

Quality Assurance Project Plan Former Fort Ord, California, Volume I Appendix C, Draft Revision 6 Soil Gas Monitoring at Sites 2 and 12



Prepared for:
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On behalf of:
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USACE Contract No. W91238-19-C-0027
Task No. 6.5



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Report Date: **April 14, 2021**
Report Version: **Draft**

Title and Approval Page (Worksheet #1)

Site Name/Project Name: Sites 2 and 12 (2/12)

Site Location: Former Fort Ord, California

Document Title: Quality Assurance Project Plan, Former Fort Ord, California, Volume I, Appendix C, Preliminary Draft Revision 6, Soil Gas Monitoring at Sites 2 and 12

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Preparation Date: February 26, 2021

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Lead Organization's Technical Lead	Bridget Floyd USACE		
Lead Organization's Project Chemist	Jonathan Whipple USACE		

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Acronyms and Abbreviations

%	percent
µg/m ³	micrograms per cubic meter
ACL	aquifer cleanup level
ADR	Automated Data Review
AEI	Ahtna Environmental, Inc.
AES	Ahtna Engineering Services
Ahtna	Ahtna Global, LLC
Army	U.S. Department of the Army
AS	air sparge
BFB	4-bromofluorobenzene
bgs	below ground surface
BRAC	Base Realignment and Closure
CCA	Comprehensive Certificate of Analysis
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
COC	chemical of concern
COD	coefficient of determination
CPR	cardiopulmonary resuscitation
CQCR	Contractor Quality Control Report
CQM	Construction Quality Management
CCRWQCB	California Regional Water Quality Control Board, Central Coast Region
DL	detection limit
DoD	Department of Defense
DQI	data quality indicator
DQO	data quality objective
DTSC	California Department of Toxic Substances Control
EDD	electronic data deliverable
EDF	electronic data format
ELAP	Environmental Laboratory Accreditation Program
EPA	U.S. Environmental Protection Agency
ESD	Explanation of Significant Differences
FADL	field activity daily logbook
FODIS	Fort Ord Data Integration System
FS	Feasibility Study
GAC	granular activated carbon
GC/MS	gas chromatography/mass spectrometry
HAZWOPER	Hazardous Waste Operations and Emergency Response
HHRA	human health risk assessment
HI	hazard index
ICAL	initial calibration
ICV	initial calibration verification

lbs/day	pounds per day
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LIMS	Laboratory Information Management System
LOD	limit of detection
LOQ	limit of quantitation
MB	method blank
MPC	measurement performance criteria
N/A	not applicable
O&M	operations and maintenance
OSHA	Occupational Safety & Health Administration
PCE	tetrachloroethene
PDF	portable document format
PQO	project quality objective
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
QSM	Quality Systems Manual
RAWP	Remedial Action Work Plan
RI	Remedial Investigation
ROD	Record of Decision
ROI	radius of influence
RPD	relative percent difference
RRT	relative retention time
RSD	relative standard deviation
RT	retention time
SGCL	soil gas cleanup level
SG-SL	soil gas screening level
Sites 2/12	Sites 2 and 12
SOP	standard operating procedure
SSHO	Site Safety and Health Officer
SVE	soil vapor extraction
SVETS	soil vapor extraction and treatment system
SVTU	soil vapor treatment unit
TAT	turnaround time
TCE	trichloroethene
USACE	U.S. Army Corps of Engineers
VOC	volatile organic compound

1.0 Introduction

On behalf of the U.S. Army Corps of Engineers (USACE), Sacramento District, Ahtna Global, LLC (Ahtna) has prepared this *Quality Assurance Project Plan, Former Fort Ord, California, Volume 1, Appendix C, Revision 6, Soil Gas Monitoring at Sites 2 and 12* (QAPP)¹ under Ahtna Contract Number W91238-19-C-0027. This work is being conducted under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or “Superfund”) to address historical releases of chemicals of concern (COCs) at the former Fort Ord. The QAPP was updated and revised to:

- Reflect recent changes in project personnel.
- Clarify decision identification and the analytic approaches for soil gas remediation and groundwater remediation presented in Worksheets #10.
- Update the sample schedules for soil gas probes and soil vapor extraction (SVE) wells based on recent progress in soil gas remedial actions (Table 1).

This QAPP is the governing document for soil gas and soil vapor extraction and treatment system (SVETS) monitoring conducted by Ahtna and associated with Sites 2 and 12 (2/12). This QAPP details the quality assurance (QA) and quality control (QC) procedures to be used during sampling and analytical activities performed so the data generated are accurate, precise, complete, and representative of field conditions of sufficient quality to support project decisions.

