53.10	106.2
Ethyl Acetate	0.17032	0.4258	0.8516	2.129	10.645	53.225	106.45	212.9
n-Hexane	0.08504	0.2126	0.4252	1.063	5.315	26.575	53.15	106.3
Chloroform	0.08464	0.2116	0.4232	1.058	5.290	26.450	52.90	105.8
1,2-Dichloroethane-d4 (S)	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**
Tetrahydrofuran	0.08496	0.2124	0.4248	1.062	5.310	26.550	53.10	106.2
Ethyl tert-Butyl Ether	0.08456	0.2114	0.4228	1.057	5.285	26.425	52.85	105.7
1,2-Dichloroethane	0.08416	0.2104	0.4208	1.052	5.260	26.300	52.60	105.2
1,4-Difluorobenzene(IS2)	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**
1,1,1-Trichloroethane	0.08592	0.2148	0.4296	1.074	5.370	26.850	53.70	107.4
Isopropyl acetate	0.16832	0.4208	0.8416	2.104	10.520	52.600	105.20	210.4
1-Butanol	0.16840	0.4210	0.8420	2.105	10.525	52.625	105.25	210.5

Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
Benzene	0.08416	0.2104	0.4208	1.052	5.260	26.300	52.60	105.2
Carbon Tetrachloride	0.08440	0.2110	0.4220	1.055	5.275	26.375	52.75	105.5
Cyclohexane	0.17040	0.4260	0.8520	2.130	10.650	53.250	106.50	213.0
tert-Amyl Methyl Ether	0.08432	0.2108	0.4216	1.054	5.270	26.350	52.70	105.4
1,2-Dichloropropane	0.08496	0.2124	0.4248	1.062	5.310	26.550	53.10	106.2
Bromodichloromethane	0.08528	0.2132	0.4264	1.066	5.330	26.650	53.30	106.6
Trichloroethene	0.08480	0.2120	0.4240	1.060	5.300	26.500	53.00	106.0
1,4-Dioxane	0.08496	0.2124	0.4248	1.062	5.310	26.550	53.10	106.2
Isooctane	0.08472	0.2118	0.4236	1.059	5.295	26.475	52.95	105.9
Methyl Methacrylate	0.16880	0.4220	0.8440	2.110	10.550	52.750	105.50	211.0
n-Heptane	0.08496	0.2124	0.4248	1.062	5.310	26.550	53.10	106.2
cis-1,3-Dichloropropene	0.08928	0.2232	0.4464	1.116	5.580	27.900	55.80	111.6
4-Methyl-2-Pentanone	0.08464	0.2116	0.4232	1.058	5.290	26.450	52.90	105.8
trans-1,3-Dichloropropene	0.08512	0.2128	0.4256	1.064	5.320	26.600	53.20	106.4
1,1,2-Trichloroethane	0.08488	0.2122	0.4244	1.061	5.305	26.525	53.05	106.1
Chlorobenzene-d5 (IS3)	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**
Toluene-d8 (S)	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**
Toluene	0.08424	0.2106	0.4212	1.053	5.265	26.325	52.65	105.3
2-Hexanone	0.08488	0.2122	0.4244	1.061	5.305	26.525	53.05	106.1
Dibromochloromethane	0.08496	0.2124	0.4248	1.062	5.310	26.550	53.10	106.2
1,2-Dibromoethane	0.08448	0.2112	0.4224	1.056	5.280	26.400	52.80	105.6
n-Butyl Acetate	0.08512	0.2128	0.4256	1.064	5.320	26.600	53.20	106.4
n-Octane	0.08456	0.2114	0.4228	1.057	5.285	26.425	52.85	105.7
Tetrachloroethene	0.08488	0.2122	0.4244	1.061	5.305	26.525	53.05	106.1
Chlorobenzene	0.08488	0.2122	0.4244	1.061	5.305	26.525	53.05	106.1
Ethylbenzene	0.08440	0.2110	0.4220	1.055	5.275	26.375	52.75	105.5
m- & p-Xylene	0.16984	0.4246	0.8492	2.123	10.615	53.075	106.15	212.3
Bromoform	0.08504	0.2126	0.4252	1.063	5.315	26.575	53.15	106.3
Styrene	0.08488	0.2122	0.4244	1.061	5.305	26.525	53.05	106.1
o-Xylene	0.08432	0.2108	0.4216	1.054	5.270	26.350	52.70	105.4
n-Nonane	0.08432	0.2108	0.4216	1.054	5.270	26.350	52.70	105.4
1,1,2,2-Tetrachloroethane	0.08448	0.2112	0.4224	1.056	5.280	26.400	52.80	105.6
4-Bromofluorobenzene (S)	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**	**12.5**
Cumene	0.08400	0.2100	0.4200	1.050	5.250	26.250	52.50	105.0
alpha-Pinene	0.08352	0.2088	0.4176	1.044	5.220	26.100	52.20	104.4
n-Propylbenzene	0.08504	0.2126	0.4252	1.063	5.315	26.575	53.15	106.3
3-Ethyltoluene	0.08400	0.2100	0.4200	1.050	5.250	26.250	52.50	105.0
4-Ethyltoluene	0.08392	0.2098	0.4196	1.049	5.245	26.225	52.45	104.9
1,3,5-Trimethylbenzene	0.08392	0.2098	0.4196	1.049	5.245	26.225	52.45	104.9
alpha-Methylstyrene	0.08400	0.2100	0.4200	1.050	5.250	26.250	52.50	105.0
2-Ethyltoluene	0.08496	0.2124	0.4248	1.062	5.310	26.550	53.10	106.2
1,2,4-Trimethylbenzene	0.08416	0.2104	0.4208	1.052	5.260	26.300	52.60	105.2

Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
n-Decane	0.08424	0.2106	0.4212	1.053	5.265	26.325	52.65	105.3
Benzyl Chloride	0.08488	0.2122	0.4244	1.061	5.305	26.525	53.05	106.1
1,3-Dichlorobenzene	0.08464	0.2116	0.4232	1.058	5.290	26.450	52.90	105.8
1,4-Dichlorobenzene	0.08464	0.2116	0.4232	1.058	5.290	26.450	52.90	105.8
sec-Butylbenzene	0.08432	0.2108	0.4216	1.054	5.270	26.350	52.70	105.4
p-Isopropyltoluene	0.08216	0.2054	0.4108	1.027	5.135	25.675	51.35	102.7
1,2,3-Trimethylbenzene	0.08216	0.2054	0.4108	1.027	5.135	25.675	51.35	102.7
1,2-Dichlorobenzene	0.08464	0.2116	0.4232	1.058	5.290	26.450	52.90	105.8
d-Limonene	0.08040	0.2010	0.4020	1.005	5.025	25.125	50.25	100.5
1,2-Dibromo-3-Chloropropane	0.08424	0.2106	0.4212	1.053	5.265	26.325	52.65	105.3
n-Undecane	0.08432	0.2108	0.4216	1.054	5.270	26.350	52.70	105.4
1,2,4-Trichlorobenzene	0.08344	0.2086	0.4172	1.043	5.215	26.075	52.15	104.3
Naphthalene	0.08664	0.2166	0.4332	1.083	5.415	27.075	54.15	108.3
n-Dodecane	0.08360	0.2090	0.4180	1.045	5.225	26.125	52.25	104.5
Hexachlorobutadiene	0.08472	0.2118	0.4236	1.059	5.295	26.475	52.95	105.9
Methacrylonitrile	0.08520	0.2130	0.4260	1.065	5.325	26.625	53.25	106.5
Cyclohexanone	0.08448	0.2112	0.4224	1.056	5.280	26.400	52.80	105.6
tert-Butylbenzene	0.08408	0.2102	0.4204	1.051	5.255	26.275	52.55	105.1
n-Butylbenzene	0.08448	0.2112	0.4224	1.056	5.280	26.400	52.80	105.6

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 3A - Standard Concentrations (SIM) (Primary Sources)[1]

Compound Name	10pg	20pg	50pg	100pg	500pg	1000pg	5000pg	10,000pg	25,000pg	50,000pg
Freon-12	10.47	20.94	52.35	104.7	523.5	1047	5235	10470	26175	52350
Chloromethane	10.05	20.10	50.25	100.5	502.5	1005	5025	10050	25125	50250
Vinyl Chloride	10.23	20.46	51.15	102.3	511.5	1023	5115	10230	25575	51150
1,3-Butadiene	10.57	21.14	52.85	105.7	528.5	1057	5285	10570	26425	52850
Bromomethane	9.93	19.86	49.65	99.3	496.5	993	4965	9930	24825	49650
Chloroethane	10.09	20.18	50.45	100.9	504.5	1009	5045	10090	25225	50450
Acrolein	10.41	20.82	52.05	104.1	520.5	1041	5205	10410	26025	52050
Acetone	53.13	106.26	265.65	531.3	2656.5	5313	26565	53130	132825	265650
Freon-11	10.49	20.98	52.45	104.9	524.5	1049	5245	10490	26225	52450
1,1-Dichloroethene	10.59	21.18	52.95	105.9	529.5	1059	5295	10590	26475	52950
Methylene Chloride	10.57	21.14	52.85	105.7	528.5	1057	5285	10570	26425	52850
Freon-113	10.49	20.98	52.45	104.9	524.5	1049	5245	10490	26225	52450
trans-1,2-Dichloroethene	10.67	21.34	53.35	106.7	533.5	1067	5335	10670	26675	53350
1,1-Dichloroethane	10.20	20.40	51.00	102.0	510.0	1020	5100	10200	25500	51000
Methyl tert-Butyl Ether	10.66	21.32	53.30	106.6	533.0	1066	5330	10660	26650	53300
cis-1,2-Dichloroethene	10.64	21.28	53.20	106.4	532.0	1064	5320	10640	26600	53200
Chloroform	10.58	21.16	52.90	105.8	529.0	1058	5290	10580	26450	52900
1,2-Dichloroethane	10.52	21.04	52.60	105.2	526.0	1052	5260	10520	26300	52600
1,1,1-Trichloroethane	10.74	21.48	53.70	107.4	537.0	1074	5370	10740	26850	53700
Benzene	10.52	21.04	52.60	105.2	526.0	1052	5260	10520	26300	52600
Carbon Tetrachloride	10.55	21.10	52.75	105.5	527.5	1055	5275	10550	26375	52750
1,2-Dichloropropane	10.62	21.24	53.10	106.2	531.0	1062	5310	10620	26550	53100
Bromodichloromethane	10.66	21.32	53.30	106.6	533.0	1066	5330	10660	26650	53300
Trichloroethene	10.60	21.20	53.00	106.0	530.0	1060	5300	10600	26500	53000
1,4-Dioxane	10.62	21.24	53.10	106.2	531.0	1062	5310	10620	26550	53100
cis-1,3-Dichloropropene	11.16	22.32	55.80	111.6	558.0	1116	5580	11160	27900	55800
trans-1,3-Dichloropropene	10.64	21.28	53.20	106.4	532.0	1064	5320	10640	26600	53200
1,1,2-Trichloroethane	10.61	21.22	53.05	106.1	530.5	1061	5305	10610	26525	53050
Toluene	10.53	21.06	52.65	105.3	526.5	1053	5265	10530	26325	52650
Dibromochloromethane	10.62	21.24	53.10	106.2	531.0	1062	5310	10620	26550	53100
1,2-Dibromoethane	10.56	21.12	52.80	105.6	528.0	1056	5280	10560	26400	52800
Tetrachloroethene	10.61	21.22	53.05	106.1	530.5	1061	5305	10610	26525	53050
Chlorobenzene	10.61	21.22	53.05	106.1	530.5	1061	5305	10610	26525	53050
Ethylbenzene	10.55	21.10	52.75	105.5	527.5	1055	5275	10550	26375	52750
m,p-Xylenes	21.23	42.46	106.15	212.3	1061.5	2123	10615	21230	53075	106150
Styrene	10.61	21.22	53.05	106.1	530.5	1061	5305	10610	26525	53050
o-Xylene	10.54	21.08	52.70	105.4	527.0	1054	5270	10540	26350	52700
1,1,2,2-Tetrachloroethane	10.56	21.12	52.80	105.6	528.0	1056	5280	10560	26400	52800
1,3,5-Trimethylbenzene	10.49	20.98	52.45	104.9	524.5	1049	5245	10490	26225	52450
1,2,4-Trimethylbenzene	10.52	21.04	52.60	105.2	526.0	1052	5260	10520	26300	52600
1,3-Dichlorobenzene	10.58	21.16	52.90	105.8	529.0	1058	5290	10580	26450	52900
1,4-Dichlorobenzene	10.58	21.16	52.90	105.8	529.0	1058	5290	10580	26450	52900
1,2-Dichlorobenzene	10.58	21.16	52.90	105.8	529.0	1058	5290	10580	26450	52900
1,2-Dibromo-3-chloropropane	10.53	21.06	52.65	105.3	526.5	1053	5265	10530	26325	52650
1,2,4-Trichlorobenzene	10.43	20.86	52.15	104.3	521.5	1043	5215	10430	26075	52150
Naphthalene	10.83	21.66	54.15	108.3	541.5	1083	5415	10830	27075	54150
Hexachloro-1,3-butadiene	10.59	21.18	52.95	105.9	529.5	1059	5295	10590	26475	52950

Note 1: The concentrations detailed in Table 3A may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 4 - Standard Concentrations (SCAN) (Secondary Sources)[1]

Compound Name	25ng	Compound Name	25ng	Compound Name	25ng
Bromochloromethane (IS1)	**12.5**	1,1,1-Trichloroethane	26.475	alpha-Pinene	26.575
Propene	26.275	Isopropyl acetate	53.050	n-Propylbenzene	26.725
Dichlorodifluoromethane (CFC 12)	26.250	1-Butanol	53.075	3-Ethyltoluene	26.550
Chloromethane	26.225	Benzene	26.525	4-Ethyltoluene	26.525
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	26.375	Carbon Tetrachloride	26.600	1,3,5-Trimethylbenzene	26.525
Vinyl Chloride	26.250	Cyclohexane	53.125	alpha-Methylstyrene	26.550
1,3-Butadiene	26.250	tert-Amyl Methyl Ether	26.525	2-Ethyltoluene	26.550
Bromomethane	26.250	1,2-Dichloropropane	26.525	1,2,4-Trimethylbenzene	26.525
Chloroethane	26.225	Bromodichloromethane	26.700	n-Decane	26.525
Ethanol	132.65	Trichloroethene	26.550	Benzyl Chloride	26.550
Acetonitrile	26.650	1,4-Dioxane	26.600	1,3-Dichlorobenzene	26.475
Acrolein	26.525	Isooctane	26.525	1,4-Dichlorobenzene	26.650
Acetone	133.05	Methyl Methacrylate	53.000	sec-Butylbenzene	26.550
Trichlorofluoromethane	26.275	n-Heptane	26.600	p-Isopropyltoluene	26.550
Isopropyl Alcohol	53.025	cis-1,3-Dichloropropene	26.275	1,2,3-Trimethylbenzene	26.550
Acrylonitrile	26.575	4-Methyl-2-Pentanone	26.575	1,2-Dichlorobenzene	26.550
1,1-Dichloroethene	26.575	trans-1,3-Dichloropropene	26.675	d-Limonene	26.550
tert-Butanol	53.275	1,1,2-Trichloroethane	26.525	1,2-Dibromo-3-Chloropropane	26.475
Methylene Chloride	26.550	**Chlorobenzene-d5 (IS3)**	**12.5**	n-Undecane	26.600
Allyl Chloride	26.500	**Toluene-d8 (S)**	**12.5**	1,2,4-Trichlorobenzene	26.500
Trichlorotrifluoroethane	26.450	Toluene	26.450	Naphthalene	26.700
Carbon Disulfide	26.675	2-Hexanone	26.575	n-Dodecane	26.550
trans-1,2-Dichloroethene	26.675	Dibromochloromethane	26.600	Hexachlorobutadiene	26.575
1,1-Dichloroethane	26.550	1,2-Dibromoethane	26.450	Methacrylonitrile	26.550
Methyl tert-Butyl Ether	26.600	Butyl Acetate	26.950	Cyclohexanone	26.575
Vinyl Acetate	132.55	n-Octane	26.500	tert-Butylbenzene	26.500
2-Butanone (MEK)	26.550	Tetrachloroethene	26.575	n-Butylbenzene	26.500
cis-1,2-Dichloroethene	26.475	Chlorobenzene	26.500		
Diisopropyl Ether	26.575	Ethylbenzene	26.450		
Ethyl Acetate	53.275	m- & p-Xylene	53.025		
n-Hexane	26.600	Bromoform	26.550		
Chloroform	26.475	Styrene	26.475		
1,2-Dichloroethane-d4 (S)	**12.5**	o-Xylene	26.450		
Tetrahydrofuran	26.575	n-Nonane	26.475		
Ethyl tert-Butyl Ether	26.525	1,1,2,2-Tetrachloroethane	26.500		
1,2-Dichloroethane	26.500	**4-Bromofluorobenzene (S)**	**12.5**		
1,4-Difluorobenzene(IS2)	**12.5**	Cumene	26.525		

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 4A – ICV/LCS Standard Concentrations (SIM) (Secondary Sources)[1]

Compound Name	500pg
Freon-12	525.0
Chloromethane	524.5
Vinyl Chloride	525.0
1,3-Butadiene	525.0
Bromomethane	525.0
Chloroethane	524.5
Acrolein	530.5
Acetone	2661.0
Freon-11	525.5
1,1-Dichloroethene	531.5
Methylene Chloride	531.0
Freon-113	529.0
trans-1,2-Dichloroethene	533.5
1,1-Dichloroethane	531.0
Methyl tert-Butyl Ether	532.0
cis-1,2-Dichloroethene	529.5
Chloroform	529.5
1,2-Dichloroethane	530.0
1,1,1-Trichloroethane	529.5
Benzene	530.5
Carbon Tetrachloride	532.0
1,2-Dichloropropane	530.5
Bromodichloromethane	534.0
Trichloroethene	531.0
1,4-Dioxane*	532.0
cis-1,3-Dichloropropene	525.5
trans-1,3-Dichloropropene	533.5
1,1,2-Trichloroethane	530.5
Toluene	529.0
Dibromochloromethane	532.0
1,2-Dibromoethane	529.0
Tetrachloroethene	531.5
Chlorobenzene	530.0
Ethylbenzene	529.0
m,p-Xylenes	1060.5
Styrene	529.5
o-Xylene	529.0
1,1,2,2-Tetrachloroethane	530.0
1,3,5-Trimethylbenzene	530.5
1,2,4-Trimethylbenzene	530.5
1,3-Dichlorobenzene	529.5
1,4-Dichlorobenzene	533.0
1,2-Dichlorobenzene	531.0
1,2-Dibromo-3-chloropropane	529.5
1,2,4-Trichlorobenzene	530.0
Naphthalene	534.0
Hexachloro-1,3-butadiene	531.5

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Attachment 1

Training Plan

Training Plan for Analysis of VOCs by GC/MS

Trainee _____ Trainer _____ Instrument _____ Training Completion Date _____

1. Read SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____

2. Read Methods TO-14A & TO-15A *Training Duration* _____ Trainer ____ Trainee ____ Date _____

3. Demonstrated understanding of the scientific basis of the analysis Trainer ____ Trainee ____ Date _____
   Whole air sample preconcentration techniques
   Gas chromatography *Training Duration* _____
   Mass spectrometry

4. Demonstrated familiarity with related SOPs Trainer ____ Trainee ____ Date _____
   SOP for Batches and Sequences; Rev. ___
   SOP for Making Entries onto Analytical Records; Rev. ___ *Training Duration* _____
   SOP for Manual Integration Policy; Rev. ____
   SOP for Significant Figures; Rev. ____
   SOP for Nonconformance and Corrective Action; Rev. ____
   SOP for Performing MDL Studies and Establishing Limits of Detection and Quantitation; Rev. ____
   SOP for Cleaning and Certification of Summa Canisters; Rev. ____

5. Observe performance of SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant introduction (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

6. Perform SOP with supervision *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant use (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