¹ This document is Appendix C to the *Quality Assurance Project Plan, Superfund Response Actions, Former Fort Ord, California, Volume I*. Volume I is also the governing document for sampling and analysis of groundwater (Appendix A), soil (Appendix B), and landfill gas (Appendix D). Volume II of the QAPP pertains to the former Fort Ord military munitions response program.

2.0 PROJECT MANAGEMENT AND OBJECTIVES

2.1 QAPP Identifying Information (Worksheet #2)

Title: Quality Assurance Project Plan, Former Fort Ord, California, Volume I, Appendix C, Soil Gas Monitoring at Sites 2 and 12

Revision Number: 6

Revision Date: February 26, 2021

Site name/project name: Soil Gas Monitoring at Sites 2 and 12, Former Fort Ord, California

Site location: Monterey County, California

Site number/code: 2/12

Operable Unit: Sites 2 and 12

Contractor name: Ahtna Global, LLC

Contract number: W91238-19-C-0027

Contract title: Former Fort Ord Basewide Groundwater and Soil Vapor Treatment and Monitoring

Work Assignment Number: N/A

Guidance used to prepare QAPP: Uniform Federal Policy for Quality Assurance Project Plans, Final, Version 1, March 2005

Regulatory program: Comprehensive Environmental Response Compensation and Liability Act (CERCLA) as amended by Superfund Amendment and Reauthorization Act

Approval entity: U.S. Environmental Protection Agency (EPA), California Department of Toxic Substance Control (DTSC), and California Regional Water Quality Control Board, Central Coast Region (CCRWQCB)

Data users: U.S. Department of the Army (Army), USACE, EPA, DTSC, CCRWQCB, Army/USACE contractors, property owners, occupants and managers, and the public.

QAPP type: Generic _____ Project-Specific **X** _____

Planning session date/s: December 16, 2020

Dates and titles of QAPP documents written for previous site work:

September 2013, Final Quality Assurance Project Plan/Field Sampling Plan, Remedial Investigation/Feasibility Study Addendum at Sites 2 and 12, Former Fort Ord, California (Appendix A to the RI/FS Addendum Work Plan, AR# BW-2665A)

March 2015, Quality Assurance Project Plan, Former Fort Ord, California, Volume I, Appendix C, Final Revision 0, Soil Gas Monitoring at Sites 2 and 12 (AR# BW-2727B)

March 2016, Quality Assurance Project Plan, Former Fort Ord, California, Volume I, Appendix C, Final Revision 1, Soil Gas Monitoring at Sites 2 and 12 (AR# BW-2792A)

May 2017, Quality Assurance Project Plan, Former Fort Ord, California, Volume I, Appendix C, Final Revision 2, Soil Gas Monitoring at Sites 2 and 12 (AR# BW-2792C)

January 2018, Quality Assurance Project Plan, Former Fort Ord, California, Volume I, Appendix C, Final Revision 3, Soil Gas Monitoring at Sites 2 and 12 (AR# BW-2792E)

February 2019, Quality Assurance Project Plan, Former Fort Ord, California, Volume I, Appendix C, Final Revision 4, Soil Gas Monitoring at Sites 2 and 12 (AR# BW-2792G)

November 2019, Quality Assurance Project Plan, Former Fort Ord, California, Volume I, Appendix C, Draft Addendum No. 1, Soil Gas Monitoring at Sites 2 and 12 (AR# BW-2792H)

August 2020, Quality Assurance Project Plan, Former Fort Ord, California, Volume I, Appendix C, Final Revision 5, Soil Gas Monitoring at Sites 2 and 12 (AR# BW-2792M)

Organizational partners (stakeholders) and connection with lead organization: USACE, Army (Lead Agency/Owner), EPA (Lead Oversight Agency), DTSC (Support Agency), CCRWQCB (Support Agency), Shea Properties (Property Owner), Target Corporation (Property Owner), Marina Community Partners (Property Owner), and public participants.

2.2 Distribution List (Worksheet #3)

The following entities will receive copies of the approved QAPP, subsequent QAPP revisions, addenda, and amendments. The distribution list may change and will be revised for each QAPP revision submitted. Ahtna field team members will also receive a copy of the QAPP. A complete copy of the original version and all revisions of the QAPP, including addenda and amendments, will be maintained by Ahtna and will be available upon request.

QAPP Recipients	Title	Organization	Telephone	Email
Duane Balch	Senior Project Manager	USACE	(916) 557-7450	Duane.C.Balch@usace.army.mil
Dana Gentry	Project Manager	USACE	(916) 557-7452	Dana.K.Gentry@usace.army.mil
William Collins	Base Realignment and Closure (BRAC) Environmental Coordinator	Army	(831) 242-7920	William.K.Collins.civ@mail.mil
Fort Ord Administrative Record	N/A	Army	(831) 393-9693	adminrecord@fortordcleanup.com
Maeve Clancy	Project Manager	EPA	(415) 947-4105	Clancy.Maeve@epa.gov
Min Wu	Project Manager	DTSC	(916) 255-3621	Min.Wu@dtsc.ca.gov
Amber Sellinger	Project Manager	CCRWQCB	(805) 549-3866	Amber.Sellinger@waterboards.ca.gov
Jonathan Whipple	Project Chemist	USACE	(916) 557-5302	Jonathan.P.Whipple@usace.army.mil
Bridget Floyd	Technical Lead	USACE	(916) 539-1348	Bridget.M.Floyd@usace.army.mil
Tom Ghigliotto	Field Coordinator and Inspector	Chenega	(831) 824-2318	Thomas.F.Ghigliotto@usace.army.mil
Chuck Holman	Environmental Program Manager	Ahtna	(916) 275-9989	cholman@ahtna.net
Bruce Wilcer	QC Manager	Ahtna	(925) 222-6595	bwilcer@ahtna.net
Derek Lieberman	Project Manager	Ahtna	(831) 224-3327	dliberman@ahtna.net
Eric Schmidt	Project Chemist	Ahtna	(831) 582-1348	eschmidt@ahtna.net
Shaelyn Hession	Task Lead, Soil Gas Remedy	Ahtna	(831) 200-6072	shession@ahtna.net
Andrew Mauck	Task Lead, Soil Gas Monitoring	Ahtna	(831) 402-0727	amauck@ahtna.net
Brian Whittaker	Project Manager	Eurofins	(916) 605-3355	BrianWhittaker@EurofinsUS.com
Mike Weaver	Co-Chair	Fort Ord Community Advisory Group	N/A	michaelweaver@mac.com

QAPP Recipients	Title	Organization	Telephone	Email
Joe Carter	EPA Consultant	TechLaw, Inc.	N/A	Joe.carter@techlawinc.com

2.3 Project Personnel Sign-Off Sheet (Worksheet #4)

Copies of this form will be signed by key project personnel from each organization to indicate that they have read the applicable QAPP sections and will perform the tasks as described. Key project personnel include the lead organization, contractors, subcontractors, lead field personnel, Project Manager, Data Reviewer, assessment personnel, and laboratory QA Manager. Supervisory or oversight personnel are responsible for communicating the requirements of the applicable portions of the QAPP to those performing the work. Each organization will forward signed sheets to Ahtna to be stored in the central project file.

Organization: Ahtna

Project Personnel	Title	Telephone	Signature
Chuck Holman	Program Manager	(916) 275-9989	
Derek Lieberman	Project Manager	(831) 224-3327	
Shaelyn Hession	Task Lead, Soil Gas Remedy O&M	(831) 200-6072	
Andrew Mauck	Task Lead, Soil Gas Monitoring	(831) 402-0727	
Eric Schmidt	Project Chemist	(831) 582-1348	
Mark Fisler	Field Services Coordinator	(831) 224-3133	
Bruce Wilcer	QC Manager	(925) 222-6595	