7. Independent performance of the SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant proficiency (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)
   ___initial demonstration of competency (4 Laboratory Control Samples)

8. Instrument operation and maintenance Trainer ____ Trainee ____ Date _____
   ___autosampler *Training Duration* _____
   ___GC and capillary column installation *Training Duration* _____
   ___mass spectrometer *Training Duration* _____
   ___data system *Training Duration* _____

Attachment 2

Initial Calibration Checklist

Initial Calibration Review Checklist - EPA Compendium Method TO-15

ICAL Date:_____ ICAL ID:_____ LIMS ICAL ID:_____

Instrument: ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21 ☐ MS22

Mode: ☐ SIM ☐ Scan Scan Low Level (0.1ng): ☐ Yes ☐ No

Analyst		Reviewer
☐	1. Is the required documentation in the ICAL file?	☐
	☐ BFB Tune analysis Report	☐
	☐ Calibration Status Report (aka Calibration History)	☐
	☐ Response Factor Report/Percent RSD	☐
	☐ Quant Report for each calibration std (including manual integration documentation)	☐
	☐ ICV Quantitation Report	☐
	☐ TO-15 Standard Calculation Spreadsheet	☐
☐	2. Was the ICAL performed continuously (not interrupted for maintenance or sample analysis)?	☐
☐	3. Have all the calibration standards been analyzed within 24 hours of each other?	☐
☐	4. Does the BFB tune check standard analysis at the start meet the tune criteria?	☐
☐	5. Are all the analytes in the blank analysis <MRL?	☐
☐	6. Does each analyte's ICAL include a minimum of 5 concentrations at 5 consecutive levels?	☐
☐	7. Were the standards analyzed from low concentration to high concentration?	☐
☐	8. For each analyte, are there no levels skipped?	☐
☐	9. For each analyte, is there only one value used for each calibration level?	☐
☐	10. For each analyte, is the lowest standard's concentration at or below the analyte's MRL?	☐
☐	11. For each analyte, is the corresponding signal to noise ratio at least 3:1 at the lowest point on the curve?	☐
☐	12. For each analyte, are the corresponding upper levels free from saturation?	☐
☐	13. If a calibration level is dropped, are all the responses for each target analyte dropped and is the information noted in the ICAL explaining the reason?	☐
☐	14. Is the average RSD ≤30% for all analytes, with no more than two exceptions ≤40%?	☐
☐	15. DoD/Navy: Is the average RSD ≤30% for all analytes?	☐
☐	16. Is the response Y at each calibration level within 40% of the mean area response over the initial calibration range for each internal standard?	☐
☐	17. Percent recovery for each analyte in the ICV 70%-130% (AZ: 50-150% for VA)?	☐
☐	18. Was the RRT for each target compound at each calibration level within 0.06RRT units of the mean RRT for the compound?	☐
☐	19. Is the retention time shift for each of the internal standards at each calibration level within 20s of the mean retention time over the initial calibration range for each standard?	☐
☐	20. If there are any manual integrations, are they performed correctly according to the corresponding SOP? If so, initial and date the appropriate pages.	☐
☐	21. Is the ICAL good at 0.5ng (or 0.1ng)–100ng (Scan) or 10-20000pg (SIM) for all compounds? ☐ Yes ☐ No Note exceptions and corresponding MRLs below – *Specify applicable range*...	☐
☐	22. Are ALL of the peak selections for each analyte correct according to retention time (all RTs must be checked by both the initial and peer reviewer)?	☐

COMMENTS:

Analyst: _____ Secondary Reviewer: _____

Attachment 3

Daily QC and Sample Review Checklists

EPA Compendium Method TO-15 - Daily QC Review Checklist
(Note exceptions in Comments and include Analysis Observations/Case Narrative Summary Form as appropriate)

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____

Instrument: ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21 ☐ MS22

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst		Reviewer
☐	1. Is the required documentation present? ☐ CORRECT BFB Tune analysis Report ☐ CCV analysis Quantitation Report & %D Report ☐ LCS analysis Quantitation Report ☐ MB analysis Quantitation Report	☐
☐	2. BFB **tune** check standard analysis meet the tune criteria for the method indicated above?	☐
☐	3. Analyses within the tune's **24-hr window or** ☐ **Client's 12hr window requirement**?	☐
☐	4. Does the **CCV** have a difference ≤30% for all analytes? [Note <u>all</u> outliers biased high and/or low]	☐
☐	5. **DoD**: Does the **Closing CCV** have a difference ≤30% for all analytes? [Note <u>all</u> outliers biased high and/or low]	☐
☐	6. All **IS** retention times within 20 seconds of the CCV RT or the RT from the midpoint (ICAL)?	☐
☐	7. All **IS** responses within ±40% of CCV or the midpoint in the ICAL?	☐
☐	8. All **surrogate** recoveries (in CCVs, MB, LCSs, etc.) within acceptance limits (70%-130%)	☐
☐	9. All analytes in the **MB** <MRL? (DoD <1/2MRL, except Acetone, MeCl2, EtOH, Carbon Disulfide)?	☐
☐	10. **LCS** %R within lab control limits for all analytes except AZ samples (70%-130%, VA 50%-150%)?	☐
☐	11. All analytes in the **Lab Duplicate / DLCS** within ±25% or the client specified limits?	☐
☐	12. **DoD/Navy**: **DLCS** analyzed?	☐

<u>Air-Phase Petroleum Hydrocarbons</u>

☐	1. Does the **CCV** meet the following criteria? • Percent difference ≤30%. • One compound or range can be >30%, but less than 50%. • No single analyte or range may be >50%. [Note outliers biased high and/or low in comments below]	☐
☐	2. Does lab **duplicate** meet an RPD of ≤30% for results >5x MRL? Repeat analysis if:	☐

RPD >30 (where both analyses are >5x RL	1st analysis detect @ >5x MRL, Dup=ND
1st analysis ≤5x RL; Dup=ND (RPD not calculable)	

☐	3. Are the analytes in the **LCS** within 70%-130% recovery?	☐

COMMENTS:

Analyst/LIMS Run Approval: _____ Secondary/LIMS Supervisor Approval: _____

EPA Compendium Method TO-15 - Sample Review Checklist

(Note exceptions in Comments and include Analysis Observations/Case Narrative Summary Form as appropriate)

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____ **Project #:** _____

Instrument: ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21 ☐ MS22

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst		Reviewer
☐	1. All analyte hits in the samples within the **calibration range** and/or noted?	☐
☐	2. All **peak integrations** acceptable?	☐
☐	3. All **manual integrations** flagged and documented?	☐
☐	4. Have **Q values** been verified for each peak?	☐
☐	5. All **calculations** correct?	☐
☐	6. Has the analyst initialed and dated each **quantitation report**?	☐
☐	7. For **TICs** are the relative intensity and other requirements met (associated MB reported)?	☐
☐	8. **Auto report** correct?	☐
☐	9. **MRL** = _____ ☐ ng ☐ pg (ethanol, acetone, vinyl acetate = 5.0ng)	☐
☐	10. Pressurized with **Helium**? Is the worksheet completed for all samples?	☐
☐	11. Report to **MDL**? ☐ Yes ☐ No	☐
☐	12. **Global Minimum Detection Limit** = _____ ☐ ng ☐ pg	☐
☐	13. **DOD:** Are **manual integrations** notated in the **case narrative?**	☐

Air-Phase Petroleum Hydrocarbons

☐	1. Are all manual **integrations** flagged and documented (except for HC ranges)?	☐
☐	2. Are the associated ICAL responses correct?	☐
☐	3. Are the sample responses entered into the template correctly?	☐
☐	4. Are the TO-15 target compounds entered into the template correctly?	☐
☐	5. Does the lab **duplicate** meet RPD ≤30% for results >5x the MRL? Otherwise, repeat analyses if:	☐

RPD >30 (where both analyses are >5x RL	1st analysis detect @ >5x MRL, Dup=ND
1st analysis ≤5x RL; Dup=ND (RPD not calculable)	

COMMENTS:

☐	1. **CASE NARRATIVE COMPLETED?**	☐

Analyst/LIMS Run Approval: _____ Secondary/LIMS Supervisor Approval: _____

Attachment 4

State and Project Specific Requirements

Minnesota Requirements	
Item	**Criteria**
Holding Time (HT)	14 days
Tedlar bags	Not allowed for sampling or sample dilution
Canisters and flow controllers	Individually certified Individually leak checked before shipment
	Samples with concentrations outside of the calibration curve will have a zero canister analysis performed to check for carryover. If carryover is detected, system bake out shall be performed and documented. Additionally, in instances where the laboratory has evidence on file that a particular compound when present at a high concentration does not exhibit carry-over, the samples will not be reanalyzed. When samples are analyzed that have a higher concentration than the evidence on file, the above requirements must be followed. Also, samples that have hits below the MRL will not be reanalyzed when analyzed after a sample with concentrations over the calibration range.
Method Reporting Verification Check	Analyze a Method Reporting Verification at the beginning of the sequence prior to analyzing samples. Acceptance criteria ±40%.
Duplicates	10 percent laboratory duplicates
Record retention	MN/NELAP 5 years MPCA (Minnesota Pollution Control Agency) compliant samples 10 years
Tier level	TIII

Arizona Requirements	
Item	**Criteria**
LCS	70-130% (vinyl acetate 50-150%)

Department of Toxic Substances Control (DTSC) Requirements	
Item	**Criteria**
Holding Time (HT)	72 hour hold time for canisters

EPA Region 9 Requirements	
Item	**Criteria**
Holding Time (HT)	14 days

Attachment 5

Tekmar AutoCan Trap Packing Instructions

Tekmar AutoCan Trap Packing Instructions

The internal sample trap on the AutoCan is a 1/8" x 12" thin-walled stainless steel tube, usually coated with fused silica (Silcosteel). It is packed with a combination of graphitized carbon black and carbon molecular sieve adsorbents, with the weakest adsorbent at the top (inlet) and the strongest at the bottom (outlet). Each bed is separated by a small plug of untreated glass wool. Untreated is used because DCMS-treated wool will release siloxanes when heated to the temperatures used for TO-15 analysis.

The adsorbents listed below are further refined at the lab by sifting in an 80-mesh sieve. This removes the smaller particles and leaves a very uniform product of about 60-mesh size. Getting rid of the "fines" helps ensure good flow through the trap during sampling and reduces the pressure drop across the trap. A tightly-packed trap can lead to problems such as poor reproducibility, slowed flow rates, and channeling (small spaces in the beds that let analytes pass through).

Adsorbent	Mesh	Supplier	Catalog #	Packing Amount (mg)
Carbosieve SIII	60/80	Supelco	10184	40
Carbosieve G	60/80	Supelco	10198	30
Carbopack Z	60/80	Supelco	20273	30
Tenax TA	20/30 or 45/60	Supelco	10257	rest of trap

Old traps can be reused if unpacked carefully and cleaned and baked out properly. Use a glass wool puller to remove the wool plugs, and gently tap the sorbent out onto a piece of paper. If necessary, use the other end of the puller to loosen the sorbent bed, being careful not to scratch the inside of the trap. Discard the old sorbent. Rinse the empty trap with methanol, then bake in a GC oven for 30 minutes at 150°C.

The total length of the adsorbent bed is 12 to 13cm. You want to leave 2 to 3cm of space above the top of the last glass wool layer to ensure that all of the material is within the heated zone of the AutoCan trap heater.

With clean hands (no lotion!) place a small amount of glass wool, about 10-15mg, into the top of the trap and work it in with a piece of wire or tubing. Then use the trap packing tool (the larger steel rod that just barely fits inside the trap) to hold the plug in the trap while you pull away any loose strands of wool. Then use the long steel tube to push the plug down about 15cm. The idea is to keep the plug very compact, so it is a good idea to use the trap packing tool to push up from the bottom while pushing the wool in from the top, meeting 15cm down. The plug should not move too easily when pushed.

Weigh out the first sorbent (Carbosieve SIII) on weighing paper using the analytical balance. Using the glass funnel and a short piece of silicone tubing, pour the sorbent into the top of the trap. Tap on counter to get it all out of the funnel, then remove the funnel and tap some more to settle the sorbent into a compact bed. It is very important that there are no air spaces in the bed. However, it is also very important not to compress the sorbents too much, so be very careful when placing the glass wool plugs.

Place a glass wool plug on top of the first bed, starting as described above for the first plug. Push it gently onto the top of the sorbent with very little pressure.

Proceed with the other three packings in the table above (Carbosieve G, Carbopack Z, and Tenax TA).

After placing the last glass wool plug on top, turn the trap over and gently tap it on a piece of white paper to see if any sorbent comes out. If it does, you need to add more glass wool.

Now the trap needs to be conditioned in the trap heater. The sorbent manufacturers recommend that they be conditioned at succeedingly higher temperatures, with the final temperature being about 20-30˚C higher than the desorb temperature. The reason is that the sieves hold a lot of air and moisture and it is better to drive these off at lower temperatures to avoid damage to the material, such as cracking and oxidation which creates active sites. The temperatures and times are:

80˚C for 30 minutes, 50 to 100ml/min nitrogen or helium flow
200˚C for 30 minutes
265˚C for at least 3 hours

These temperatures are set using the variable power controller and thermocouple meter. Repeat for the other temperatures (low to high). Make sure the gas toggle valve in back is open, and measure flow at the top of the trap.

ATTACHMENT D

FIELD SAMPLING STANDARD OPERATING PROCEDURES

UTILITY CLEARANCE

1.0 PURPOSE

This standard operating procedure (SOP) describes the process for determining the presence of subsurface utilities and other cultural features (e.g., vault or tank) at locations where planned site activities involve the physical disturbance of subsurface materials. The definition of subsurface disturbance varies by base. Each base may have specific required procedures. These procedures are made available to the contractor through the Naval Technical Representative (NTR), or other government point of contact. The SOP applies to the following activities: soil gas surveying, excavating, trenching, drilling of borings and installation of monitoring and extraction wells, use of soil recovery or slide-hammer hand augers, and all other intrusive sampling activities. The primary purpose of the SOP is to minimize the potential for damaging underground utilities and other subsurface features, which could result in physical injury, disruption of utility service, or disturbance of other subsurface cultural features.

2.0 PROCEDURES

The following steps shall be followed at all sites where subsurface exploration will include excavations, drilling, or any other subsurface investigative method that could damage utilities at a site. In addition to the steps outlined below, personnel must always exercise caution while conducting any subsurface exploratory work.

2.1 PREPARE PRELIMINARY SITE PLAN

A preliminary, scaled site plan depicting the proposed exploratory locations shall be prepared as part of the work plan. This plan should include as many of the cultural and natural features as practical.

2.2 REVIEW BACKGROUND INFORMATION

A search of existing plan files to review the as-built plans is necessary to identify the known location of utilities at the site. Copies of as-built plans shall be copied and maintained for project use. If necessary, the locations of utilities identified shall be plotted onto a preliminary, scaled site plan. Personnel reviewing these files shall inform the Project Manager (PM) if utilities lie within close proximity to a proposed exploration or excavation location. The PM will determine if it is necessary to relocate proposed sampling or excavation locations.

For removal or remedial actions, the utility location information gathered during investigation (e.g., remedial investigation or remedial site evaluation) work shall be included in the project design documents. In this manner, information regarding utility locations collected during implementation of a Task Order can be shared with the Remedial Action Contract (RAC) Contractor during implementation of a particular Delivery Order (DO).

It may be necessary to conduct interviews with onsite and facility personnel familiar with the site in order to obtain information regarding the known and suspected locations of underground utilities. The local 1-800-“Before-U-Dig” service must be contacted a minimum of two business days prior to intrusive work. Other appropriate utility or locating companies should be contacted. The dimensions, orientation, and depth of utilities other than those identified on the as-built plans should be penciled in at their approximate locations on the preliminary plans. The

type of utility, the personnel who provided the information, and the date the information was provided should be entered into the field log.

2.3 SITE VISIT - LOCATE UTILITIES - TONING

Prior to the initiation of field activities, a qualified staff member shall visit the site and note existing structures and evidence of associated utilities, such as fire hydrants, irrigation systems, manhole and vault box covers, standpipes, telephone switch boxes, free-standing light poles, gas or electric meters, pavement cuts, and linear depressions. All areas where subsurface exploration is proposed shall be accurately located or surveyed and clearly marked with stakes, pins, flags, paint, or other suitable devices.

Local private utility contractors, familiar with individual base operations and procedures should be subcontracted to identify utilities not located by the "Before U Dig" service. The private locator may be utilized earlier in the project to conduct map research if they are familiar with the base operations. The locator should utilize appropriate sensing equipment to attempt to locate any utilities that may not have appeared on the as-built plans. This may involve the use of surface geophysical methods (SOP I-B-2, *Geophysical Testing Procedures*). At a minimum, a utility locator, metal detector, and/or magnetometer should be utilized; however, it is important to consider the possibility that non-metallic utilities or tanks may be present at the site. If non-metallic cultural features are likely to be present at the site, other appropriate surface geophysical methods, such as Ground Penetrating Radar, should be used. Proposed exploration areas shall be cleared of all utilities in the immediate area where subsurface exploration is proposed. All anomalous areas should be clearly toned.

Any anomalous areas detected and toned that are in close proximity to the exploration or excavation areas shall be reported to the Field Manager. The Field Manager shall determine the safe distance to maintain from the known or suspected utility. It may be necessary to relocate proposed exploration or excavation areas. If this is required, the field manager or a similarly qualified individual shall relocate them and clearly mark them using the methods described above. The markings at the prior location shall be completely removed. In some instances, such as in areas extremely congested with subsurface utilities, it is strongly recommended to dig by hand to determine the location of the utilities.

2.4 PREPARE SITE PLAN

Prior to the initiation of some field activities, notably remedial action projects, a final site plan shall be drafted which indicates the location of subsurface exploration areas and all known or suspected utilities present at the site. Copies of this site plan shall be provided to the Field Manager, the PM and the subcontractor who is to conduct the subsurface exploration/excavation work. The site plan should be reviewed with the Navy Remedial Project Manager (RPM) to verify its accuracy prior to initiating subsurface sampling activities.

3.0 DOCUMENTATION

An approved field logbook detailing the pertinent activities conducted during the utility locating procedure shall be kept. The logbook will describe any changes and modifications made to the original exploration plan. Details of the appropriate procedures for maintaining a logbook are documented in SOP III-D, *Logbooks*.

4.0 REFERENCES

SOP I-B-2, *Geophysical Testing Procedures*

SOP III-D, *Logbooks*

5.0 ATTACHMENTS

None.

GENERAL FIELD OPERATION

1.0 PURPOSE

This standard operating procedure (SOP) defines the general field organization and the field structure of sample collection, sample identification, record keeping, field measurements, and data collection. These SOPs are used to ensure the activities used to document sampling and field operations provide standardized background information and identities.

2.0 PROCEDURES

2.1 MOBILIZATION/DEMOBILIZATION

The SM or designee ensures that all purchase requests have been reviewed and approved by the PM. Then, the SM and PM assemble the project team in order to review the scope of work, disseminate the project plans, and complete the field equipment checklist (provided as Attachment I-A-9-1). After review by the project team, if additional items are required, additional purchase requests are prepared and approved by the PM.

The SM and project team upon arrival at the site inspects all equipment. Packing slips, bills of lading, or other documentation received with the shipment are initialed and returned to the purchasing department and a copy placed into the field file. Quantities, types, and makes of items received are checked against the original purchase requests to validate the shipment. Prior to validation of the shipping receipt, equipment is inspected to ensure all components are present and that the equipment calibrates and is fully functional. Any equipment received that is not fully functional is returned immediately and the vendor contacted to arrange a replacement.

The SM provides copies of the appropriate SOPs to the project team prior to the start of field activities. The most current versions of the SOPs are brought to the field. Any revisions to the SOPs must be approved by the PM and recorded in the field logbook.

It is imperative that rental equipment be cleaned (decontaminated), packaged, and returned immediately following the completion of a task. If any problems occurred on site with any equipment, the problems should be noted in detail in the field logbook and the SM notified. The SM will forward this information to the purchasing department and the vendor.