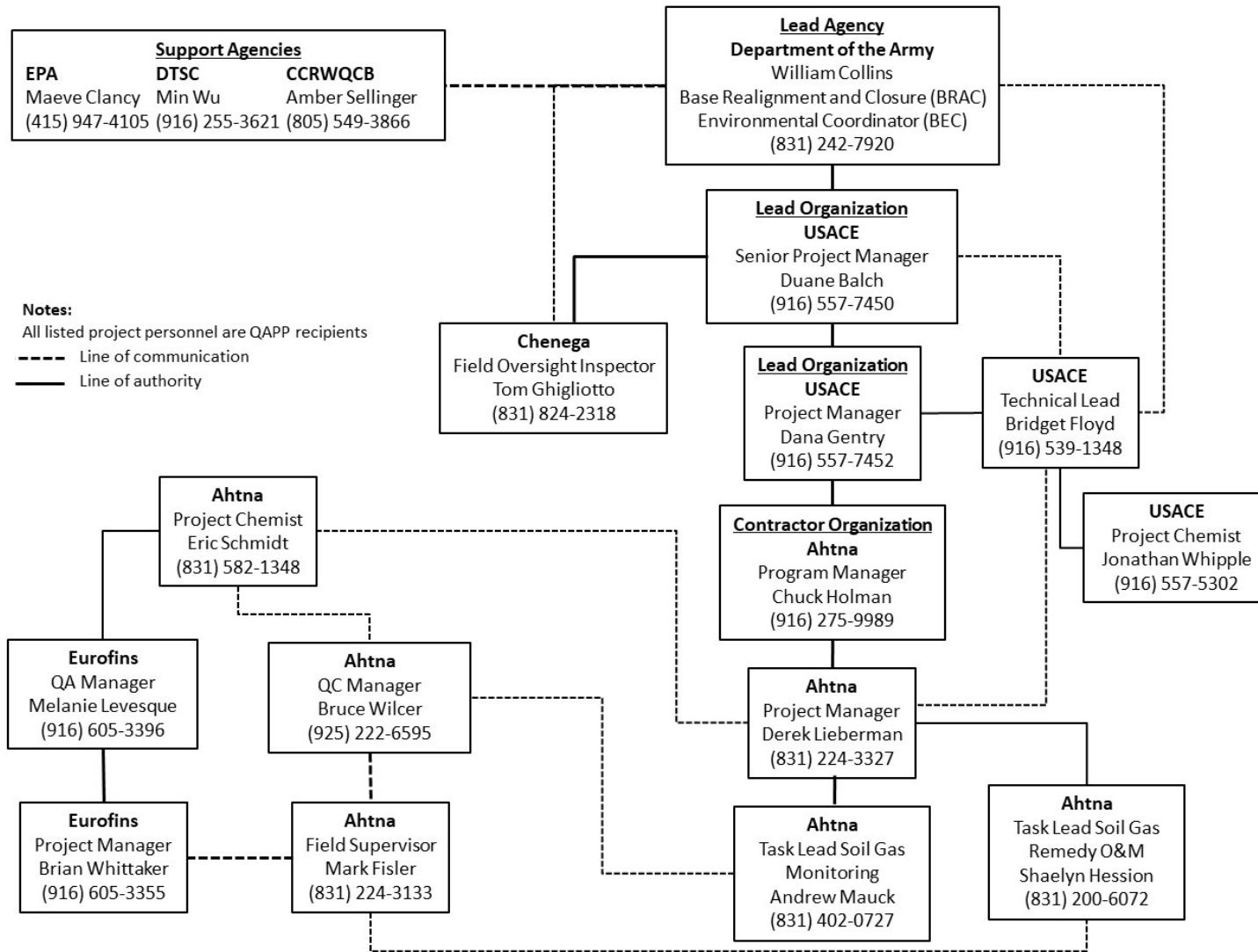
Project Personnel Sign-Off Sheet (Worksheet #4)

Organization: Eurofins (Ahtna Subcontractor)

Project Personnel	Title	Telephone	Signature
Melanie Levesque	QA Manager	(916) 605-3396	
Sepideh Saeed	Lab Director	(916) 605-3383	
Brian Whittaker	Project Manager	(916) 605-3355	

2.4 Project Organizational Chart (Worksheet #5)

Reporting relationships between organizations involved in the project, including the lead organization, contractors, and subcontractor organizations are identified below.



2.5 Communication Pathways (Worksheet #6)

This section describes the communication pathways and modes of communication to be used during the project. Procedures for requesting and obtaining approval between project personnel and subcontractors are included. Procedures for modification to QAPP-specified requirements (if needed) are also included.

Communication Drivers	Responsible Entity	Name	Telephone	Procedure (Timing, pathways, etc.)
Point of contact with Army BRAC Office	USACE Technical Lead	Bridget Floyd	(916) 539-1348	Materials and information regarding the project will be forwarded to Army BRAC Office through USACE Technical Lead.
Point of contact for the Lead Organization Project Manager	Ahtna Project Manager	Derek Lieberman	(831) 224-3327	Materials and information regarding the project will be forwarded to USACE by the Ahtna Project Manager.
QAPP changes in the field	Ahtna Project Manager	Derek Lieberman	(831) 224-3327	Ahtna Project Manager will be notified of proposed field changes to the QAPP and shall notify the USACE Technical Lead and USACE Project Chemist prior to implementation.
Daily progress reports/ Field QC issues	Ahtna Field Supervisor	Mark Fisler	(831) 224-3133	Ahtna Field Supervisor will report field/sampling progress and field QC issues to Ahtna Project Manager and Task Leads daily. Ahtna Project Manager or Task Leads will notify the Ahtna QC Manager of issues within one business day.
Field and laboratory data quality issues	Ahtna Project Chemist	Eric Schmidt	(831) 582-1348	Ahtna Project Chemist will report lab QC issues to USACE Technical Lead/USACE Project Manager within two business days.