2.2 SHIPPING

If it is possible and /or practical, equipment and supplies should be shipped directly to the field site. If sensitive field equipment is to be shipped to the site, care shall be taken to ensure the equipment is not damaged en route. All original packaging material should be retained for return shipment of the equipment. Additional packing material (e.g., bubble wrap, bubble bags) may be required to provide additional protection for the shipped items. Equipment should always be shipped in its original carrying case. Each piece being shipped must have an address label on the shipping container separate from the shipping air bill.

2.3 CHAIN OF COMMAND

Chain of command protocols are implemented by the PM. These protocols should be strictly followed while performing field tasks. All decisions concerning priorities, project team assignments, sampling procedures, equipment management, and task approach are made by the

PM, the SM, or an approved appointee. The SM or an approved designee will conduct a daily meeting prior to the start of field activities to discuss individual responsibilities. The meeting will also address potential contaminants that may be encountered, safety items (such as use of heavy equipment or protection against noise), special sampling requirements, and site control(s) to be employed to prevent injuries or exposure.

2.4 SAMPLING ORGANIZATION

The SM ensures the sampling design, outlined in project plans, is followed during all phases of the sampling activities at the site. For each sampling activity, field personnel record the information required by the applicable SOPs in their logbooks and on the exhibits provided in the SOPs.

2.5 REVIEW

The PM, SM, and, on occasion, the QAO or an approved designee checks field logbooks, daily logs, and all other documents that result from field operations for completeness and accuracy. Any discrepancies on these documents are noted and returned to the originator for correction. The reviewer acknowledges that review comments have been incorporated into the document by signing and dating the applicable reviewed documents.

3.0 DOCUMENTATION

Project activities shall be recorded in the field logbooks. The logbooks shall be kept current for the daily activities including documentation of all samples collected and the information relevant to the sample collection. All project required field forms shall be completed within a timely manner upon completion of the field task. All required field forms and specific logbook notations should be detailed in the field sampling plan.

4.0 REFERENCES

None.

5.0 ATTACHMENTS

Attachment IA91 Field Equipment Checklist.

Attachment I-A-9-1
Field Equipment Checklist

General

___ 1. Health and Safety Plan
___ 2. Site base map
___ 3. Hand calculator
___ 4. Brunton compass
___ 5. Personal clothing and equipment
___ 6. Personal Protective Equipment (First Aid kit)
___ 7. Cell or radio telephone

Environmental Monitoring Equipment

___ 1. Shovels
___ 2. Keys to well caps
___ 3. pH meter (with calibrating solutions)
___ 4. pH paper
___ 5. Thermometer
___ 6. Conductivity meter (with calibrating solution)
___ 7. Organic vapor analyzer or photoionization detector with calibration gas
___ 8. H2S, O2, combustible gas indicator
___ 9. Draeger tubes

Shipping Supplies

___ 1. Sample preservatives (nitric, hydrochloric, sulfuric acid/sodium hydroxide)
___ 2. Heavy-duty aluminum foil
___ 3. Coolers
___ 4. Ice packs
___ 5. Large zipper locking plastic bags
___ 6. Heavy-duty garbage bags
___ 7. Duct tape
___ 8. Strapping tape
___ 9. Paper towels
___ 10. Bubble pack, foam pellets, or shredded paper
___ 11. Vermiculite
___ 12. Cooler labels (“This Side Up,” “Hazardous Material,” “Fragile”)
___ 13. Federal Express/DHL labels

Sampling Equipment

___ 1. Tool box with assorted tools (pipe wrenches, screwdrivers, socket set and driver, open and box end wrenches, hacksaw, hammer, vice grips)
___ 2. Geologic hammer
___ 3. Trowel
___ 4. Stainless steel and/or Teflon spatula
___ 5. Hand auger
___ 6. Engineer’s tape
___ 7. Steel tape
___ 8. Electric water level sounder
___ 9. Petroleum Interface Probe
___ 10. Batteries
___ 11. Bailers (Teflon, stainless steel, acrylic, PVC)
___ 12. Slug test water displacement tube
___ 13. Vacuum hand pump
___ 14. Electric vacuum pump
___ 15. Displacement hand pump
___ 16. Mechanical pump (centrifugal, submersible, bladder)
___ 17. Portable generator
___ 18. Gasoline for generator
___ 19. Hose
___ 20. Calibrated buckets
___ 21. Stop watch
___ 22. Orifice plate or equivalent flow meter
___ 23. Data logger and pressure transducers
___ 24. Strip chart recorders
___ 25. Sample bottles
___ 26. 0.45-micron filters (prepackaged in holders)
___ 27. Stainless steel bowls
___ 28. SW scoop
___ 29. Peristaltic pump/tubing
___ 30. Sample tags
___ 31. SOPs, HAZWOPER training certificates, MSDs, FSP, QAPP

Decontamination Equipment

___ 1. Non-phosphate laboratory-grade detergent
___ 2. Selected high purity, contaminant free solvents
___ 3. Long-handled brushes
___ 4. Drop cloths (plastic sheeting)
___ 5. Trash container
___ 6. Galvanized tubs or equivalent (e.g., baby pools)
___ 7. Tap Water
___ 8. Contaminant free distilled/deionized water
___ 9. Metal/plastic container for storage and disposal of contaminated wash solutions
___ 10. Pressurized sprayers, H_2O
___ 11. Pressurized sprayers, solvents
___ 12. Aluminum foil
___ 13. Sample containers
___ 14. Emergency eyewash bottle
___ 15. Documentation Supplies

Documentation Supplies

___ 1. Weatherproof, bound field logbooks with numbered pages
___ 2. Daily Drilling Report forms
___ 3. Field Borehole Log forms
___ 4. Monitoring Well Installation Log forms
___ 5. Well Development Data forms
___ 6. Groundwater Sampling Log forms
___ 7. Aquifer Test Data forms
___ 8. Sample Chain-of-Custody forms
___ 9. Custody seals
___ 10. Communication Record forms
___ 11. Documentation of Change forms
___ 12. Camera and film
___ 13. Paper
___ 14. Permanent/indelible ink pens
___ 15. Felt tip markers (indelible ink)
___ 16. Munsell Soil Color Charts

MONITORING/SAMPLING LOCATION RECORDING

1.0 PURPOSE

This standard operating procedure (SOP) describes the guidelines for generating the descriptions and information to be recorded for each physical location where monitoring, or sampling is conducted.

2.0 PROCEDURES

2.1 SAMPLING LOCATION MARKING

Sampling locations are based on criteria presented in the SAP. Whenever possible, each sampling location will be marked by a wooden lathe stake, directly marking the surface with marking paint, or with surveyors flagging. Each should be labeled with the location identifier outlined in the SAP. This should be done during the site visit or as soon as is feasible during field activities. This is to give the utility locators a better idea of the specific area to be cleared. Having the locations marked will also assist the field crew gain a better perspective of the locations to be worked

2.2 PHOTOGRAPHIC DOCUMENTATION

Site photographs showing monitoring/sampling locations with respect to structures or the site in general are encouraged. At certain installations, photography must be approved by the Navy. Prior to commencing work, the Navy must be notified to determine if cameras are allowed at the installation. The Note that the Navy will likely inspect your camera and may purge/delete some pictures if they feel there is a security issue. When possible, a menu board included in the photograph can be used to give relative information regarding the project and location.
For each photograph, record the following information in the field logbook:

- Photo number
- Date and time of the photo
- Orientation of the photo (direction facing)
- Subject-a description of what is contained within the photo. Others may be using the photos that are unfamiliar with the site and locations.

A detailed description of field logbook entries can be found in SOP III-D, *Logbooks*.

2.3 MONITORING/SAMPLING LOCATION INFORMATION FORM

A Monitoring/Sampling Location Information form must be filled out to establish each new sampling location. This form must be provided to the Navy for inclusion into the NAVFAC NW NIRIS Database. Established locations should not be re-established unless new information (such as survey information) is recorded about a location. A location description may be provided about a sampling location. It should contain detailed information regarding the physical features surrounding the location, including relevant site information (i.e., obvious contamination, measurements to physical features, topographical relief, etc.). This description may be a copy of the field logbook or notes on project plan maps. These descriptions shall be attached to the field form. The PM is responsible for insuring that the project personnel have and

use consistent terminology and descriptions as established in the SAP. The reverse of the field form contains a brief discussion of the form and descriptions of the information requested on the front.

3.0 DOCUMENTATION

None.

4.0 REFERENCES

SOP III-D, *Logbooks*

5.0 ATTACHMENTS

Attachment IA101 Example Monitoring/Sampling Location Information Form

FORM 11-1A
MONITORING/SAMPLING LOCATION SUMMARY

Installation ID:	Establishing Contract ID:	Prime Contractor Name:	
Site Name:	DO/CTO:	Establishing Phase:	Date Established:
Survey Contractor:	Local System Description:		

Location Name	Location Type	Projection Specification	Coordinates		Ground Elevation (feet msl)
			Northing (feet)	Easting (feet)	

Location Types

ACID Acid Pit

ADIT Adit

AGT Above ground tank

AIR Air (not inside a building - ambient conditions)

AMB Ambient drinking water aquifer monitoring well

AOVM Ambient organic vapor monitor

ASBTS Asbestos-Containing Area

BAY Bay

BF Backfill

BH Borehole/Soil boring

BIN Roll-off bin

BIOL Biological (plant or animal)

BLDG Building (includes building air and building materials)

BULK Bulk sample

BURN Burn pit

CB Concrete boring

CENT Location surveyed at the center of a UST field

CLGP Canal Level Gauging Point

CPT Cone penetrometer

CY Cryopile

DCON Decontamination pad

DITCH Channel/Ditch

DP Direct Push/Geoprobe

DRN Drain

DRUM Drum/Container contents

DRW Drywell

DU Decision Unit

DW Domestic well

D_RIG_W Drill Rig Fluid Container

EC Electrode

ECT Electrode

EF System effluent

EVAP EVAPORATION POND

EXCV Excavation

FAGT Former above ground tank location

FL Fuel line

FLOOD Flood Plain

FLOOD_GATE Flood Control Gate

FLOOR Floor

FLOOR_SCRP Floor scrapings

FW Faucet/Tap/Spigot

GAGE Gaging station (not USGS)

GW Geoprobe well

GWTH Groundwater Test Hold

HA Hand auger

HDPCH Hydropunch

HOLE Hole

HP Holding pond/Lagoon

ID Indoors

IMP Import material

IN System influent

IT Intertidal

LAGOON Lagoon

LENTIC Freshwater, lentic

LF Landfarm

LGV Landfill Gas Vent

LH Leachate (Landfill)

LK Lake/pond/open reservoir

LOTIC Freshwater, lotic

LYS Lysimeter

MH Manhole/Catch basin

MS Sediment e.g., Marine Sediment

NQ Quality Control sample

ON Ocean, open water (not bay)

OTHER Other

OUTFALL Outfall

OW Oil-Water Separator

PARK Plantation/park/forest

PC Paint chip

PIPE Pipeline

PUBW Public drinking water well

PUMP_STATN Pumping station

RAIN_STATN Rainfall station

REF Reference

RES Residential garden/yard

RV River/stream

RW Recovery well

SBAG Soil bag

SE Seep

SG Soil Gas Probe

SIDEW Side Wall

SLAG Slag heap

SND_BLST Sandblast material pile

SP Spring/Seep

SPT Septic tank

SR Sewer System

SS Ground surface

STEAM_LN Steam Line

STKP Stockpile

STRM_DRN Storm drain

STRM_MH Storm drain manhole

SUBS Ground, sub-surface

SUBSLAB Subslab

SUBT Subtidal

SUMON Survey monument

SUMP Sump

SV Soil vapor extraction system

SWS Surface water body - nonspecific

SWSD Surface Water/Sediment

SWWP Wipe

SYSTEM Treatment system air or water

T Trench

TAA Temporary accumulation area

TAIL Mine tailings pile

TK Tank

TMPM Temperature Monitoring Point

TP Test Pit

TRANS Transformer

TUNNEL Steam tunnel sampling location

TWP Temporary well point

UGA Geophysical anomaly

UNK Unknown

USGS USGS gauging station

UST Underground storage tank

UXO UXO

UXO_G UXO grid

UXO_P UXO point

VAULT Vault

VPB Vertical profile boring

WALL Wall

WEEP Weep hole

WF Waste water treatment facility

WL Well

WLAM Alluvial Monitoring Well

WLBM Bedrock Monitoring Well

WLE Extraction well

WLEA Alluvial Extraction Well

WLEB Bedrock Extraction Well

WLHM Hybrid Monitoring Well

WLI Injection well

WLIA Alluvial Injection Well

WLIM Interface Monitoring Well

WLL Leaching Well

WLM Monitoring well

WLS Sparge well

WLSG Soil gas probe/Well

WRP Waste rock pile

WSFI Water system facility intake

WT Wetlands

WW Waste water

Recorder: _____ Date: _____

Checker: _____ Date: _____

SAMPLE NAMING

1.0 PURPOSE

This standard operating procedure (SOP) describes the naming convention to be used for samples collected, analyzed, and reported for the U.S. Naval Facilities Engineering Command Northwest (NAVFAC NW) projects. Unique sample identifiers are used to facilitate tracking by laboratory and project personnel and for purposes of storing, sorting, and querying data in the NAVFAC NW NIRIS database.

2.0 PROCEDURES

The contractor is responsible for assigning a unique sample ID to every individual sample collected. The contractor may use his or her own designations as long as the sample ID does not already exist in the NIRIS database. The contractor must also clearly identify which samples are field duplicates. This applies to both historical and planned sampling events. The used sampling identification scheme shall be identified and outlined in the field sampling plan.

3.0 DOCUMENTATION

All sample collection information must be recorded within the field logbook. Each sample collected will be clearly associated with the sample location (installation, site, and well or sample point location), matrix type, sample type (i.e. environmental, field duplicate, equipment rinsate), collection date and time, sampling method, and sampling depth (if appropriate). Only data codes and location IDs associated with NIRIS and NAVFAC NW's electronic deliverables SOP (NAVFAC NW 2015) shall be used.

Any sample submitted for analysis shall be documented using a completed chain-of-custody (COC) form that must accompany the shipment and a copy retained for the project records. Samples submitted to an EPA laboratory shall also include a completed EPA analysis request form. The COC/analytical request form must be used to track all sample IDs.

4.0 REFERENCES

NAVFAC NW. 2015. Navy Environmental Data Transfer, Version 5.0.

5.0 ATTACHMENTS

None.

BOREHOLE ABANDONMENT

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to outline the methods by which all U.S. Naval Facilities Engineering Command Northwest (NAVFAC NW) personnel and/or their contractors should perform borehole abandonment. The procedure provides descriptions of equipment, field procedures, and necessary documentation.

2.0 PROCEDURE

The procedures used in boring abandonment should ideally accomplish two objectives: (1) protect aquifers from cross-contamination by sealing the borehole, and (2) restore the strata in the borehole to nearly original conditions by selective placement of fill material. Be advised that regulators in all states may not approve objective 2.
Field staffs are required to be familiar with applicable state regulations concerning borehole abandonment. The work plan should be consulted for project-specific requirements. The following sections discuss various types of abandonment procedures.

2.1 EQUIPMENT

Equipment and materials used during borehole abandonment include:

- Drill rig (trailer-mounted mixer and grout pump)
- Wheelbarrow or drum
- Filter pack material
- Bentonite powder to make appropriately weighted slurry
- Bentonite pellets (seal) or chips
- Portland cement, Type II
- Water from an approved source
- Weighted tape measure
- Tremie pipe (small-diameter, rigid PVC pipe)
- Weatherproof bound field logbook with numbered pages
- Indelible/permanent marking pens
- Appropriate health and safety equipment
- Appropriate equipment for managing investigation-derived waste

2.2 CLOSING BOREHOLES

The following procedures should be used for the closure of boreholes located more than 15 feet from an existing or planned well:

1. Pull, drill out, or thoroughly pierce any temporary casing.
2. Use tremie pipe to place grout from the bottom of the hole to within 3 feet of the ground surface.
3. Allow grout to settle for 24 hours.
4. Fill the remainder of the hole with concrete.

5. Mound and smooth the surface of the concrete and inscribe it with "ABD" (for abandoned), any assigned well or boring designation, and the date the hole was abandoned.
6. Include all boring logs, samples, completion records, and abandonment procedures in the records of work on the site.
7. File any required documentation with state regulatory authority.

2.3 CLOSING BOREHOLES WITHIN 15 FEET OF A MONITORING WELL

If the hole is within 15 feet of a monitoring well in the same aquifer, or if a replacement well is to be installed within 15 feet of the abandoned borehole, the following procedure should be followed. This procedure must be pre-approved by regulators in some states.

1. Pull, drill out, or thoroughly pierce any temporary casing.
2. Use tremie pipe to backfill the hole with filter pack material opposite sand strata and bentonite or grout opposite substantial (2 feet or thicker) clay and silt strata. Where the filter pack material approaches the ground surface, 2 feet of bentonite should be placed above the filter pack material, and a 3-foot concrete plug placed to the surface. Otherwise, backfill materials should be placed from the bottom of the hole to within 3 feet of the ground surface.
3. Allow these materials to settle for 24 hours.
4. Fill the remainder of the hole with concrete.
5. Mound and smooth the surface of the concrete and inscribe it with "ABD" (for abandoned), any assigned well or boring designation, and the date the hole was abandoned.
6. Include all boring logs, samples, completion records, and abandonment procedures in the records of work on the site.
7. File any required documentation with state regulatory authority.

2.4 SHALLOW BORINGS NOT PENETRATING WATER TABLE

Shallow borings made for the collection of subsurface soil samples should be abandoned by backfilling the hole with cuttings from the hole, if and only if the boring does not penetrate the water table. Clean sand should be used to make up any volume not filled by the cuttings. Some states DO NOT permit the backfilling of any borehole with cuttings in any instance and require that borings be sealed with grout.

2.5 SHALLOW BORINGS PENETRATING THE WATER TABLE

Shallow borings that were made for the collection of subsurface soil samples and that penetrate the water table should be abandoned by grouting the hole from the bottom to the surface as described in Section 2.2.

2.6 DEEP STRATIGRAPHIC BORINGS

Deep stratigraphic borings are normally located in areas that, by virtue of the historical record, are presumed relatively uncontaminated. Therefore, these borings are usually more than 100 feet from any sampling well locations. Any boring located within 15 feet of a proposed well location, or located directly upgradient or downgradient (on anticipated flow line) of a proposed well location, should be abandoned by placing clean sand in the aquifer intervals and bentonite or grout in aquitard intervals as described above (if allowed by state regulations). If the boring is

over 15 feet from or not upgradient of a proposed well location, the boring will be completely filled with grout.

2.7 GROUT

Grout used in construction will be composed as follows:

- 20 parts by weight of cement (Portland cement, Type II or V)
- 0.4 to 1 part, by weight, of bentonite (maximum) (2 to 5 percent)
- 8 gallons (maximum) approved water per 94-pound bag of cement

Local regulations may state the requirements for grout. For instance, the New Jersey regulations list sterilized clay slurry weighing not less than 14 pounds per gallon, cement grout, neat cement, or concrete.

Additives or borehole cuttings should not be mixed with the grout. Bentonite should be added after the required amount of cement is mixed with the water.

All grout material should be combined in an aboveground container and mechanically blended to produce a thick, lump-free mixture. The mixed grout should be recirculated through the grout pump prior to placement.

A trailer-mounted mixer with grout pump and attached hose can be used in place of a mud rotary-equipped drill rig. For shallow borings 1 to 3 feet below ground surface, the grout may be mixed in a wheelbarrow or drum and poured or shoveled into the borehole.

Grout should be placed using a commercially available grout pump and a rigid tremie pipe. Grouting should be accomplished in stages, aquifer by aquifer, sealing the boring from the bottom to ground surface. This should be accomplished by placing a tremie pipe to the bottom and pumping grout through the pipe until undiluted grout reaches the bottom of the next higher section of casing or, for the topmost section, until grout flows from the boring at ground surface. After 24 hours, the abandoned drilling site should be checked for grout settlement. On that day, any settlement depression should be filled with grout and rechecked 24 hours later. This process should be repeated until firm grout remains at the ground surface.

3.0 DOCUMENTATION

The following information should be logged in the field logbook:

<u>General requirements</u>

- Date
- Time
- Boring location
- Personnel/subcontractor on site
- Visitors

<u>Specific to abandonment</u>

- Start/end times
- Depth of boring
- Materials used to seal each stratum
- Detailed description of procedure
- Date/time of return visit(s)
- Activities performed on return visit(s)

All entries in the field logbook must be printed in black ink and legible.

4.0 REFERENCES

SOP III-D, *Logbooks*

5.0 ATTACHMENTS

None.

GPS SURVEYING

1.0 PURPOSE

This standard operating procedure (SOP) sets forth the protocols for recording and processing Global Positioning System (GPS) data. This document describes GPS data management for field data collection referencing Trimble systems including Pro 6T/6H, GeoXH 6000, Geo 7x or other handheld units capable of obtaining sub-meter accuracy.

2.0 PROCEDURE

2.1 PROCESS OVERVIEW

Surveyors typically collect GPS data using a data logger and record data both manually and electronically. In addition to the field forms and electronic field data, each surveyor maintains field notebooks summarizing their observations and other pertinent field data. The electronic data is exported electronically from the device, post-processed (if applicable), and then imported into GIS or CAD software.

In many areas of the U.S., GPS data can be recorded in real-time using the Coast Guard Differential GPS beacon system, Omnistar® or other satellite service. However, random positional errors can be encountered when collecting real-time data that makes positional validation difficult. Therefore, it is important to post-process all GPS data if corrections are not applied in real-time. Post-processing removes the random drift of the GPS positional signal by correcting the field rover GPS unit against a GPS base station that has a known position. Most projects will use CORS (Continuously Operated Reference Station) base stations surveyed by the NGS (National Geodetic Survey) to provide the post-processing solutions. This is an accepted industry standard. Using Trimble GPS rover units corrected against CORS base stations should allow projects to achieve sub-meter positional accuracy.

2.2 FIELD DATA COLLECTION

The GPS equipment is portable and uses satellite technology to provide accurate location information. Each object or *feature* collected with a GPS data collector can be one of three shapes: a *point*, a *line*, or an *area / polygon*. Each feature has real-world coordinates, as well as descriptive information or *attributes* such as site name or type, or observations recorded. The location and attribute information gained from the GPS data collection effort can be integrated with existing base data.

Preparatory tasks completed prior to the start of GPS data collection and field work include preparation and loading of “waypoints” to GPS units for field navigation, development of a data dictionary / data structures or interactive data collection forms for collecting survey information, and unit configuration to ensure that only good quality GPS data is recorded.

2.2.1 GPS Operation

Data dictionaries / structures, electronic forms, GIS MXD files and base layer data can be loaded onto the GPS devices. These electronic files facilitate collection of field measurements directly into the GPS device. They also serve to standardize the data collection effort, verify proper data

recording, and ensure that all required data fields are present. This approach also reduces the amount of equipment surveyors will need to carry when they are collecting their measurements.

2.2.2 GPS Accuracy

The accuracy of GPS receivers without real-time or post-processed differential correction is on the order of 100 meters / 330 feet (2dRMS). After differential correction, the horizontal accuracy of each position can be better than 50 cm / 1.6 feet (RMS) + 1 ppm times the distance between the base and rover. The vertical accuracy of each position can be sub-meter + 2 ppm times the distance between the base and rover. Using real-time corrections, the accuracy of each position can be as good as a submeter, but is subject to a number of operational conditions. Note: 2dRMS means that approximately 95% of the positions are within the specified value. RMS means that approximately 68% of the positions are within the specified value.

2.2.3 Increasing GPS Accuracy

To verify the positional accuracy of a survey, standard survey practice requires that a known control be recorded during a survey. At a minimum, one first-order NGS monument (or equivalent) must be recorded during the GPS survey for each day of the survey. Therefore, during post-processing, general errors in the base or rover GPS units may be revealed. However, in recording only one monument, there is no way of fixing the error (only in knowing that error exists). Ideally, three first-order NGS control points completely surrounding the survey area should be recorded during the survey day – ideally one at the start of the survey, and two at the end of the day. This will allow a Professional Land Surveyor (PLS)/GIS post-processor to be able to shift and rotate the data if serious positional errors are found with all three control points.

2.2.4 Field Data Recording

To ensure that only quality data is recorded, the following data collection settings are recommended for the GPS unit:

Number of Satellites: Over-determined 3D (>=5 satellites)
PDOP Mask: 6
SNR: 6
Elevation Mask: 15 degrees

2.2.5 Naming the Data Logger Files

File naming conventions should be developed for all electronic field data. The file name should include information about the field surveyor / crew, date stamp for when the data was collected, and a unique identifier for the file if more than one file is collected throughout the day. It is recommended that the user save data several times throughout the day in the rare case that a data file becomes corrupt. An example file naming convention is shown in the following example:

Field Data File: JGB_20140607_A
JGB: First, second, and third characters represent initials of GPS operator or field crew
20140607: YYYYMMDD
A: Eighth character represents a unique character if more than one file is collected in the day.

2.2.6 Base Station Data Processing

The field staff and the PLS/GIS specialist should review GPS data for attribute correctness. Then, the PLS/GIS specialist should post-processes the data. In the post-processing, the

uncontrolled drift of the measurements recorded in the rover GPS units are corrected against the known drift recorded in the base station GPS units using Trimble Positions (or equivalent / most current) software. The .SSF file created by the GPS device is corrected by use of the base station data. The correction process converts the raw data file (.SSF) to a corrected file (.COR). In addition to post-processing to only one base station, the data can be post-processed to several base stations to give the data more positional accuracy. After post-processing, the data is converted to an ESRI file geodatabase or point file (.csv/.txt) for import to GIS or CAD software.

2.2.7 Field Staff Spatial/Attribute Review

After each survey, all field data should be reviewed by a field crew member for accuracy and completeness. This can be done during or after the post-processing as this review is performed only to ensure that the field crew assigned the proper attribute data to the file. Any incomplete data can be filled-in by referencing the field notes. Field staff compares the number of data points collected in the GPS device file to the number of data points listed on the field forms to make sure they match. During this step, the field crew checks to see that all of the data in the data file is accounted for.

2.2.8 PLS/GIS Specialist Post-Processing

For Trimble GPS units, Positions software is used to download the electronic file to review the source file content, and post-process the data. The post-processor reviews the data file to check the settings the GPS data was collected under by the field crew. This process ensures the field crew used the proper GPS configuration settings while collecting the data. Thereafter, the data is post-processed against a base station. Ideally, it is post-processed against a 5-second base station within close proximity to the survey site. The referenced Trimble GPS units should be able to achieve a horizontal accuracy of 50 cm / 1.6 feet (RMS) at a 1 km base line (distance from the base to the rover). Accuracy degrades by 1 ppm as the distance between the base station and the rover increases. For example, 1 mm of degradation occurs for every kilometer between the base and rover. Data must be captured within 500 km (310 miles) of the base station to obtain sub-meter accuracy (RMS). If a 5-second base is not available, a thirty-second base is acceptable as long as the rover GPS units are recording each position for a minimum of one minute. This process ensures both the rover and the base records a minimum of one epoch per location. Recorded time that is less than this amount causes the position to be interpolated by the software, decreasing its accuracy.

Every 100 km (62 miles) in distance between the rover and base adds 0.1 m (0.33 feet) to the positional accuracy. Therefore, it is best to use a base that is very close to the survey site.

To help guarantee sub-meter results at the 95% (2dRMS) level, three NGS control points can be surveyed as stated above in the “Increasing GPS Accuracy” section. Therefore, if the three NGS control points show corrected horizontal accuracy of 0.2 (0.66 feet) , 0.4 (1.31 feet), and 0.6 meters (2.0 feet) respectively, it can be determined that the average of those values reflects the relative GPS survey accuracy for that day, i.e. 0.4 meters (1.31 feet).

In addition, the GPS field survey positions can be post-processed against several base stations in a short amount of additional time. It also allows the PLS/GIS Specialist to verify the positional accuracy of the GPS data by computing average and standard deviation values for the field survey positions in relation to more than one base. Thus, ensuring there are no errors in the base correction.

The post-processing methods, GPS configuration settings, and GPS collection methods should be recorded in metadata documentation defending the stated accuracy of the GPS survey.

2.2.9 GIS File Production

After the geographic and attribute data has been reviewed, and the file has been post-processed, the data is exported to an ESRI file geodatabase or shapefile format for use in a GIS. A shapefile should only be used if a CAD platform is anticipated for map production. Alternatively, the data may also be exported in a simple ASCII point file (.csv or .txt) with delimiters separating attributes.

3.0 DOCUMENTATION

Surveyors shall record field notes daily using industry accepted practices. The data shall also be neat, legible and easily reproducible. Copies of the surveyor's field notes and calculation forms generated during the work shall be transferred to the Navy.
Surveyor's field notes / documentation shall, at a minimum, clearly indicate:

- The date of the survey
- General weather conditions
- The name of the surveying firm
- The names and job titles of personnel performing the survey work
- Equipment used, including serial numbers and calibration records
- Field book designations, including page numbers

Drawings and calculations submitted by the surveyor shall be signed, sealed and certified by a Professional Land Surveyor (PLS) registered in the state or territory in which the work was done. Dated records of land surveying equipment calibration and equipment serial numbers shall also be provided in the in the submitted documentation.

4.0 REFERENCES

The detailed requirements in the Geographic Data, Survey Specifications subsection of the parent compendium (NAVFAC Northwest SOPs V5.0) also apply and are not repeated here in this field procedure. These should be consulted as part of any GPS Surveying effort. In addition, NAVFAC Northwest Cadastral Team, Record of Survey or other requirements may apply to the project, an example of their requirements can be found with the Survey Specifications referenced above.

5.0 ATTACHMENTS

None.

FIELD QC SAMPLES (AIR)

1.0 PURPOSE

This procedure describes the standard quality control (QC) and quality assurance (QA) procedures for air monitoring field samples.

2.0 PROCEDURE

NAVFAC NW air monitoring programs for airborne pollutants consist of complex activities using monitoring equipment and laboratory analysis techniques. This approach is necessary to accurately quantify concentrations of airborne pollutants in ambient air. Therefore, it is critical that one ensures and maintains a high-quality program, by implementing the appropriate QA/QC program elements.

The terms quality assurance and quality control (QA/QC) are often confused. Both activities are concerned with maintaining consistent and verifiable quality in each element of the program. Strictly speaking, QC applies to measures taken, on an ongoing basis, by personnel involved in producing the primary output of the activity. These actions are taken to maintain performance parameters within acceptable levels. An example of a QC activity is a routine zero/span calibration check of a monitoring instrument by the responsible operating technician.

Quality assurance, on the other hand, refers to checks or tests performed by personnel other than the primary operators to verify that the performance parameters have, in fact, been maintained within acceptable limits. Examples of QA activities are performing a quarterly audit of monitoring instruments and checking output data for "out-of-limits" values. In the discussion that follows, QA/QC is used as a general term to encompass both QA and QC activities.

A rigorous QA/QC effort is necessary during the operation of NAVFAC NW site air monitoring programs to meet monitoring objectives. Major QA/QC elements that should be implemented during the operational phase of an air monitoring program include QA/QC management, sample QA/QC, analytical QA/QC, and data reduction QA/QC.

QA management involves implementing project-specific task order administrative procedures to control QA/QC functions. The potential for, and types of, quality problems vary depending on the activity: sampling, analysis, or data reduction. Therefore, individual QA/QC requirements must be developed for each of these activities. Summaries of typical sampling and analysis frequencies, QA/QC requirements, and calibration requirements for sampling and analysis instrumentation are presented in Tables III-C-1 and III-C-2, respectively. Data recording procedures to be specified in the air sampling activity include: (1) periodic readings of the temperature, air flow, volumes, and other parameters; (2) documentation of meteorological conditions at appropriate time points; (3) documentation of instrument operating variables (i.e., resin cartridge number); (4) documentation of any upset conditions such as sudden leakage or pressure surges; and (5) documentation of calibration or maintenance activities. A logbook for the overall field program in which sampling descriptions, meteorological data, and upset conditions are documented should be maintained. In addition, a sampling data sheet should be prepared for each sample or set of samples in which the periodic readings and instrument parameters are recorded. Certain measurements, such as filter numbers and weights or impinger

volumes that are required for analytical purposes, may be recorded on a separate sheet with provisions for recording subsequent analytical data on the same sheet. Separate maintenance and calibration logbooks should be maintained for each sampling/monitoring instrument. In most cases, sampling data forms specific for a given Task Order must be prepared because of difference in the sampling design between Task Orders.

Table III-C-1
Typical Sampling/Analysis Frequencies
for QC Samples (Air)

Type of Sample*	Description	Typical Frequency
Field Blanks	Collection media shipped to the field and exposed to the sampling environment, recapped or reclosed without a volume of air passing into or through them.	At least one per day or one per each sample batch up to 10% of sample total.
Laboratory Blanks	Unexposed media that do not go to the field but are analyzed by the laboratory to confirm that analyte(s) of concern are not present.	At least one per day or one per each sample batch up to 10% of sample total.
Trip Blanks	Collection media shipped to the field, remains unopened or unexposed to the test environment and are returned to the laboratory.	At least one per shipment container.
Spiked Samples	Media to which a known amount of the analyte(s) of interest have been added by the laboratory and shipped to the field for use.	At least one per batch up to 10% of sample total.
Collocated Samples	Air samples collected immediately adjacent to each other in the same time period.	At least one per matrix in each sampler array matrix up to 10% of sample total.
Instrument Calibration Standards	Calibration devices or material traceable to known certified standards.	Test at least twice daily at beginning and ending of sampling period.

* Specification of the manner in which these types of samples are collected shall be described within the specific Task Order Work Plan or Field Sampling Plan.

Table III-C-2
Calibration Requirements for
FIELD Air Sampling and Analysis Instrumentation

Device	Parameter Calibrated	Method of Calibration	Approximate Frequency	Comments
Sampling Instrumentation				
Sampling flow rate measurement device	Flow rate	Flow calibration kit; primary standard film calibrator; calibration flow meter; dry test meter	Depends on sampler; generally immediately prior to and after sampling event	
Sample volume measurement device (usually a dry test meter)	Total volume	Wet test meter or any appropriate volume standard	Depends on sampler; generally immediately prior to and after sampling event	Must be determined at known atmospheric pressure and temperature. Flow rate should be similar to that used for sampling.
Analytical Instruments				
Continuous monitors (i.e. FID, PID, FPD, etc.)*	Response	Use standard concentrations. Calibrate with chemical of concern or reference compound if relative response of analyte(s) is known	Daily or more frequently, if required	
Field Gas Chromatographic Instruments	Calibrate instrument and verify column performance and retention time for each analyte	Injection of standard using the same process as for sample injection	Daily or more frequently, if required	
GC/MS*	Calibrate instrument and verify response and retention time for each analyte	Same as for other gas chromatographic instruments	Same as for other chromatographic gas instruments	Same as for other gas chromatographic instruments

Device	Parameter Calibrated	Method of Calibration	Approximate Frequency	Comments
GC/MS	Mass spectral resolution and tuning parameters	(a) Introduction of perfluorotributyl-amine compound (tuning compound) directly into MS. (b) Injection of tuning verification standard (i.e., 4-bromo-fluoro-benzene) into GC	Daily	Selection of tuning standards (i.e., decafluorotriphenyl phosphine or 4-bromofluoro-benzene) will be dependent on type of analysis being performed

FID - Flame Ionization Detector
PID - Photoionization Detector
FPD - Photometric Detector
GC/MS - Gas Chromatograph/Mass Spectrometer
NIST - National Institute of Standards and Technology
SRM - Standard Reference Material
CRM - Certified Reference Material

In addition to site-specific air sampler(s) and meteorological station parameters, the monitored area or locale elevation (i.e., feet above mean sea level) should be noted, and data for the following meteorological parameters taken/recorded every four hours: ambient air temperature, relative humidity, and barometric pressure. The two former values are obtained with a field or pocket type thermo-hygrometer; barometric pressure values may be obtained from either a nearby National Weather Service station or an airport that measures/records this parameter. Most meteorological data is recorded as either 15-minute or 1-hour averages; this includes wind speed and wind direction.

Sample labeling, preservation, storage, and transport procedures should be specified, and these procedures should be carefully explained to field personnel prior to sampling to ensure proper implementation. Sample labels, prepared in advance, should include sufficient information to associate the sample with a particular data sheet as well as the overall program record notebook. In general, each sample should be given a unique identification number with a prefix describing the type of sample.

Sample preservation, storage, and transport procedures must be appropriate for the type of analyses required. Particulate samples generally should be placed in air-tight containers and stored in the dark to minimize analyte degradation. Resin cartridges and impingers generally require more attention, because of analyte instability in the matrix, and should be shipped to the laboratory on the same day that the sample was collected for analysis. These sample types should be placed in airtight, glass containers and stored at subambient temperatures (U.S. Environmental Protection Agency methods generally specify a storage temperature of 4 degrees Celsius) until analysis. Exposure to solvents must be avoided for resin cartridges during all stages of handling in order to avoid sample contamination. Air samples collected in Tedlar™ bags should be placed within an opaque plastic bag (i.e., plastic garbage can bag) and then placed in a cardboard box with blue ice for shipment to the laboratory on the same day that the sample was collected for analysis.

Chain-of-custody forms are required. The objective of the chain-of-custody procedure is to document the movement of a sample from collection until analysis to ensure its integrity.

2.1 ROUTINE QA/QC CHECKS

The field air monitoring program should incorporate the following four-component approach for routine QC and QA checks:

- Use collocated (i.e., two separate ambient air samples collected side-by-side at the same sampling location) samples for precision checks. Alternatively, split each sample stream into two collection devices (collocation does not guarantee collection of identical samples).
- Use blanks (i.e., field and trip blanks) for ambient locale and shipping container contamination checks.
- Use analytical standards and equipment calibrations for accuracy checks.
- Perform data review for internal consistency.

During each air monitoring program, one station with two sets of collocated air samplers should be used in accordance with siting criteria. The goal should be to obtain at least 10% of collocated samples for each monitoring network. The analytical results from the collocated air samples should be used to assess the precision and overall homogeneity of the samples, including the influence of the combined field and analytical procedures.

The purpose for collecting sample blanks (i.e., field blank) is to document that extraneous concentrations of the target analyte(s) are not introduced into the collection medium simply by handling or working with it in a normal, routine fashion. Generally, one field blank per collection medium per day is sufficient to demonstrate that the levels of target analyte(s) found in the normal handling of the media in the field. In some instance, where the field environment is known to have high levels of contaminants, collecting more field blanks may be deemed appropriate. However, those requirements should be identified in the Field Sampling Plan. The frequency of field blanks collected for a particular project is project-specific and should be presented in detail in the Work Plan or Field Sampling Plan.

The exact procedure for collecting field blanks is specific to each type of medium. However, the general concept is to handle the field blank media in exactly the same fashion as the media used for actual sample collection, except no sample volume of air is moved through the media. For example, glass sorbent tubes used for field blank are shipped, labeled, have their ends broken open, are placed in the sampling mechanism, removed from the sampling mechanism, capped, logged, and packed for shipment in an identical manner to the sorbent tubes used to collect air samples. The difference is that no air is pulled through the field blank sorbent tubes. Table III-C-3 lists general procedures for collecting field blanks for some of the collection media.

Canisters (i.e., Summa™ - stainless steel, SilcoCan™ - fused silica/stainless steel) used for ambient air sampling purposes should be tested to determine vacuum/pressure condition before and after sampling. Evacuated canisters should undergo two separate tests. First, the canister is checked with a vacuum gauge to determine negative pressure, and then the attached critical flow orifice that is used to control flow during the prescribed sampling interval is tested with a rotometer. Canisters used with a positive displacement sampling pump (i.e., canister at atmospheric pressure at the start of the sampling period and then pressurized under constant flow pump conditions to approximately two atmospheres) should be tested for pressure conditions with a pressure gauge. Additionally, the sample pump flow rate should be determined with either a film calibrator or a flowmeter kit and stopwatch.