Communication Drivers	Responsible Entity	Name	Telephone	Procedure (Timing, pathways, etc.)
Data Usability issues	USACE Technical Lead/USACE Project Manager	Bridget Floyd Dana Gentry	(916) 539-1348 (916) 557-7452	USACE Technical Lead/USACE Project Manager to inform USACE Project Chemist of any field/laboratory data quality issues that could impact data quality.
Field and analytical corrective actions	Ahtna Field Supervisor	Mark Fisler	(831) 224-3133	Within two business days of the occurrence, non-conformance and QC issues will be communicated to the Ahtna Project Manager and Program Chemist, who will determine the need for corrective action.
Release of analytical data	Ahtna Project Chemist	Eric Schmidt	(831) 582-1348	Analytical data will not be released until review or validation is completed, as appropriate. The Ahtna Project Chemist will approve the release of data to the Ahtna Project Manager.
Changes to the QAPP	Ahtna Project Manager	Derek Lieberman	(831) 224-3327	Significant changes to the QAPP must be approved by the Ahtna Project Manager, USACE Technical Lead, and USACE Project Chemist prior to implementation. The Lead Organization (USACE) will approve significant corrective actions or procedural changes prior to implementation.
Data import and export	Ahtna Data Manager	Teri Farrell-Bage	(925) 915-6255	The Ahtna Data Manager will upload field data and lab results into the database tracking system.

2.6 Personnel Responsibilities and Qualifications Table (Worksheet #7)

Project personnel associated with Ahtna, USACE and subcontractor staff are identified and include data users, decision-makers, project managers, QA/QC and health and safety personnel, engineers, hydrogeologists, field staff and subcontractors. Resumes for project team members are maintained by Ahtna in the project file and are available upon request.

Name	Title	Organizational Affiliation	Responsibility	Education and Experience Qualifications
Duane Balch	Senior Project Manager	USACE	Lead organization's senior project manager	Resume in USACE file
Dana Gentry	Project Manager	USACE	Lead organization's project manager	Resume in USACE file
Bridget Floyd	Technical Lead	USACE	Lead organization's technical lead	Resume in USACE file
Jonathan Whipple	Project Chemist	USACE	Lead organization's QA chemist	Resume in USACE file
Chuck Holman	Program Manager	Ahtna	Contractor organization's program manager	Resume in Ahtna file
Pete Rice	Project Health & Safety Officer	Ahtna	Contractor organization's health and safety program manager	Resume in Ahtna file
Holly Dillon	Site Safety & Health Officer	Ahtna	Oversees health and safety for field operations	Resume in Ahtna file
Andrew Mauck	Task Lead, Soil Gas Monitoring	Ahtna	Soil gas sampling fieldwork	Resume in Ahtna file
Shaelyn Hession	Task Lead, Soil Gas Remedy O&M	Ahtna	Oversees operations and maintenance (O&M) of the SVETS	Resume in Ahtna file
Derek Lieberman	Project Manager	Ahtna	Oversees Fort Ord projects and technical plans, manages daily tasks and field activities, performs inspections, and manages report preparation	Resume in Ahtna file
Bruce Wilcer	QC Manager	Ahtna	Analyzes and interprets analytical and geologic data	Resume in Ahtna file
Eric Schmidt	Project Chemist	Ahtna	Manages QAPP preparation, analytical laboratories, data review, data validation, and QC	Resume in Ahtna file
Teri Farrell-Bage	Data Manager	Ahtna	Uploads field data and lab results into the database tracking system	Resume in Ahtna files
Mark Fisler	Field Supervisor	Ahtna	Supervises field activities and performs inspections	Resume in Ahtna file

Name	Title	Organizational Affiliation	Responsibility	Education and Experience Qualifications
Andrew Mauck	Field Technician	Ahtna	Conducts fieldwork	Resume in Ahtna file
Brian Whittaker	Project Manager	Eurofins	Laboratory point of contact, manages project samples	Resume in Ahtna file
Melanie Levasque	QA Manager	Eurofins	Laboratory analytical data quality	Resume in Ahtna file

2.7 Special Training Requirements and Certification (Worksheet #8)

Special training requirements for Ahtna and subcontractor personnel working on the Sites 2/12 project may include one or more of the following:

- HAZWOPER 40-Hour Training and Annual 8-hour Refresher Training: Ahtna and subcontractor field staff working at Sites 2/12 are required to have certification of completion of the 40-Hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training course in compliance with Title 29 of the Code of Federal Regulations, Part 1910.120 (29 CFR 1910.120). Annual re-certification of the 40-Hour Training must be maintained through the completion of annual 8-Hour OSHA HAZWOPER refresher courses.
- First Aid and Cardiopulmonary Resuscitation (CPR) training and certification: First Aid and CPR training and certification are required for designated field personnel as prescribed by the Site Safety and Health Officer (SSHO). Each field staff member will be trained and maintain certification in First Aid and CPR.
- USACE Construction Quality Management Certification: Ahtna QC staff is required to have USACE Construction Quality Management (CQM) certification. CQM incorporates the Three Phase Quality Control Process, as described in Worksheet #31.

Training records are maintained in the Ahtna project file.