A vacuum/pressure gauge known to be free of contaminants is attached to the fitting upstream of the canister's main valve. The main valve is then opened and the gauge is read to confirm that the evacuated canister has maintained the same vacuum reading, within ± 5%, reported by the

laboratory or provider of the canister. The same method is used by the laboratory to confirm the fill of a pressurized canister. The canister should be within ± 5% of the pressure valve reported by the field crew. Canisters outside the gauge error margin should be flagged as suspect and their data should be qualified accordingly.

2.2 PERIODIC QA/QC CHECKS

Periodic field QA/QC checks should be implemented to supplement the more frequent routine QC checks required by the project. These periodic checks will serve to determine compliance with siting and operating criteria and should be made after the specific Task Order air monitoring plan is in full operation. The periodic QA/QC checks should include air matrix spiked samples, instrument performance audits of the air monitoring and meteorological equipment, and system audits.

The accuracy of sample analysis can be routinely checked by submitting spiked and blank gas samples as part of the laboratory analysis package. Spiked samples should contain a known concentration of some of the same compounds for which the laboratory is performing analysis. Blank samples are collection media that have no measurable amounts of the substance(s) of interest. The analysis of spiked and blank samples should be reported along with the normal samples collected during the project.

Instrument performance audits conducted on air sampling and meteorological measurement equipment are to be conducted by qualified air quality technicians who are not directly involved in the routine operation of the air monitoring activity. In addition, the auditing equipment used to conduct the tests must be independent and different from that used to calibrate or maintain the air monitoring instrumentation. The audited instruments are challenged with known input values (e.g. air flow rates, electrical signals, timing mechanisms, temperature environments, etc.) and the instruments' observed response to the known inputs is reported.

The system audit provides an onsite qualitative evaluation of the installation of air sampler array and the meteorological monitoring station. The system audit documents the following:

Table III-C-3
Common Field Blank Collection Procedures (Air)

Media	Field Blank Collection Procedure
Glass Sorbent Tubes	The tubes are removed from their shipping package and labeled as if they were to be used to collect samples. The tube ends are snapped off and the tubes are placed in the sampler mechanism (e.g., personal sampling pump or flow control device). Without turning on the sampler mechanism, the tubes are then removed, capped, logged, and placed in the shipping container along with regular samples for transport to the laboratory. Analysis of the field blank tubes is identical to the tubes used to collect air samples.
Filters	The filters to be used as field blanks are removed from their shipping package and tagged or labeled along with the filters intended for sample collection. They are installed in the filter holder, attached to the sampling mechanism (pump), removed without turning on the sampling mechanism, placed in a protective envelope, logged on the appropriate form, and shipped along with regular samples to the laboratory for analysis. Note: Many filter media come preloaded in individual cassettes with capped ends. If filter cassettes are used, field blank procedures similar to those described for sorbent tubes will apply. Filters used for particulate measurement are pre-numbered and pre-weighed. Handling should be minimized.
Liquid Impingers	The impinger solution is placed in the impinger as it would be under normal sampling procedures. Without moving a volume of air through the impinger, an aliquot of solution (5-10 milliliters) is transferred into a shipment bottle, labeled, logged, and packaged for shipment to the laboratory along with the exposed sample impinger solutions.
Tedlar™ Bags	The empty bags intended for field blanks are removed from the shipping package and labeled as if they are to be used for normal sample collection. The field blank bags are then filled with ultra-pure nitrogen, logged, and packaged for shipment to the laboratory along with normally collected air samples.
Summa™ Canisters	The canisters intended for field blanks are removed from the shipping container and labeled as if they are to be used for normal sample collection. The field blank canisters are then filled with ultra-pure nitrogen, logged, and packaged for shipment to the laboratory along with canisters used for normally collected air samples.

General physical condition and operability of the air sampling and meteorological instrumentation

- Operational QC procedures in use (i.e., calibrations, single-point checks, instrument operation check lists, documentation)
- Instrument siting and exposure criteria
- Data acquisition, validation, and reporting procedures

The frequency of periodic QA/QC checks depends on the duration of the project. Where a long-term (i.e., 6 to 12 months, or more) project is in effect, the periodic QA/QC checks should be performed quarterly. For short-term projects lasting only a few weeks or less, an initial QA/QC check at the beginning, followed by a final check at the project's end, may be sufficient. Any problems or discrepancies discovered during the performance and system audits are documented in a report and discussed with the respective Project Manager who will initiate the required corrective actions.

2.3 LABORATORY QA/QC PROGRAM

Laboratory analytical techniques must properly identify the sample components and accurately and precisely measure concentrations. This requires the preconcentration and/or storage of air

samples. Therefore, methods chosen for time-integrated monitoring usually involve a longer analytical time period, more sophisticated equipment, and more rigorous QA procedures. Canister sampling includes replicate analyses and duplicate canisters to assess analytical and sampling precision. Analysis of co-located duplicate samples with laboratories is desirable to check laboratory analytical performance.

Laboratory QC methods for an NAVFAC NW project site must include the requirements noted in Section 3.0 of the *Navy Installation Restoration Laboratory Quality Assurance Guide (Interim Guidance Document)* (NFESC 1996). For air monitoring projects, these requirements should address the following elements: laboratory control samples, matrix spikes/matrix spike duplicates, duplicates, blanks, surrogates, other laboratory QC samples, field QC samples, internal standards, calibration standards, and canister cleanup and certification. Inter-laboratory analysis of duplicate or collated samples is desirable to check laboratory analytical performance.

3.0 DOCUMENTATION

Field QA/QC Samples (Air) shall be documented as prescribed in the respective NAVFAC NW Task Order and/or associated Air Monitoring Plan. These items shall be sent to the Project Manager and the project files.

4.0 REFERENCES

ASTM. 1995. "Standard Practice for Planning the Sampling of Ambient Atmosphere," D-1357-95, American Society for Testing and Materials, Philadelphia, PA.

CMA. 1990. "Chemicals in the Community: Implementing Regional Air Monitoring Programs," Chemical Manufacturers Association, Washington, DC.

NIOSH. 1984. "NIOSH Manual of Analytical Methods," PB 85-179018, National Institute of Occupational Safety and Health, Cincinnati, OH.

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Ogden. 1991. "Ambient Air Quality Monitoring Program Quality Assurance/Quality Control Manual," Ogden Environmental and Energy Services Co., Inc., San Diego, CA.

USEPA. 1977. "Quality Assurance Handbook for Air Pollution Measurement Systems, Volumes I and II," EPA-600/9-76-005, Office of Research and Development, Research Triangle Park, NC.

USEPA. 1983. "Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air," EPA-600/4-83-027, Office of Research and Development, Research Triangle Park, NC.

USEPA. 1983. "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans," QAMS-005/80, EPA-600/4-83-004, Quality Assurance Division, Washington, DC.

USEPA. 1984. "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air," EPA-600/4-84-041, Office of Research and Development, Research Triangle Park, NC.

USEPA. 1984. "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I - Principals," EPA-600/9-76-005, Research Triangle Park, NC.

USEPA. 1986. "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air," EPA-600/4-87-006, Office of Research and Development, Research Triangle Park, NC.

USEPA. 1986. "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II - Ambient Air Specific Methods," EPA-600/9-77-027a, Research Triangle Park, NC.

USEPA. 1987. "Compendium Method TO-12: Method for the Determination of Non-Methane Organic Compounds (NMOC) in Ambient Air Using Cryogenic Preconcentration and Direct Flame Ionization Detection," Quality Assurance Division, Research Triangle Park, NC.

USEPA. 1987. "Ambient Monitoring Guidelines for Prevention of Significant Deterioration (PSD)," EPA-450/4-87-007, Research Triangle Park, NC.

USEPA. 1988. "Compendium Method TO-14: The Determination of Volatile Organic Compounds in Ambient Air Using Summa Passivated Canister Sampling and Gas Chromatographic Analysis," Quality Assurance Division, Research Triangle Park, NC.

USEPA. 1989. "Air/Superfund National Technical Guidance Study Series, Volume IV-Procedures for Dispersion Modeling and Air Monitoring for Superfund Air Pathway Analysis," EPA-450/1-89-004, Section 3, Office of Air Quality Planning and Standards, Research Triangle Park, NC.

USEPA. 1989. "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV - Meteorological Measurements," EPA-600/4-90-003, Research Triangle Park, NC.

5.0 ATTACHMENTS

None.

LOGBOOKS

1.0 PURPOSE

This standard operating procedure (SOP) describes the activities and responsibilities of U.S. Naval Facilities Engineering Command Northwest (NAVFAC NW) personnel and/or their contractors pertaining to the identification, use, and control of logbooks and associated field data records. This SOP establishes a standard format for recording field observations and describes the methods for use and maintenance of field logbooks.

2.0 PROCEDURE

2.1 EQUIPMENT

- Waterproof hardbound field logbook (typically 4-inch by 7-inch to 8-inch by 10.5-inch) with numbered pages
- Waterproof/indelible marking pen
- Ruler/straight edge
- Clipboard

2.2 LOGBOOK MAINTENANCE

Prior to commencement of field work, logbooks will be assigned to field personnel by the Project Manager. If personnel changes must be made during a project, the successor may use the same logbook. In this case, the logbook cover page will indicate all persons who have made entries and the dates. This may be inappropriate if there are a large number of people involved.
The logbook user is responsible for recording pertinent data into the logbook to satisfy project requirements and for attesting to the accuracy of the entries by dated signature. The logbook user is also responsible for safeguard of the logbook while having custody of it.
Individuals performing specific tasks associated with a field project may keep a separate logbook; however, these logbooks must conform to this procedure and will become a permanent part of the central project file. The Project Manager is responsible for reviewing and signing all field logbooks associated with the project.

2.3 RECORDING FIELD ACTIVITIES

The field team provides a permanent record of daily activities, observations, and measurements through the use of a field logbook. All logbook entries will be made in indelible black or blue ink. No erasures are permitted. If an incorrect entry is made, the data will be crossed out with a single line and initialed and dated by the originator. Entries can be organized into easily understood tables if possible.
All logbook pages will be signed and dated at the bottom. Times will be recorded next to each entry. If a full page is not used during the course of a workday, a diagonal line will be drawn through the unused portion of the page and signed (in this case, it would not be necessary to sign the bottom of the page). If the project is completed and the logbook has not been completely

filled, a diagonal line will be drawn across the first blank page after the last entry, and "no further entries" written before the page is signed and dated.

Daily entries will be made during field activities by, at a minimum, one field team member to provide daily records of all significant events, observations, and measurements during field operations. Notes will start at the beginning of the first blank page and extend through as many pages as necessary. All page numbers will be consecutively numbered as the logbook is filled.

The inside cover page of each logbook will contain the following information:

- Book number
- Project name
- Contract number
- Project number
- Navy Activity/Installation
- Site name
- Start date
- End date
- Person to whom the logbook is assigned
- Agency/Company name
- Agency/Company address
- Agency/Company phone number

The field logbook serves as the primary record of field activities. When possible, the field book should be dedicated to a singular Navy Activity/Installation to facilitate long-term records archiving. Entries shall be made chronologically and in sufficient detail to allow the writer or a knowledgeable reviewer to reconstruct the applicable events. Individual data forms may be generated to provide systematic data collection documentation. Entries on these forms shall meet the same requirements as entries in the logbook and shall be referenced in the applicable logbook entry. Individual data forms shall reference the applicable logbook and page number. At a minimum, names of all samples collected shall be included in the logbook even if recorded elsewhere.

All field descriptions and observations are entered into the logbook, as described in Attachment III-D-1.

Typical information to be entered includes, but is not limited to, the following:

- Date and time of all onsite activities
- Site location and description
- Weather conditions
- Field work documentation
- Descriptions of and rationale for approved deviations from the Work Plan or Field Sampling Plan
- Field instrumentation readings
- Personnel present
- Photograph references

- Sample locations
- Sample identifications, as described in SOP I-A-11, Sample Naming
- Field QC sample information
- Field descriptions, equipment used, and field activities accomplished to reconstruct field operations
- Meeting information
- Daily health and safety meeting notes
- Important times and dates of telephone conversations, correspondence, or deliverables
- Field calculations
- PPE level
- Calibration records
- Subcontractors present
- Equipment decontamination procedures and effectiveness
- Procedures used for containerization of investigative-derived waste

Logbook page numbers shall appear on each page to facilitate identification of photocopies.
If a person's initials are used for identification, or if uncommon acronyms are used, these should be identified on a page at the beginning of the logbook.
At least weekly and preferably daily, the preparer shall photocopy and retain the pages completed during that session for backup. This will prevent loss of a large amount of information if the logbook is lost.
A technical review of each logbook shall be performed by a knowledgeable individual such as the Project Manager.

3.0 DOCUMENTATION

The field logbook shall be retained as a permanent project record. If a particular Task Order requires submittal of photocopies of logbooks, this shall be performed as required.

4.0 REFERENCES

SOP I-A-11, *Sample Naming*

5.0 ATTACHMENTS

Attachment III-D-1 Description of Logbook Entries

Attachment 1
Description of Logbook Entries

Logbook entries shall contain the following information, as applicable, for each activity recorded. Some of these details may be entered on data forms as described previously.

Name of Activity	For example, Asbestos Bulk Sampling, Charcoal Canister Sampling, Aquifer Testing.
Task Team Members and Equipment	Name all members on the field team involved in the specified activity. List equipment used by serial number or other unique identification, including calibration information.
Activity Location	Indicate location of sampling area as specified in the Field Sampling Plan. Record valid Navy Installation/Active and Site, at a minimum.
Weather	Indicate general weather and precipitation conditions.
Level of Personal Protective Equipment	The level of personal protective equipment (PPE), e.g., Level D, should be recorded.
Methods	Indicate method or procedure number employed for the activity.
Sample IDs	Indicate the unique identifier associated with the physical samples. Identify QC samples. Value can be numeric or alphanumeric and must not already exist in the database.
Sample Type and Volume	Indicate the medium, container type, preservative, and the volume for each sample.
Sample Collection Information	Indicate the location of sample, date and time of collection, sample matrix, sample depth interval, sample methods, sample handling, including filtration and preservation, analysis required and packaging and shipping information.
Time and Date	Record the time and date when the activity was performed (e.g., 0830/08/OCT/89). Use the 24-hour clock for recording the time and two digits for recording the day of the month and the year.
Analyses	Indicate the appropriate code for analyses to be performed on each sample, as specified in the Field Sampling Plan.
Field Measurements	Indicate measurements and field instrument readings taken during the activity.
Chain of Custody and Distribution	Indicate chain-of-custody for each sample collected and indicate to whom samples are transferred and the destination.
References	If appropriate, indicate references to other logs or forms, drawings or photographs employed in the activity.

Narrative (including time and location)	Create a factual, chronological record of the team's activities throughout the day, including the time and location of each activity. Include descriptions of any general problems encountered and their resolution. Provide the names and affiliations of non-field team personnel who visit the site, request changes in activity, impact to the work schedule, requested information, or observe team activities. Record any visual or other observations relevant to the activity, the contamination source, or the sample itself. It should be emphasized that logbook entries are for recording data and chronologies of events. The logbook author must include observations and descriptive notations, taking care to be objective and recording no opinions or subjective comments unless appropriate.
Recorded by	Include the signature of the individual responsible for the entries contained in the logbook and referenced forms.
Checked by	Include the signature of the individual who performs the review of the completed entries.

RECORD KEEPING, SAMPLE LABELING, AND CHAIN-OF-CUSTODY PROCEDURES

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to establish standard protocols for all U.S. Naval Facilities Engineering Command Northwest (NAVFAC NW) field personnel and their contractors for use in maintaining field and sampling activity records, writing sample logs, labeling samples, ensuring that proper sample custody procedures are utilized, and completing chain-of-custody/analytical request forms.

2.0 PROCEDURES

Standards for documenting field activities, labeling the samples, documenting sample custody, and completing chain-of-custody and analytical request forms are provided in this procedure. The standards presented in this section shall be followed to ensure that samples collected are maintained for their intended purpose and that the conditions encountered during field activities are documented.

2.1 RECORD KEEPING

The field logbook serves as the primary record of field activities. Entries shall be made chronologically and in sufficient detail to allow the writer or a knowledgeable reviewer to reconstruct each day's events. Field logs such as soil boring logs and ground-water sampling logs will also be used. These procedures are described in SOP III-D, *Logbooks*.

2.2 SAMPLE LABELING

A sample label with adhesive backing shall be affixed to each individual sample container. Clear tape shall be placed over each label (preferably prior to sampling) to prevent the labels from tearing off, falling off, or being smeared, and to prevent loss of information on the label. The following information shall be recorded with a waterproof marker on each label:

- Project name or number (optional)
- Sample ID
- Date and time of collection
- Sampler's initials
- Matrix (optional)
- Sample preservatives (if applicable)
- Analysis to be performed on sample. This shall be identified by the method number or name identified in the subcontract with the laboratory. For water samples, a separate container is typically used for each separate test method, whereas with soil samples, multiple analyses can be performed on the soil obtained from one sample container. In order to avoid lengthy lists on each container and confusion, soil sample containers may not list every analysis to be performed.

These labels may be obtained from the analytical laboratory or printed from a computer file onto adhesive labels. The adhesive glue used on the labels must be such that it does not contaminate the sample.

2.3 CUSTODY PROCEDURES

For samples intended for chemical analysis, sample custody procedures shall be followed through collection, transfer, analysis, and disposal to ensure that the integrity of the samples is maintained. Custody of samples shall be maintained in accordance with EPA chain-of-custody guidelines as prescribed in EPA's *NEIC Policies and Procedures*, National Enforcement Investigations Center, Denver, Colorado, revised May 1986; EPA *RCRA Ground Water Monitoring Technical Enforcement Guidance Document* (TEGD), *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA* (EPA OSWER Directive 9355 3-01), Appendix 2 of the *Technical Guidance Manual for Solid Waste Water Quality Assessment Test (SWAT) Proposals and Reports*, and *Test Methods for Evaluating Solid Waste* (EPA SW-846). A description of sample custody procedures is provided below.

2.3.1 Sample Collection Custody Procedures

According to EPA's *NEIC Policies and Procedures*, a sample is considered to be in custody if:

- It is in one's actual physical possession or view
- It is in one's physical possession and has not been tampered with (i.e., it is under lock or official seal)
- It is retained in a secured area with restricted access
- It is placed in a container and secured with an official seal such that the sample cannot be reached without breaking the seal

Custody seals shall be placed on sample containers immediately after sample collection and on shipping coolers if the cooler is to be removed from the sampler's custody. Custody seals will be placed in such a manner that they must be broken to open the containers or coolers. The custody seals shall be labeled with the following information:

- Sampler's name or initials
- Date and time that the sample/cooler was sealed.

These seals are designed to enable detection of sample tampering. An example of a custody seal is shown in Attachment III-E-1.

Field personnel shall also log individual samples onto carbon copy chain-of-custody forms when a sample is collected. These forms may also serve as the request for analyses. Procedures for completing these forms are discussed in Section 2.4 indicating sample number, matrix, date and time of collection, number of containers, analytical methods to be performed on the sample, and preservatives added (if any). The samplers will also sign the COC form signifying that they were the personnel who collected the samples. The COC form shall accompany the samples from the field to the laboratory. When a cooler is ready for shipment to the analytical laboratory, the person delivering the samples for transport will sign and indicate the date and time on the accompanying COC form. One copy of the COC form will be retained by the sampler and the remaining copies of the COC form shall be placed inside a self-sealing bag and taped to the inside of the cooler. Each cooler must be associated with a unique COC form. Whenever a transfer of custody takes place, both parties shall sign and date the accompanying carbon copy COC forms, and the individual relinquishing the samples shall retain a copy of each form. One exception is when the samples are shipped; the delivery service personnel will not sign or receive a copy because they do not open the coolers. The laboratory shall attach copies of the completed COC forms to the reports containing the results of the analytical tests. An example COC form is provided in Attachment III-E-2. An example of a completed COC form is provided in Attachment III-E-3 and described in Section 2.4.

2.3.2 Laboratory Custody Procedures

The following are custody procedures to be followed by an independent laboratory receiving samples for chemical analysis; the procedures in their Laboratory Quality Assurance Plan (LQAP) must follow these same procedures. A designated sample custodian shall take custody of all samples upon their arrival at the analytical laboratory. The custodian shall inspect all sample labels and COC forms to ensure that the information is consistent, and that each is properly completed. The custodian will also measure the temperature of the samples in the coolers upon arrival. The custodian shall also note the condition of the samples including:

- If the samples show signs of damage or tampering.
- If the containers are broken or leaking.
- If headspace is present in sample vials.
- Proper preservation of samples (made by pH measurement, except VOCs and purgeable TPH). The pH of these samples will be checked by the laboratory analyst, after the sample aliquot has been removed from the vial for analysis.
- If any sample holding times have been exceeded.

All of the above information shall be documented on a sample receipt sheet by the custodian. Any discrepancy or improper preservation shall be noted by the laboratory as an out-of-control event and shall be documented on an out-of-control form with corrective action taken. The out-of-control form shall be signed and dated by the sample control custodian and any other persons responsible for corrective action. An example of an out-of-control form is included as Attachment III-E-4.

The custodian shall then assign a unique laboratory number to each sample and distribute the samples to secured storage areas maintained at 4°C. The unique laboratory number for each sample, contractor sample ID, client name, date and time received, analysis due date, and storage details shall also be manually logged onto a sample receipt record and later entered into the laboratory's computerized data management system. The custodian shall also sign the shipping bill and maintain a copy.

Laboratory personnel shall be responsible for the care and custody of samples from the time of their receipt at the laboratory through their exhaustion or disposal. Samples should be logged in and out on internal laboratory COC forms each time they are removed from storage for extraction or analysis.

2.4 COMPLETING CHAIN-OF-CUSTODY/ANALYTICAL REQUEST FORMS

COC form/analytical request completion procedures are crucial in properly transferring the custody and responsibility of samples from field personnel to the laboratory. This form also is important for accurately and concisely requesting analyses for each sample; it is essentially a release order from the analysis subcontract.

Attachment III-E-2 is an example of a generic COC/analytical request form that may be used by field personnel. Multiple copies may be tailored to each project so that much of the information described below need not be handwritten each time. Attachment III-E-3 is an example of a completed site-specific COC/analytical request form, with box numbers identified and discussed in text below.

Box 1 Project Manager: This name shall be the name that will appear on the report. Do not write the name of the Project Coordinator or point of contact for the project instead of the Project Manager.

Project Name: Write it, as it is to appear on the report.

Project Number: Write it as it is to appear on the report. It shall include the project number, task number, and general ledger section code. The laboratory subcontract number should also be included.

Box 2 Bill to: List the name and address of the person/company to bill only if it is not in the subcontract with the laboratory.

Box 3 Sample Disposal Instructions: These instructions will be stated in the Basic Ordering Agreement (BOA) or each Task Order statement of work with each laboratory.

Shipment Method: State the method of shipment, e.g., hand carry; air courier via FEDEX, AIRBORNE, DHL or equivalent.

Comment: This area shall be used by the field team to communicate observations, potential hazards, or limitations that may have occurred in the field or additional information regarding analysis. For example: a specific metals list, explanation of Mod 8015, Mod 8015 + Kerosene, samples expected to contain high analyte concentrations.

Box 4 Cooler Number: This will be written somewhere on the inside or outside of the cooler and shall be included on the COC. Some laboratories attach this number to the trip blank identification, which helps track VOC samples. If a number is not on the cooler, field personnel shall assign a number, write it on the cooler, and write it on the COC.

QC Level: Enter the reporting/QC requirements, e.g., NAVFAC NW QC Level C, D, or E.

Turnaround time (TAT): TAT for contract work will be determined by a sample delivery group (SDG), which may be formed over a 14-day period, not to exceed 20 samples. Standard turnaround time once the SDG has been completed is 35 calendar days from receipt of the last sample in the SDG. Entering NORMAL or STANDARD in this field will be acceptable. If quicker TAT is required, it shall be in the subcontract with the laboratory and reiterated on each COC to remind the laboratory.

Box 5 Type of containers: The type of container used, e.g., 1-liter glass amber, for a given parameter in that column.

Preservatives: Field personnel must indicate on the COC the correct preservative used for the analysis requested. Indicate the pH of the sample (if tested) in case there are buffering conditions found in the sample matrix.

Box 6 Sample number: Five-character alpha-numeric identifier to be used by the laboratory to identify samples. The use of this identifier is important since the labs are restricted to the number of characters they are able to use. See SOP I-A-11, Sample Naming.

Description (sample identification): This name will be determined by the location and description of the sample, as described in SOP I-A-11, Sample Naming. This sample identification should not be submitted to the laboratory, but should be left blank. If a computer COC version is used, the sample identification can be input but printed with this block black. A cross-referenced list of sample number and sample identification must be maintained separately.

Date Collected: Collection date must be recorded in order to track the holding time of the sample. Note: For trip blanks, record the date it was placed in company with samples.

Time Collected: When collecting samples, record the time the sample is first collected. Use of the 24-hour military clock will avoid a.m. or p.m. designations; e.g., 1815 instead of 6:15 p.m. Record local time; the laboratory is responsible for calculating holding times to local time.

Lab Identification: This is for laboratory use only.

Box 7	Matrix and QC: Identify the matrix: e.g., water, soil, air, tissue, fresh water sediment, marine sediment, or product. If a sample is expected to contain high analyte concentrations, e.g., a tank bottom sludge or distinct product layer, notify the laboratory in the comment section. Mark an "X" for the sample(s) that have extra volume for laboratory QC matrix spike/matrix spike duplicate (MS/MSD) purposes. The sample provided for MS/MSD purposes is usually a field duplicate.
Box 8	Analytical Parameters: Enter the parameter by descriptor and the method number desired. When requesting metals that are modifications of the standard lists, define the list in the comment section. This would not be necessary when requesting standard list metals such as priority pollutant metals (PPM), target compound list from ILM03.0, and Title 22 metals which are groups of metals commonly requested and should not cause any confusion as to what metals are being analyzed. Whenever possible, list the parameters as they appear in the laboratory subcontract to maintain consistency and avoid confusion. In the boxes below the analytical parameter, indicate the number of containers collected for each parameter by marking an "X". If more than one container is used for a sample, write a number in the desired box to indicate a request for analysis and to indicate the number of containers sent for that analysis.
Box 9	Sampler's Signature: The person who collected samples must sign here. Relinquished By: This space shall contain the signature of the person who turned over the custody of the samples to a second party other than an express mail carrier such as FEDEX, DHL or Air Borne Express. Received By: Typically, this is a written signature by a representative of the receiving laboratory, or a field crewmember who delivered the samples in person from the field to the laboratory. A courier such as FedEx or DHL does not sign because they do not open the coolers. It must also be used by the prime contracting laboratory when samples are sent to a subcontractor. Relinquished By: In the case of subcontracting, the primary laboratory will sign the Relinquished By space and fill out an additional COC to accompany the samples being subcontracted. Received By (Laboratory): This space is for the final destination (e.g., at a subcontracted laboratory).
Box 10	Lab Number and Questions: This box is to be filled in by the laboratory only.
Box 11	Control Number: This number is the "COC" followed by the first sample number in a cooler, or contained on a COC. This control number must be unique and never used twice. Record the date the COC is completed. It should be the same date the samples are collected.
Box 12	Total No. of Containers/row: Sum the number of containers in that row.
Box 13	Total No. of Containers/column: Sum the number of containers in that column.

Because COC forms contain different formats based upon who produced the form, not all of the information listed in items 1 to 13 may be recorded. However, as much of this information as possible shall be included.

COC forms tailored to each Task Order can be drafted and printed onto multi-ply forms. This eliminates the need to rewrite the analytical methods column headers each time. It also eliminates the need to write the project manager, name, and number; QC Level; TAT; and the same general comments each time.

Complete one COC form per cooler. Whenever possible, reduce the number of trip blanks by placing all samples to be analyzed for VOA, gasoline, and BTEX compounds into one cooler.

Complete all sections and be sure to sign and date the COC form. One copy of the COC form must remain with the field personnel.

3.0 DOCUMENTATION

The COC/analytical request form shall be faxed daily, if possible, to the Task Order Laboratory Coordinator for accuracy verification. Following the completion of sampling activities, the sample logbook and COC forms will be transmitted to the Project Manager for storage in project files. The Project Manager shall review COC forms on a monthly basis at a minimum. The data validators shall also receive a copy. Along with the data delivered, the original COC/analytical request form shall be submitted by the laboratory. Any changes to the analytical requests that are required shall be made in writing to the laboratory. A copy of this written change shall be sent to the data validators and placed in the project files. The reason for the change shall be included in the project files so that recurring problems can be easily identified.

4.0 REFERENCES

SOP I-A-11, *Sample Naming*
SOP III-D, *Logbooks*
State of California Water Resources Control Board. 1988. Technical Guidance Manual for Solid Waste Water Quality Assessment Test (SWAT) Proposals and Reports.
USEPA. 1986. EPA NEIC Policies and Procedures, National Enforcement Investigations Center, Denver, Colorado.
USEPA. 1988. Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA (EPA OSWER Directive 9355 3-01).
USEPA. 1992. RCRA Ground Water Monitoring Technical Enforcement Guidance Document (TEGD).
USEPA. 1995 and as updated. Test Methods for Evaluating Solid Waste (SW-846), Third edition.

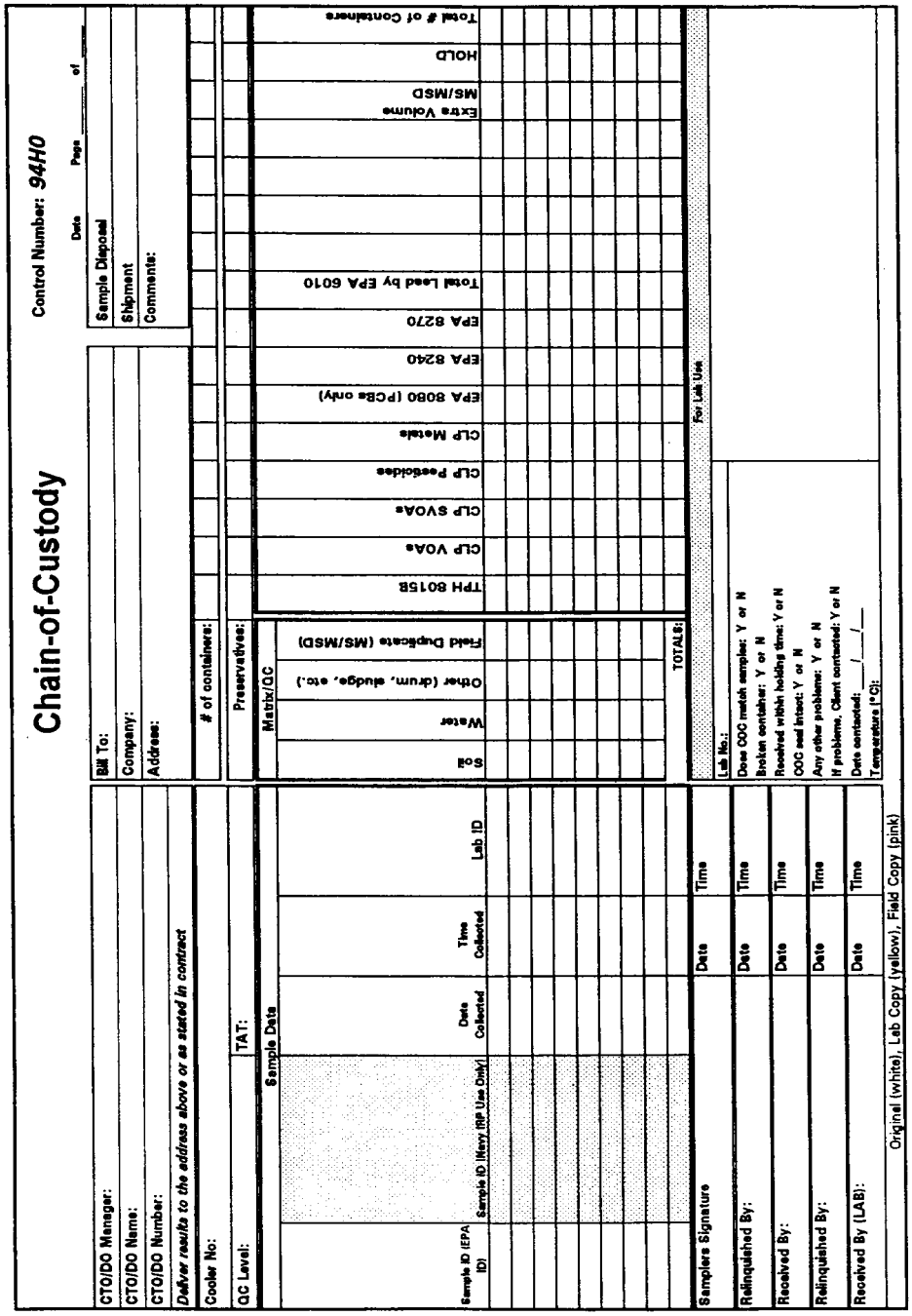
5.0 ATTACHMENTS

Attachment III-E-1 Chain-of-Custody Seal
Attachment III-E-2 Generic Chain-of-Custody/Analytical Request Form
Attachment III-E-3 Sample Completed Chain-of-Custody/Analytical Request Form
Attachment III-E-4 Sample Out-of-Control Form

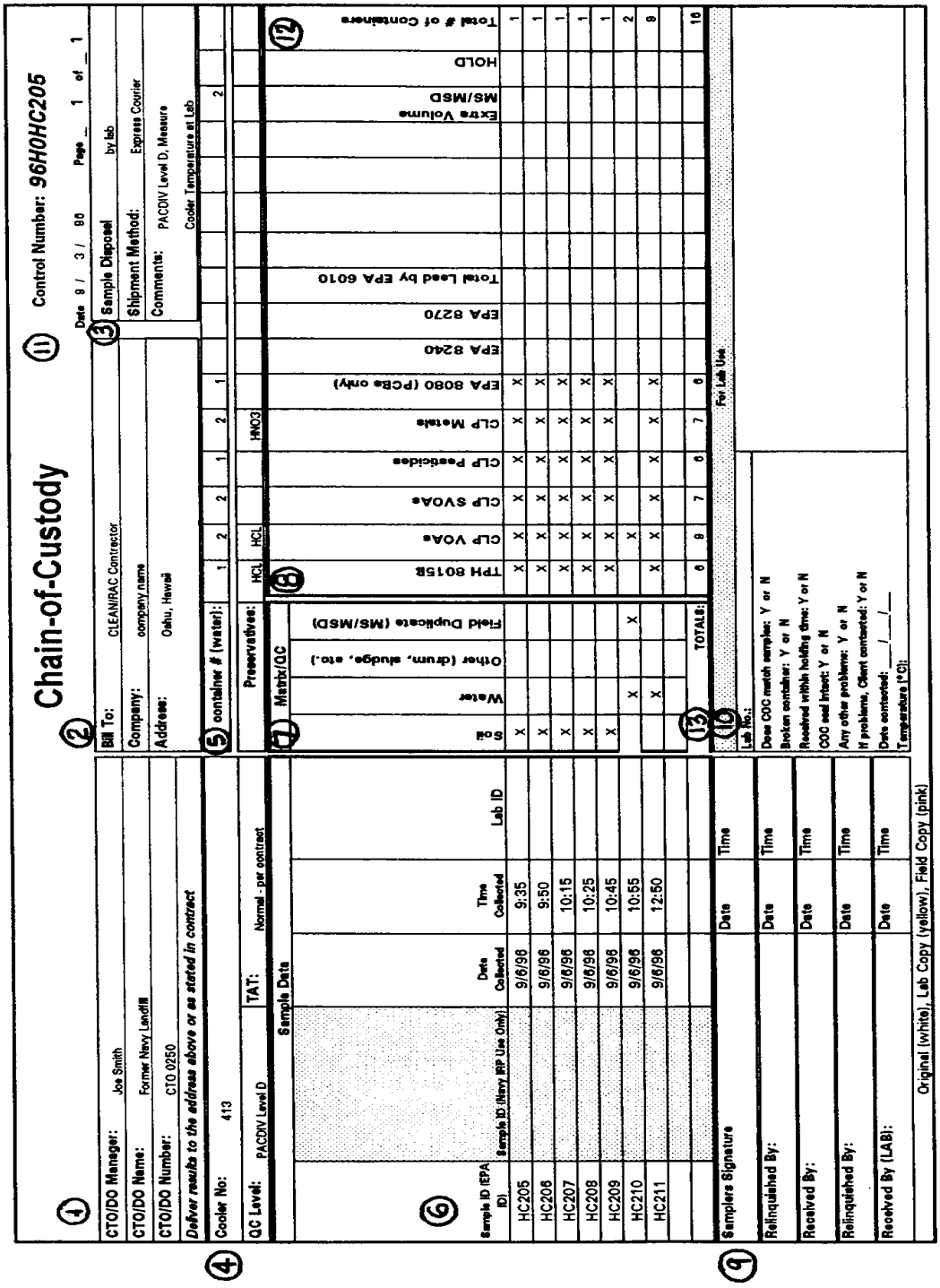
Attachment III-E-1
Chain-of-Custody Seal

<table>
<tr><td rowspan="3">[LABORATORY]</td><td>SAMPLE NO.</td><td>DATE</td><td>SEAL BROKEN BY</td></tr>
<tr><td colspan="2">SIGNATURE</td><td>DATE</td></tr>
<tr><td colspan="3">PRINT NAME AND TITLE (Inspector, Analyst or Technician</td></tr>
</table>

Attachment III-E-2
Generic Chain-of-Custody/Analytical Request Form



Attachment III-E-3
Sample Completed Chain-Of-Custody/
Analytical Request Form



Attachment III-E-4
Sample Out-Of-Control Form

OUT OF CONTROL FORM	Status	Date	Initial
	Noted OOC		
	Submit for CA*		
	Resubmit for CA*		
	Completed		

Date Recognized:	By:
Dated Occurred:	Matrix
Parameter (Test Code):	Method:
Analyst:	Supervisor:

Samples Affected (List by Accession AND Sample No.)

1. Type of Event (Check all that apply)		2. Corrective Action (CA)* (Check all that apply)	
	Calibration Corr. Coefficient <0.995		Repeat calibration
	%RSD>20%		Made new standards
	Blank >MDL		Reran analysis
	Does not meet criteria:		Sample(s) redigested and rerun
	Spike		Sample(s) reextracted and rerun
	Duplicate		Recalculated
	LCS		Cleaned system
	Calibration Verification		Ran standard additions
	Standard Additions		Notified
	MS/MSD		Other (please explain)
	BS/BSD		
	Surrogate Recovery		
	Calculations Error		
	Holding Times Missed		
	Other (Please explain	Comments:	

3. Results of Corrective Action

- ☐ Return to Control (indicated with)
- ☐ Corrective Actions Not Successful - DATA IS TO BE FLAGGED with _____.

Analyst:	Date:
Supervisor:	Date:
QA Department:	Date:

SAMPLE HANDLING, STORAGE, AND SHIPPING

1.0 PURPOSE

This standard operating procedure (SOP) sets forth the methods for use by U.S. Naval Facilities Engineering Command Northwest (NAVFAC NW) field personnel and their contractors engaged in handling, storing, and transporting water, soil and/or sediment samples.