2.8 Project Planning Session Summary (Worksheet #9)

Project Name: Soil Gas Monitoring at Sites 2 and 12		Site Name: Sites 2 and 12		
Start Date: Ongoing		Site Location: Former Fort Ord, California		
Project Manager: Derek Lieberman, Ahtna				
Date of Planning Session: December 16, 2020				
Planning Session Purpose: Define data quality objectives (DQOs) and analytic approach criteria for the SVETS.				
Name	Title/Role	Affiliation	Telephone	email Address
Derek Lieberman	Project Manager	Ahtna	(831) 224-3327	dlieberman@ahтна.net
Sylvester Kosowski	Task Lead, Soil Gas Remedy O&M	Ahtna	(831) 402-5850	skosowski@ahтна.net
Eric Schmidt	Project Chemist	Ahtna	(831) 582-1348	eschmidt@ahтна.net
Holly Dillon	SSHO	Ahtna	(831) 324-3299	hdillon@ahтна.net

Planning Session Summary:

Review contract to determine QAPP requirements and reviewed QAPP Revision 5 for potential updates needed.

Action Items:

Based on this review, Ahtna will:

- Initiate QAPP Revision 6 update.
- Operation of individual SVE wells will depend on analytical results for samples collected from the soil gas probes and groundwater monitoring and extraction wells.
- After review of the previous four quarters of data (Fourth Quarter 2019 through Third Quarter 2020) and comparison to the analytic approach in the QAPP, update the list of sampled soil gas probes quarterly and annually.
- Add updated Eurofins standard operating procedures (SOPs).
- Clarify the analytic approach for SVETS operation related to soil gas concentrations and groundwater remediation.

2.9 Problem Definition/Data Quality Objectives (Worksheet #10)

Data Quality Objectives (DQOs) were applied to optimize and describe the data collection objectives for soil gas at Sites 2/12. The DQO process, as prescribed by the *Guidance on Systematic Planning using the Data Quality Objectives Process* (EPA, 2006), is divided into seven steps:

- State the problem
- Identify the decisions
- Identify inputs to the decisions
- Define study boundaries
- Develop the analytic approach
- Specify limits on decision errors
- Optimize study design

The seven steps of the DQO process as applied to each project are described in the following sections.

2.9.1 Problem Statement

Historical activities at Sites 2/12, including disposal of solvents at Site 12, resulted in releases of volatile organic compounds (VOCs), primarily trichloroethene (TCE) and tetrachloroethene (PCE), into soil and groundwater. Although groundwater remediation activities have been conducted since 1999 and have successfully reduced TCE concentrations in groundwater at Sites 2/12, PCE concentrations began increasing in 2011 (ITSI, 2012), creating the potential for vapor intrusion to the overlying retail area. A human health risk assessment (HHRA) was conducted using indoor air and sub-slab soil gas data collected as part of the Remedial Investigation/Feasibility Study (RI/FS) Addendum at Sites 2/12. The HHRA concluded the vapor intrusion pathway does not present an unacceptable risk to human health (AES, 2015). Groundwater in the upper portion of the Upper 180-Foot Aquifer, where soil vapors may form, was investigated in 2013 and found to contain a plume of PCE above its aquifer cleanup level (ACL; AES, 2015). Soil was found to be uncontaminated at Site 12. Soil gas was also investigated at Site 12 in 2013 and distinct PCE and TCE plumes were identified in the vadose (or unsaturated) zone of the Upper 180-Foot Aquifer (AES, 2015).

Follow up investigations and operation of an SVE and air sparge (AS) pilot study treatment system in 2014 (Figure 1) identified a groundwater plume and soil gas plume of TCE in the southern Site 12 area. The pilot study demonstrated that SVE and AS are effective technologies for remediation of soil gas and groundwater at Site 12 (AES, 2015); however, it was determined SVE and additional groundwater extraction and treatment (instead of AS) would likely be more effective for achieving remedial action objectives as described in the Explanation of Significant Differences No. 1 (ESD No. 1; Army, 2016). Accordingly, the SVETS and one additional groundwater extraction well were constructed per the *Final Remedial Action Work Plan Addendum, Sites 2 and 12 Groundwater Remediation, Former Fort Ord, California* (RAWP Addendum; AEI, 2015).

Further data collection is needed in the Sites 2/12 area to:

- Monitor the nature and extent of COC contamination in groundwater and soil gas;
- Support the continued remediation of the COCs in groundwater and soil gas (COCs in soil gas are a source of contamination in groundwater at Site 12);
- Ensure compliance with air quality standards identified in Regulation II (New Sources), Rule 207 (Air District, 2011) and Regulation X (Toxic Air Contaminants), Rule 1000 (Air District, 2017);
- Evaluate and optimize SVETS operations; and
- Support site closure.