2.0 PROCEDURE

2.1 HANDLING AND STORAGE

Immediately following collection, all samples will be labeled according to the procedures in SOP III-E, *Record Keeping, Sample Labeling, and Chain-of-Custody Procedures*. The lids of the containers shall not be sealed with duct tape, but may be covered with custody seals or placed directly into sealed plastic bags. The sample containers shall be placed in an insulated cooler with frozen gel packs (such as "blue ice") or ice in double, self-sealing bags. Samples should occupy the lower portion of the cooler, while the ice should occupy the upper portion. An absorbent material (e.g., proper absorbent cloth material) may be placed on the bottom of the cooler to contain liquids in case of spillage. All empty space between sample containers shall be filled with bubble wrap, Styrofoam "peanuts," or other appropriate material. Prior to shipping, glass sample containers should be wrapped on the sides, tops, and bottoms with bubble wrap or other appropriate padding and/or surrounded by packing material to prevent breakage during transport. Prior to shipment, the ice or cold packs in the coolers may require replacement to maintain samples as close to 4°C as possible during transport of the samples to the analytical laboratory. Samples shall be shipped as soon as possible to allow the laboratory to meet holding times for analyses. The procedures for maintaining sample temperatures at 4°C, pertains to all water, soil, and sediment field samples.

2.2 SHIPPING

All appropriate U.S. Department of Transportation (DOT) regulations (e.g., 49 Code of Federal Regulations (CFR), Parts 171-179) shall be followed in shipment of air, soil, water, and other samples.

2.2.1 Hazardous Materials Shipment

Field personnel must state whether any sample is suspected to be a hazardous material. A sample should be assumed to be hazardous unless enough evidence exists to indicate it is nonhazardous. If not suspected to be hazardous, shipments may be made as described in the Section 2.2.2 for non-hazardous materials. If hazardous, the procedures summarized below must be followed. Any substance or material that is capable of posing an unreasonable risk to life, health, or property when transported is classified as hazardous. Hazardous materials identification should be performed by checking the list of dangerous goods for that particular mode of transportation. If not on that list, materials can be classified by checking the Hazardous Materials Table (49 CFR

172.102 including Appendix A) or by determining if the material meets the definition of any hazard class or division (49 CFR Part 173), as listed in Attachment III-G-2.

All persons offering for shipment any hazardous material must be properly trained in the appropriate regulations, as required by HM-126F, Training for Safe Transportation of Hazardous Materials. The training covers loading, unloading, handling, storing, and transporting of hazardous materials, as well as emergency preparedness in the case of accidents. Carriers such as commercial couriers must also be trained.

When shipping hazardous materials, including bulk chemicals or samples suspected of being hazardous, the proper shipping papers (49 CFR 172 Subpart C), package marking (49 CFR 172 Subpart D), labeling (49 CFR 172 Subpart E), placarding (49 CFR 172 Subpart F, generally for carriers), and packaging must be used. Attachment III-G-1 shows an example of proper package markings. A copy of 49 CFR should be referred to each time a hazardous material or potentially hazardous samples are shipped.

According to Section 2.7 of the International Air Transport Association (IATA) Dangerous Goods Regulations publication, very small quantities of certain dangerous goods may be transported without certain marking and documentation requirements as described in 49 CFR Part 172. However, other labeling and packing requirements must still be followed. Attachment III-G-2 shows the volume or weight for different classes of substances. A "Dangerous Goods in Excepted Quantities" label must be completed and attached to the associated shipping cooler (Attachment III-G-3). Certain dangerous goods are not allowed on certain airlines in any quantity.

As stated in item 4 of Attachment III-G-4, the Hazardous Materials Regulations do not apply to hydrochloric acid (HCl), nitric acid (HNO_3), sulfuric acid (H_2SO_4), and sodium hydroxide (NaOH) added to water samples if their pH or percentages by weight criteria are met. These samples may be shipped as non-hazardous materials as discussed below.

2.2.2 Nonhazardous Materials Shipment

If the samples are suspected to be nonhazardous, based on previous site sample results, field screening results, or visual observations, if applicable, then samples may be shipped as nonhazardous.

When a cooler is ready for shipment to the laboratory, copies of the chain-of-custody form shall be placed inside a sealed plastic bag and placed inside of an insulated cooler. The coolers will then be sealed with waterproof tape and labeled "Fragile," "This-End-Up" (or directional arrows pointing up), or other appropriate notices. Custody seals will be placed on the coolers as discussed in SOP III-E, *Record Keeping, Sample Labeling, and Chain-of-Custody Procedures*.

2.2.3 Shipments from Outside the Continental United States

Shipment of sample coolers to the U.S. from locations outside the continental U.S. is controlled by the USDA and is subject to their inspection and regulation. Documentation is required to prove that the analytical laboratory receiving samples is certified. The laboratory must have certification by USDA to receive and properly dispose of soil; this is called a "USDA Soil Import Permit." In addition, all sample coolers must be inspected by a USDA representative, affixed with a label indicating that the coolers contain environmental samples, and shipping forms stamped by the USDA inspector prior to shipment. In addition, samples shipped from U.S. territorial possessions or foreign countries, must be cleared by the U.S. Customs Service upon entry into the United States. As long as the commercial invoice is properly completed (see

below), shipments typically pass through U.S. Customs without the need to open coolers for inspection.

Completion and use of proper paperwork will, in most cases, minimize or eliminate the need of the USDA and U.S. Customs to inspect the contents. Attachment III-G-5 shows an example of how paperwork may be placed on the outside of coolers for nonhazardous materials. For hazardous materials, refer to Section 2.2.1.

In summary, the paperwork listed below should be taped to the outside of the coolers to assist sample shipments. If a shipment is made up of multiple pieces (e.g., more than one cooler), the paperwork need be attached only to one cooler, provided that the courier agrees. All other coolers in the shipment need only be taped and have address and chain-of-custody seals affixed.

1. **Courier Shipping Form & Commercial Invoice** - See Attachments III-G-6, III-G-7, and III-G-8 for examples of the information to be included on these forms. Both forms should be placed inside a clear plastic adhesive-backed pouch, which adheres to the package (typically supplied by the courier) and placed on the cooler lid as shown in Attachment 5.
2. **Soil Import Permit and USDA Letter** (soil only) - See Attachments III-G-9 and III-G-10 for examples. The laboratory shall supply these documents prior to mobilization. The USDA in Hawaii often does stop shipments of soil without these documents. The 2" x 2" USDA label (described below), the USDA letter, and soil impact permit should be stapled together and placed inside a clear plastic pouch. Clear plastic and adhesive-backed pouches are typically supplied by the mailing courier.
3. The analytical laboratory should supply the Soil Import Permit. Although original labels are preferred, copies of this label, which are cut out to the 2" x 2" dimensions, are acceptable. Placing one label (as shown in Attachment III-G-5) covered with clear packing tape and one stapled to the actual permit is suggested.
4. The USDA does not control water samples, thus the requirements for soils listed above do not apply.
5. **Custody Seals**. Task Order personnel must sign and date custody seals. At least two seals should be placed in such a manner that they stick to both the cooler lid and body. The seals shall be placed so the cooler/container cannot be opened without breaking the seal. The custody seals are then covered with clear packing tape. This prevents the seal from coming loose and enables detection of tampering.
6. **Address Label**. A label stating the destination (the sending and laboratory, company, or location address) should be affixed to each cooler. The label should also include both telephone numbers.
7. **Special Requirements for Hazardous Materials** - see Section 2.2.1.

Upon receipt of sample coolers at the laboratory, the sample custodian shall inspect the sample containers as discussed in SOP III-E, *Record Keeping, Sample Labeling, and Chain-of-Custody Procedures*. The samples shall then be immediately extracted and/or analyzed, or stored in a refrigerated storage area until they are removed for extraction and/or analysis. Whenever the samples are not being extracted or analyzed, they shall be returned to refrigerated storage.

3.0 DOCUMENTATION

Records shall be maintained as required by implementing these procedures.

4.0 REFERENCES

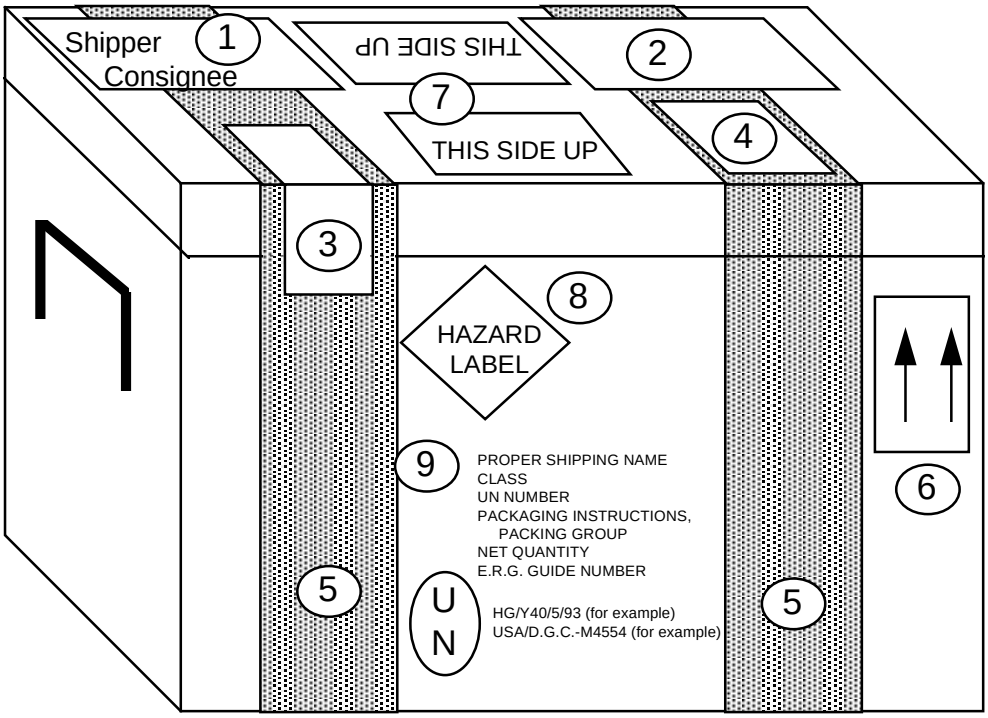
HM-126F, Training for Safe Transportation of Hazardous Materials

SOP III-E, Record Keeping, Sample Labeling, and Chain-of-Custody Procedures

5.0 ATTACHMENTS

Attachment III-G-1 Example Package Marking
Attachment III-G-2 Packing Groups
Attachment III-G-3 Label for Dangerous Goods in Excepted Quantities
Attachment III-G-4 SW-846 Preservative Exception
Attachment III-G-5 Sample Cooler Marking Figure
Attachment III-G-6 Example Courier Form
Attachment III-G-7 Commercial Invoice - Soil
Attachment III-G-8 Commercial Invoice - Water
Attachment III-G-9 Soil Import Permit
Attachment III-G-10 Soil Samples Restricted Entry Labels

Attachment III-G-1
Example Hazardous Material Package Marking



1. **AIR BILL/COMMERCIAL INVOICE**
2. **USDA PERMIT (Letter to Laboratory from USDA)**
3. **CUSTODY SEAL**
4. **USDA 2" X 2" SOIL IMPORT PERMIT**
5. **WATERPROOF STRAPPING TAPE**
6. **DIRECTION ARROWS STICKER - TWO REQUIRED**
7. **THIS SIDE UP STICKERS**
8. **HAZARD LABEL**
9. **HAZARDOUS MATERIAL INFORMATION**
10. **PACKAGE SPECIFICATIONS**

Attachment III-G-2
Packing Groups

Packing Group of the Substance		Packing Group I		Packing Group II		Packing Group III	
CLASS or DIVISION of PRIMARY or SUBSIDIARY RISK		Packagings		Packagings		Packagings	
		Inner	Outer	Inner	Outer	Inner	Outer
1:	Explosives	---------- Forbidden (Note A) ----------					
2.1:	Flammable Gas	---------- Forbidden (Note B) ----------					
2.2:	Non-Flammable, non-toxic gas	---------- See Notes A and B ----------					
2.3:	Toxic gas	---------- Forbidden (Note A) ----------					
3.	Flammable liquid	30 mL	300 mL	30 mL	500 mL	30 mL	1 L
4.1	Self-reactive substances	Forbidden		Forbidden		Forbidden	
4.1:	Other flammable solids	Forbidden		30 g	500 g	30 g	1 kg
4.2:	Pyrophoric substances	Forbidden		Not Applicable		Not Applicable	
4.2	Spontaneously combustible substances	Not Applicable		30 g	500 g	30 g	1 kg
4.3:	Water reactive substances	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
5.1:	Oxidizers	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
5.2:	Organic peroxides (Note C)	See Note A		30 g or 30 mL	500 g or 250 mL	Not Applicable	
6.1:	Poisons - Inhalation toxicity	Forbidden		1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.1:	Poisons - oral toxicity	1 g or 1 mL	300 g or 300 mL	1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.1:	Poisons - dermal toxicity	1 g or 1 mL	300 g or 300 mL	1 g or 1 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
6.2:	Infectious substances	---------- Forbidden (Note A) ----------					
7:	Radioactive material (Note D)	---------- Forbidden (Note A) ----------					
8:	Corrosive materials	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L
9:	Magnetized materials	---------- Forbidden (Note A) ----------					
9:	Other miscellaneous materials (Note E)	Forbidden		30 g or 30 mL	500 g or 500 mL	30 g or 30 mL	1 kg or 1 L

Note A: Packing groups are not used for this class or division.

Note B: For inner packagings, the quantity contained in receptacle with a water capacity of 30 mL. For outer packagings, the sum of the water capacities of all the inner packagings contained must not exceed 1 L.

Note C: Applies only to Organic Peroxides when contained in a chemical kit, first aid kit or polyester resin kit.

Note D: See 6.1.4.1, 6.1.4.2 and 6.2.1.1 through 6.2.1.7, radioactive material in excepted packages.

Note E: For substances in Class 9 for which no packing group is indicated in the List of Dangerous Goods, Packing Group II quantities must be used.

Attachment III-G-3
Label For Dangerous Goods In Excepted Quantities

DANGEROUS GOODS IN EXCEPTED QUANTITIES

This package contains dangerous goods in excepted small quantities and is in all respects in compliance with the applicable international and national government regulations and the IATA Dangerous Goods Regulations.

_____
Signature of Shipper

_____ _____
Title Date

_____

_____
Name and address of Shipper

This package contains substance(s) in Class(es)
(check applicable box(es))

Class:	2	3	4	5	6	8	9
	o	o	o	o	o	o	o

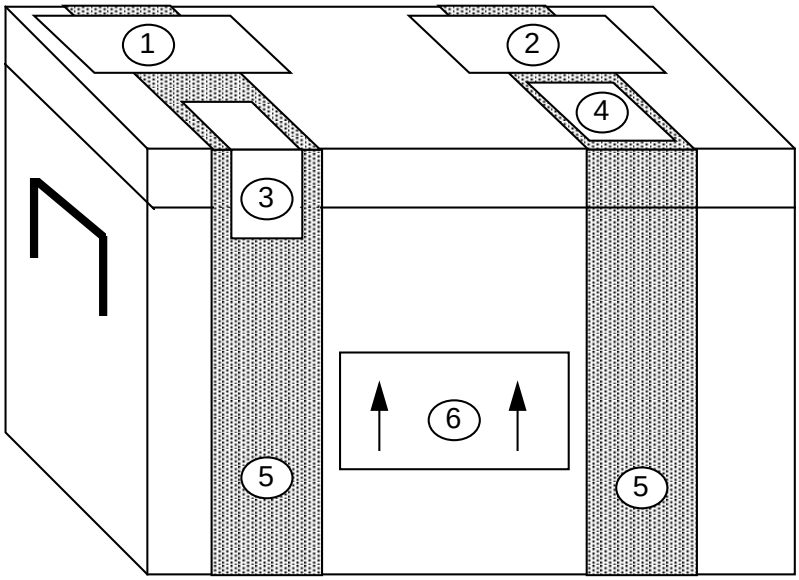
and the applicable UN Numbers are:

ATTACHMENT III-G-4
Preservative Exception

Measurement	Vol. Req. (mL)	Container[2]	Preservative [3,4]	Holding Time[5]
MBAS	50[2]	P,G	Cool, 4ºC	48 Hours
NTA	0[5]	P,G	Cool, 4ºC	24 Hours

1. More specific instructions for preservation and sampling are found with each procedure as detailed in this manual. A general discussion on sampling water and industrial wastewater may be found in ASTM, Part 31, p. 72-82 (1976) Method D-3370.
2. Plastic (P) or Glass (G). For metals, polyethylene with a polypropylene cap (no liner) is preferred.
3. Sample preservation should be performed immediately upon sample collection. For composite samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then samples may be preserved by maintaining at 4ºC until compositing and sample splitting is completed.
4. When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. for the preservation requirements of Table 1, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentration of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or grater); Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).
5. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of sample under study are stable for the longer time, and has received a variance from the Regional Administrator. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample stability.
6. Should only be used in the presence of residual chlorine.

Attachment III-G-5
Non-Hazardous Material Cooler Marking Figure For Shipment From Outside the Continental United States



1. **AIR BILL/COMMERCIAL INVOICE**
2. **USDA PERMIT (Letter to Laboratory from USDA)**
3. **CUSTODY SEAL**
4. **USDA 2" X 2" SOIL IMPORT PERMIT**
5. **WATERPROOF STRAPPING TAPE**
6. **DIRECTION ARROWS STICKER - TWO REQUIRED**

Attachment III-G-6
Example Courier Form

FedEx. USA Airbill FedEx Tracking Number 801704855619 0200 Form I.D. No. SPL11 Sender's Copy

1 From (please print and press hard)

Date _____ Sender's FedEx Account Number Account Number

Sender's Name Joe Smith Phone (808) 545-2462

Company OGDEN ENVIRONMENTAL/CRC ACCT

Address 680 IWILEI RD STE 660 Dept./Floor/Suite/Room

City HONOLULU State HI ZIP 96817

2 Your Internal Billing Reference Information (Optional) (First 24 characters will appear on invoice)

3 To (please print and press hard)

Recipient's Name Sample Receipt Phone ( ) Lab Phone #

Company Lab Name

Address Lab Address (We Cannot Deliver to P.O. Boxes or P.O. ZIP Codes) Dept./Floor/Suite/Room

Check here if residence (Extra charge applies for FedEx Express Saver)

(To "HOLD" at FedEx location, print FedEx address here)

City State ZIP

For HOLD at FedEx Location check here
Hold Weekday (Not available with FedEx First Overnight) Hold Saturday (Not available at all locations) (Available for FedEx Priority Overnight and FedEx 2Day only)

For Saturday Delivery check here (Extra Charge. Not available to all locations) (Available for FedEx Priority Overnight and FedEx 2Day only)

4a Express Package Service Packages under 150 lbs. Delivery commitment may be later in some areas.
FedEx Priority Overnight (Next business morning) FedEx Standard Overnight (Next business afternoon) FedEx 2Day* (Second business day) FedEx Express Saver* (Third business day) FedEx First Overnight (Earliest next business morning delivery to select locations) (Higher rates apply)
* FedEx Letter Rate not available. Minimum charge: One pound rate.

4b Express Freight Service Packages over 150 lbs. Delivery commitment may be later in some areas.
FedEx Overnight Freight (Next business day) FedEx 2Day Freight (Second business day) FedEx Express Saver Freight (Up to 3 business days)
(Call for delivery schedule. See back for detailed descriptions of freight services.)

5 Packaging FedEx Letter FedEx Pak FedEx Box FedEx Tube Other Pkg. Declared value limit $500

6 Special Handling
Does this shipment contain dangerous goods? Yes (As per attached Shipper's Declaration) Yes (Shipper's Declaration not required)
Dry Ice (Dry Ice, 9, UN 1845 III) x kg. 904 (Dangerous Goods Shipper's Declaration not required)
CA Cargo Aircraft Only

7 Payment
Bill to: Sender (Account no. in section 1 will be billed) Recipient Third Party Credit Card Cash/Check (Enter FedEx account no. or Credit Card no. below)
FedEx Account No.
Credit Card No. Exp. Date

Total Packages	Total Weight	Total Declared Value*	Total Charges
		$ .00	$

* When declaring a value higher than $100 per shipment, you pay an additional charge. See SERVICE CONDITIONS, DECLARED VALUE, AND LIMIT OF LIABILITY section for further information.

8 Release Signature Sign to authorize delivery without obtaining signature.
Your signature authorizes Federal Express to deliver this shipment without obtaining a signature and agrees to indemnify and hold harmless Federal Express from any resulting claims.