The modified groundwater cleanup levels, soil gas cleanup levels (SGCLs), and modified groundwater remedy are described in ESD No. 1 (Army, 2016). Project action limits for soil gas are also summarized in Worksheet #15.

2.9.2 Decision Identification

The primary decisions associated with the Sites 2/12 remediation project are whether:

- The soil gas monitoring program adequately assesses site conditions within the site physical and temporal boundaries;
- The Sites 2/12 remedy is in continued compliance with the *Record of Decision, Basewide Remedial Investigation Sites, Fort Ord, California* (RI Sites ROD; Army, 1997) and ESD No. 1 (Army, 2016);
- COCs in soil gas will partition into groundwater at concentrations exceeding ACLs; and
- Operation of the SVETS is required to reduce COC concentrations in soil gas that may partition into groundwater at concentrations exceeding ACLs.

Soil gas monitoring at Sites 2/12 will be conducted to determine whether:

- The SVETS is effectively and efficiently reducing COC concentrations in the vadose zone that could partition into groundwater at concentrations exceeding ACLs;
- Soil gas COC concentrations exceed the SGCLs at points near the groundwater interface may partition into groundwater at concentrations exceeding ACLs; and
- Site closure is warranted.²

SVETS monitoring at Sites 2/12 will be conducted to determine whether:

- SVTU effluent meets Air District discharge requirements;
- SVTU granular activated carbon (GAC) requires change-out;
- COC mass is being removed from the vadose zone and at what rate;
- SVE well extraction performance is optimal; and
- Current SVE well sampling frequency is adequate to meet project objectives.

² Site closure is dependent on decision criteria for completion of the groundwater restoration remedial action as described in QAPP Appendix A. Soil gas monitoring is only relevant for determining whether soil gas may be a continuing source of COCs to groundwater.

2.9.3 Decision Inputs

Inputs to decisions are as follows:

- COC concentrations in soil gas;
- COC concentrations in groundwater; determined in accordance with QAPP Appendix A (Ahtna, 2021);
- Soil gas screening levels (SG-SLs, Worksheet #15);
- SGCLs (Worksheet #15);
- Groundwater ACLs (identified in QAPP Appendix A [Ahtna, 2021] and ESD No. 1 [Army, 2016]);
- COC concentrations in the SVTU effluent to confirm whether discharge requirements are being met;
- COC concentrations in the SVTU effluent to determine whether GAC requires change-out;
- COC concentrations in the SVTU influent to determine the amount and rate of COC mass removal from the vadose zone;
- SVETS flow rate, vacuum and temperature data collected to evaluate and optimize system operation;
- SVE well flow rates as a function of applied vacuum;
- Applied vacuum in the SVE wells; and
- Induced vacuum in soil gas probes.

2.9.4 Definition of Study Boundaries

The Sites 2/12 study area is defined by the retail development tracts east of State Route 1, south of Imjin Parkway, west of 2nd Avenue, and north of the former 10th Street (Figure 1). Soil gas samples will be collected from soil gas probes at 10-foot intervals from approximately 10 feet below ground surface (bgs) to 70 feet bgs, and from SVE wells screened from approximately 45 feet bgs to 65 feet bgs. Twenty-four (24) soil gas probe locations and ten (10) SVE well locations at Site 12 are shown on Figure 1. Soil gas samples will also be collected from the SVTU located in a fenced compound adjacent to the Sites 2/12 groundwater treatment plant. A process flow diagram for the SVTU with potential sampling locations is shown in Figure 2.

2.9.5 Development of the Analytic Approach

2.9.5.1 Soil Gas Monitoring

The following analytic approach will be applied to soil gas probe sampling frequency:

- If two consecutive quarters of monitoring data from a soil gas probe show concentrations of COCs less than or equal to their respective SGCLs, but greater than or equal to their SG-SLs, then the soil gas probe shall be evaluated for annual sampling.
 - If it is determined that quarterly data from the soil gas probe is necessary for defining the soil gas plume and/or evaluating remedy status, then the soil gas probe shall continue to be sampled quarterly.

- If it is determined that soil gas probes laterally or vertically adjacent to the soil gas probe have detections of COCs greater than their respective SGCLs, then the soil gas probe shall continue to be sampled quarterly.
- If it is determined that annual data from the soil gas probe is sufficient for defining the soil gas plume and/or evaluating remedy status, then the soil gas probe shall be sampled annually.
- If two consecutive annual monitoring results from a soil gas probe show concentrations of COCs less than or equal to their respective SG-SLs, then the soil gas probe will be evaluated for removal from the sampling program.
- If an annual monitoring event shows COC concentrations above their respective SG-SLs at an annual soil gas probe, then the soil gas probe sampling frequency may be increased to quarterly.
- If soil gas probes laterally or vertically adjacent to a soil gas probe sampled annually have detections of COCs greater than their respective SGCLs, then the soil gas probe may be returned to a quarterly monitoring schedule.
- If monitoring indicates the soil gas monitoring network no longer provides vertical or lateral control of COCs, then additional soil gas probes may be proposed for sampling.
- If a soil gas probe cluster is no longer needed for the soil gas monitoring program, then it will be proposed for decommissioning.

This analytic approach is also shown graphically in Figure 3. Exceptions to the analytic approach that may be implemented based on decision inputs are:

- If a soil gas probe is located vertically and laterally adjacent to a storefront and is in an area with concentrations of soil gas COCs historically greater than SGCLs,³ then it will be sampled quarterly or at an appropriate frequency based on historical data and anticipated data needs as determined by the Army and concurred with by the EPA, DTSC and CCRWQCB (collectively the “regulatory agencies”).
- If a soil gas probe is located vertically adjacent to groundwater with concentrations of groundwater COCs greater than ACLs,⁴ then it will be sampled quarterly or at an appropriate frequency based on historical data and anticipated data needs as determined by the Army and concurred with by the regulatory agencies.

2.9.5.2 Soil Gas Plume Limits

For defining soil gas plume limits, the parameter of interest is the maximum COC concentrations detected in a soil gas probe as compared to the SGCLs, SG-SLs, or historical COC concentration trends at the soil gas probe.

- If the maximum COC concentration detected in a soil gas probe is greater than or equal to the SGCL, then that monitoring point is within the soil gas plume limits.

³ Probes SG-12-02-10, SG-12-04-10 and SG-12-06-10 currently meet these criteria (Table 1).

⁴ Probes SG-12-01-65 and SG-12-04-65 currently meet these criteria (Table 1).

- If the maximum COC concentration detected in a soil gas probe is less than the SGCL, then that monitoring point is outside the soil gas plume limits.

2.9.5.3 Perimeter Control

For perimeter control, the minimum value detected in the monitoring point (e.g., non-detect at the limit of detection [LOD]) is the parameter of interest.

- If COCs are not detected in a soil gas probe and COCs are not detected in all adjacent monitoring points, then that soil gas probe is outside the study area boundary as defined in Section 2.9.4 and not needed for perimeter control.
- If COCs are not detected in a soil gas probe, but COCs are detected in adjacent monitoring points, then that soil gas probe defines the outer perimeter of the study area and that soil gas probe may continue to be monitored for perimeter control in accordance with the analytic approach for soil gas monitoring described above.

2.9.5.4 Discharge Limit Compliance

Discharge limit compliance is determined when the requirements of both Rule 207 (Air District, 2011) and Rule 1000 (Air District, 2017) are met. Non-compliance with either rule indicates a discharge exceedance.

Under Rule 207 (Air District, 2011), Best Available Control Technology is required for any new or modified stationary source with a potential to emit specific pollutants at rates greater than or equal to those listed in Table 4.1.1 of Rule 207 or Section 5.2 of Rule 207, whichever is more stringent. Of the 13 pollutant categories listed in Table 4.1.1 and Section 5.2 of Rule 207, only VOCs are expected to be emitted.⁵ The more stringent requirement for VOCs is in Section 5.2 at 25 pounds per day (lbs/day).

For each quarterly period, the total VOC emission rate shall be calculated using the maximum detected total VOC concentration at the SVTU effluent for the quarter and the average SVTU influent flow rate for the quarter.⁶ The following analytic approach shall then be applied to determine whether discharge limits are being met at the SVTU effluent with respect to Rule 207:

- If the calculated total VOC emission rate is less than 25 lbs/day, then the SVETS will continue to operate.
- If the calculated total VOC emission rate is greater than or equal to 25 lbs/day, then a confirmation sample will be collected from the SVTU effluent and analyzed with a 24-hour turnaround time (TAT).
- If the total VOC emission rate calculated using analytical results from the confirmation sample is greater than or equal to 25 lbs/day, then the SVETS will be shut down, operating conditions and GAC loading evaluated, SVE well flow rates adjusted as necessary, and a variance report issued for any out-of-limits operation. Following operational corrective actions, which may include a

⁵ Based on the results of the RI/FS (HLA, 1995) and the RI/FS Addendum (AES, 2015), and the determinations of the RI Sites ROD (Army, 1997) and ESD No. 1 (Army, 2016).

⁶ Total VOCs includes PCE and TCE, the Sites 2/12 soil gas COCs (Army, 2016).

GAC change-out, the SVETS will be restarted and a verification sample will be collected and analyzed to ensure compliance post-adjustment.

- If the total VOC emission rate calculated using analytical results from the verification sample and calculated SVETS flow rates is less than 25 