287

Service Conditions, Declared Value, and Limit of Liability – By using this Airbill, you agree to the service conditions in our current Service Guide or U.S. Government Service Guide. Both are available on request. SEE BACK OF SENDER'S COPY OF THIS AIRBILL FOR INFORMATION AND ADDITIONAL TERMS. We will not be responsible for any claim in excess of $100 per package whether the result of loss, damage, or delay, non-delivery, misdelivery, or misinformation, unless you declare a higher value, pay an additional charge, and document your actual loss in a timely manner. Your right to recover from us for any loss includes intrinsic value of the package, loss of sales, interest, profit, attorney's fees, costs, and other forms of damage, whether direct, incidental, consequential, or special, and is limited to the greater of $100 or the declared value but cannot exceed actual documented loss. The maximum declared value for any FedEx Letter and FedEx Pak is $500. Federal Express may, upon your request, and with some limitations, refund all transportation charges paid. See the FedEx Service Guide for further details.

Questions? Call 1·800·Go·FedEx (800)463-3339 The World On Time

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RETAIN THIS COPY FOR YOUR RECORDS

Attachment III-G-7
Commercial Invoice - Soil

DATE OF EXPORTATION 1/1/94	EXPORT REFERENCES (i.e., order no., invoice no., etc.) <CTO #>
SHIPPER/EXPORTER (complete name and address) Joe Smith Ogden c/o <hotel name> <hotel address>	CONSIGNEE Sample Receipt <Lab Name> <Lab Address>
COUNTRY OF EXPORT Guam, USA	IMPORTER - IF OTHER THAN CONSIGNEE
COUNTRY OF ORIGIN OF GOODS Guam, USA	
COUNTRY OF ULTIMATE DESTINATION USA	

INTERNATIONAL AIR WAYBILL NO. _____ (NOTE: All shipments must be accompanied by a Federal Express International Air Waybill)

MARKS/ NOS	NO. OF PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	QTY	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
	3	coolers	Soil samples for laboratory analysis only				$1.00	$3.00

TOTAL NO. OF PKGS.	TOTAL WEIGHT	TOTAL INVOICE VALUE
3		$3.00

Check one
☐ F.O.B.
☐ C&F
☐ C.I.F.

THESE COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN.

DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED.

I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT

SIGNATURE OF SHIPPER/EXPORTER (Type name and title and sign)

Joe Smith, Ogden	Joe Smith	1/1/94
Name/Title	Signature	Date

ATTACHMENT III-G-8
Commercial Invoice - Water

DATE OF EXPORTATION 1/1/94	EXPORT REFERENCES (i.e., order no., invoice no., etc.) <CTO #>
SHIPPER/EXPORTER (complete name and address) Joe Smith Ogden c/o <hotel name> <hotel address>	CONSIGNEE Sample Receipt <Lab Name> <Lab Address>
COUNTRY OF EXPORT Guam, USA	IMPORTER - IF OTHER THAN CONSIGNEE
COUNTRY OF ORIGIN OF GOODS Guam, USA	
COUNTRY OF ULTIMATE DESTINATION USA	

INTERNATIONAL AIR WAYBILL NO. _____ (NOTE: All shipments must be accompanied by a Federal Express International Air Waybill)

MARKS/ NOS	NO. OF PKGS	TYPE OF PACKAGING	FULL DESCRIPTION OF GOODS	QTY	UNIT OF MEASURE	WEIGHT	UNIT VALUE	TOTAL VALUE
	3	coolers	Water samples for laboratory analysis only				$1.00	$3.00
	TOTAL NO. OF PKGS.					TOTAL WEIGHT		TOTAL INVOICE VALUE
	3							$3.00
								Check one ☐ F.O.B. ☐ C&F ☐ C.I.F.

THESE COMMODITIES ARE LICENSED FOR THE ULTIMATE DESTINATION SHOWN.

DIVERSION CONTRARY TO UNITED STATES LAW IS PROHIBITED.

I DECLARE ALL THE INFORMATION CONTAINED IN THIS INVOICE TO BE TRUE AND CORRECT

SIGNATURE OF SHIPPER/EXPORTER (Type name and title and sign)

Joe Smith, Ogden	Joe Smith	1/1/94
Name/Title	Signature	Date

Attachment III-G-9
Soil Import Permit

UNITED STATES DEPARTMENT OF AGRICULTURE
ANIMAL AND PLANT HEALTH INSPECTION SERVICE
PLANT PROTECTION AND QUARANTINE PROGRAMS

COMPLIANCE AGREEMENT

1. NAME AND MAILING ADDRESS OF PERSON OR FIRM	2. LOCATION
Ogden Environmental & Energy Service Co. 680 Iwilei Road, Suite 660 Honolulu, HI 96817	680 Iwilei Road, Suite 660 Honolulu, HI 96817 Telephone: 545-2462 Fax: 528-5379

3. REGULATED ARTICLE(S)
Foreign soil samples destined to approved laboratories in the Continental United States transiting through Honolulu International Airport and military facilities on Oahu, Hawaii.

4. APPLICABLE FEDERAL QUARANTINE(S) OR REGULATIONS

7 CFR 330.300

6. I/We agree to the following:

See the attached Addendum, Foreign Soil Samples Destined To Approved Laboratories In The Continental United States Transiting Through Honolulu International Airport And Military Facilities On Oahu, Hawaii

THIS COMPLIANCE AGREEMENT IS VALID FOR 2 YEARS FROM THE DATE OF ISSUANCE.
For renewal, call our office at 861-8446 or Fax 861-8450.

EXPIRATION DATE: SEPTEMBER 30, 2000

7. SIGNATURE	8. TITLE	9. DATE SIGNED
Betsy D. Disparagle	Air & HAZARDOUS WASTE Group MANAGER	9/9/98

The affixing of the signatures below will validate this agreement which shall remain in effect until canceled, but may be revised as necessary or revoked for noncompliance.

10. AGREEMENT NO.
OAHU-ST-002

11. DATE OF AGREEMENT
September 2, 1998

12. PPQ OFFICIAL *(Name and Title)*	13. ADDRESS
Michael M. Jodoi, Supervisor, Satellite Operations	USDA, APHIS, PPQ 3375 Koapaka Street, Suite G330 Honolulu, HI 96819
14. SIGNATURE	
15. STATE AGENCY OFFICIAL *(Name and Title)*	16. ADDRESS
N/A	N/A
17. SIGNATURE	
N/A	

PPQ FORM 519
AUG. 1977

REPLACES PPQ 274, 519, 560, AND AQI 83, WHICH ARE OBSOLETE

Soil - Foreign\Foreign Soil - Transit Comp Agree Form 519.frp

Attachment III-G-10
Soil Samples Restricted Entry Labels

U.S. DEPARTMENT OF AGRICULTURE

ANIMAL AND PLANT HEALTH INSPECTION SERVICE

PLANT PROTECTION AND QUARANTINE

HYATTSVILLE, MARYLAND 20782

soil samples

restricted entry

The material contained in this package is imported under authority of the Federal Plant Pest Act of May 23, 1957.

For release without treatment if addressee is currently listed as approved by Plant Protection and Quarantine.

PPQ FORM 550 (JAN 83) Edition of 12/77 may be used

U.S. DEPARTMENT OF AGRICULTURE

ANIMAL AND PLANT HEALTH INSPECTION SERVICE

PLANT PROTECTION AND QUARANTINE

HYATTSVILLE, MARYLAND 20782

soil samples

restricted entry

The material contained in this package is imported under authority of the Federal Plant Pest Act of May 23, 1957.

For release without treatment if addressee is currently listed as approved by Plant Protection and Quarantine.

PPQ FORM 550 (JAN 83) Edition of 12/77 may be used

U.S. DEPARTMENT OF AGRICULTURE

ANIMAL AND PLANT HEALTH INSPECTION SERVICE

PLANT PROTECTION AND QUARANTINE

HYATTSVILLE, MARYLAND 20782

soil samples

restricted entry

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Federal Plant Pest Act of May 23, 1957.

For release without treatment if
addressee is currently listed as
approved by Plant Protection and
Quarantine.

PPQ FORM 550 Edition of 12/77 may be used

(JAN 83)

EQUIPMENT DECONTAMINATION

1.0 PURPOSE

The standard operating procedure (SOP) describes general methods of equipment decontamination (decon) for use by U.S. Naval Facilities Engineering Command Northwest (NAVFAC NW) field personnel and their contractors during field sampling activities. Some sites may require additional steps (e.g. nitric rinses for metals, hexane for chlorinated pesticides) to insure equipment is properly deconned. These should be identified and addressed in the Work Plans and/or the Quality Assurance Project Plans (QAPPs)

2.0 PROCEDURES

Decontamination of equipment is necessary to prevent cross-contamination and to maintain the highest integrity possible in collected samples. Planning a decontamination program should include consideration of the following factors:

- The location where the decon procedures will be conducted
- The types of equipment requiring decon
- The frequency of equipment decontamination
- The cleaning technique and types of cleaning solutions appropriate to the contaminants of concern
- The method for containing the residual contaminants and wash water from the deconning process
- The use of a quality control measure to determine the effectiveness of the decontamination procedure (e.g. equipment rinsate samples)

This subsection describes standards for decontamination, including the techniques to be used, frequency of decontamination, cleaning solutions, and effectiveness.

2.1 DECONTAMINATION AREA

An appropriate location for the decontamination area at a site shall be selected on the basis of the ability to control access to the area, control residual material removed from equipment, the need to store dirty and clean equipment, and the ability to restrict access to the area being investigated. The decontamination area shall be located an adequate distance away and upwind from potential contaminant sources to avoid contamination of clean equipment.

2.2 TYPES OF EQUIPMENT

Examples of drilling equipment that must be deconned includes drill bits, auger sections, split spoon samplers, and hand tools. Decontamination of monitoring well development and ground-water sampling equipment includes submersible pumps, non-disposable bailers, interface probes, water level meters, bladder pumps, airlift pumps, and lysimeters. Other sampling equipment that may require decontamination includes, but is not limited to, hand trowels, hand augers, slide hammer samplers, shovels, stainless steel spoons and bowls, soil sample liners and caps, wipe sampling templates, COLIWASA samplers, and dippers. Equipment with a porous surface, such as rope, cloth hoses, and wooden blocks, cannot be thoroughly decontaminated and should be properly disposed of after one use.

2.3 FREQUENCY OF EQUIPMENT DECONTAMINATION

Down-hole drilling equipment and equipment used in monitoring well development and purging shall be decontaminated prior to initial use and between each borehole or well. However, down hole drilling equipment may require more frequent cleaning to prevent cross-contamination between vertical zones within a single borehole. When drilling through a shallow contaminated zone and installing a surface casing to seal off the contaminated zone, the drilling tools shall be decontaminated prior to drilling deeper. Groundwater sampling should be initiated by sampling ground water from the monitoring well where the least contamination is suspected. This is more important when not using disposable equipment. All groundwater, surface water, and soil sampling devices shall be decontaminated prior to initial use and between collection of each sample to prevent the possible introduction of contaminants into successive samples.

2.4 CLEANING SOLUTIONS AND TECHNIQUES

Decontamination can be accomplished using a variety of techniques and fluids. The preferred method of decontaminating major equipment such as drill bits, augers, drill string, pump drop-pipe, etc., is steam cleaning. Steam cleaning is accomplished using a portable, high-pressure steam cleaner equipped with a pressure hose and fittings. For this method, equipment shall be thoroughly steam washed and rinsed with potable tap water to remove particulates and contaminants.

A rinse decontamination procedure is acceptable for equipment such as bailers, water level meters, new and re-used soil sample liners, and hand tools. The decontamination procedure shall consist of the following: (1) wash with a non-phosphate detergent (Citrinox®, Liquinox®, or other suitable phosphate free detergent) and potable water solution, (2) rinse with potable water, and (3) rinses with deionized or distilled water. Equipment shall be disassembled as much as is practical, prior to cleaning. An initial gross wash scrub down and quick rinse should be completed at the beginning of the process if equipment is heavily soiled. After decontamination, care needs to be taken that the cleaned equipment does not become contaminated. This may require wrapping items in foil or plastic and storing the equipment in a specified “clean” area.

Decontaminating submersible pumps requires additional effort because internal surfaces become contaminated during usage. The pumps shall be decontaminated by circulating fluids through the pump while it is operating. This circulation can be done using a clean 4-inch or greater diameter pipe equipped with an end cap. The pipe shall be filled with enough decon fluid to submerge the pump, the pump placed within the capped pipe, and the pump operated while circulating the fluids within the pipe. The decontamination sequence shall include (1) detergent and potable water, (2) potable water rinse, and (3) deionized or distilled water rinse. The decontamination fluids shall be changed after each cycle. Changing of the fluids may include dumping of the detergent water, mixing detergent in the potable water rinse, using the deionized water as the potable rinse and renewing the distilled/deionized water. All decon water shall be disposed of as outlined in the field work plans.

Decontamination solvent(s) to be used during field activities will be specified in Project Work Plans or QAPPs. If solvents are used, sufficient time must be allowed to insure the solvent has evaporated from the equipment prior to reuse.

Equipment used for measuring field parameters such as pH, temperature, specific conductivity, and turbidity shall be rinsed with deionized or distilled water. New, unused soil sample liners and caps will be cleaned using the three step process, outlined above, to remove any dirt or cutting oils that may be on them prior to use.

2.5 CONTAINMENT OF RESIDUAL CONTAMINANTS AND CLEANING SOLUTIONS

Decontamination program for equipment exposed to potentially hazardous materials requires a provision for catchment and disposal of the contaminated material, cleaning solution, and wash water. This may require setting up a containment area with a system for pumping the water generated decontamination water into proper containers.
Clean equipment should be stored in a separate location to prevent recontamination.
Decontamination fluids contained within the bermed area shall be collected and disposed of as outlined in the field sampling plan.
Containment of fluids from the decontamination of lighter-weight drilling equipment and hand-held sampling devices shall be accomplished using wash buckets or tubs. The decontamination fluids shall be collected and disposed of as outlined in the field sampling plan.

2.6 EFFECTIVENESS OF DECONTAMINATION PROCEDURES

A decontamination program must incorporate quality control measures to determine the effectiveness of cleaning methods. Quality control measures typically include collection of equipment rinsate samples or wipe testing. Equipment rinsates consist of analyte-free water that has been poured over or through the sample collection equipment after its final decontamination rinse. Wipe testing is performed by wiping a cloth over the surface of the equipment after cleaning. Further descriptions of these samples and their required frequency of collection are provided in SOP III-B, *Field QC Samples (Water, Soil).* These quality control measures provide "after-the fact" information that may be useful in determining whether or not cleaning methods were effective in removing the contaminants of concern.

3.0 DOCUMENTATION

The decontamination process shall be recorded in the field logbook.

4.0 REFERENCES

SOP III-B, *Field QC Samples (Water, Soil).*

5.0 ATTACHMENTS

None.

EQUIPMENT CALIBRATION, OPERATION, AND MAINTENANCE

1.0 PURPOSE

This standard operating procedure (SOP) describes the activities and responsibilities of the U.S. Naval Facilities Engineering Command Northwest (NAVFAC NW) personnel pertaining to the operating, calibration, and maintenance of equipment used to collect environmental data. Reliable measurements of data required by the field sampling plan are necessary because the information recorded may be the basis for development of remedial action and responses.

2.0 PROCEDURES

2.1 EQUIPMENT CALIBRATION

All water quality monitoring equipment will be calibrated and adjusted to operate within the manufacturers' specifications. Water quality instruments and equipment that require calibration are to be calibrated to specifications prior to field use. In addition, a one-point calibration check is made at midday and at intervals outlined in the field sampling plan. A final check is conducted at the end of each field day. This is not a recalibration of the meter but a check of the calibration to ensure the continued accuracy of the meter. All calibration information shall be recorded in the project logbook.

Special attention shall be paid to instruments that may be affected by the change in the ambient temperature or humidity. Calibration checks should also be performed when sampling conditions change significantly, a change of sample matrix, and/or readings are unstable or there is a change of parameter measurements that appear unusual.

2.2 EQUIPMENT MAINTENANCE

All field monitoring equipment, field sampling equipment, and accessories are to be maintained in accordance with the manufacturer's recommendations and specifications and/or established field practices. All maintenance will be performed by qualified personnel and documented in the field logbook.

Equipment requiring battery charging shall be charged as recommended by the manufacturer. Backup batteries for meters requiring them shall be included as part of the meters accessories. Care must be taken to protect meters from adverse elements. This may involve placing the meter in a large plastic bag to shield it from the weather.

3.0 DOCUMENTATION

All field equipment calibration, maintenance, and operation information shall be recorded within the field logbook. This is to document that appropriate procedures have been followed and to track the equipment operation. All entries in the field logbook must be written accurately and legibly as outlined in the SOP III-D, *Logbooks*.

Logbook entries shall contain, but are not necessarily limited to, the following:

- Equipment model and serial numbers
- Date and time of calibration or maintenance performed
- Calibration standard used

- Calibration lot number and expiration date if listed on bottle
- Calibration procedure used if there are multiple options
- Calibration and calibration check readings including units used
- Problems and solutions regarding use, calibration or maintenance of the equipment
- And other pertinent information

4.0 REFERENCES

SOP III-D, *Logbooks*

5.0 ATTACHMENTS

None.

ATTACHMENT E

LABORATORY CHAIN OF CUSTODY

Air - Chain of Custody Record & Analytical Service Request

Page _____ of _____

2655 Park Center Drive, Suite A
Simi Valley, California 93065
Phone (805) 526-7161
Fax (805) 526-7270

Requested Turnaround Time in Business Days (Surcharges) please circle
1 Day (100%) 2 Day (75%) 3 Day (50%) 4 Day (35%) 5 Day (25%) 10 Day-Standard

ALS Project No.

Company Name & Address (Reporting Information)

Project Name

Project Number

ALS Contact:

Analysis Method

Project Manager

P.O. # / Billing Information

Phone | Fax

Email Address for Result Reporting

Sampler (Print & Sign)

Comments
e.g. Actual Preservative or specific instructions

Client Sample ID	Laboratory ID Number	Date Collected	Time Collected	Canister ID (Bar code # - AC, SC, etc.)	Flow Controller ID (Bar code #- FC #)	Canister Start Pressure "Hg	Canister End Pressure "Hg/psig	Sample Volume			

Report Tier Levels - please select

Tier I - Results (Default if not specified) _____
Tier II (Results + QC Summaries) _____
Tier III (Results + QC & Calibration Summaries) _____
Tier IV (Data Validation Package) 10% Surcharge _____

EDD required Yes / No
Type: _____ Units:_____

Chain of Custody Seal: (Circle)
INTACT BROKEN ABSENT

Project Requirements (MRLs, QAPP)

Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:

Cooler / Blank Temperature ____°C

ATTACHMENT F

SOIL VAPOR FIELD FORM

SOIL VAPOR FIELD FORM

Well ID _____ ☐ Sub-slab Probe ☐ Nested Probe ☐ Single-depth Probe

Date:		PID (make/model/serial number):	
Project Name:		Landtech (model/serial number):	
Project Number:		Helium Detector (make/model/serial number):	
Site Location:		Manometer (make/model/serial number):	
Field Personnel:			

Surface Type: ☐ Concrete ☐ Asphalt ☐ Grass ☐ Other		Surface Thickness (inches): ☐ Unknown	
Shut-in Testing		Weather:	
Prior to Purge	OK ☐ @	Air Temperature (°C/°F):	
Prior to Sample Collection	OK ☐ @	Atmospheric Pressure (in. Hg):	

Date	Start Time	End Time	Elapsed Time (min)	Bag Volume (L)	Purge Rate (LPM)	CH_4 (%)	CO_2 (%)	O_2 (%)	Total Organic Vapors (ppmv)	Helium beneath Shroud (%)		Helium in Purge Interval (%)
										Min	Max	

Date	Time	Sample ID	Canister ID	Flow Controller #	Vacuum Gauge #	Initial Vacuum	Final Vacuum	Helium beneath Shroud (%)	
								Min	Max

Comments: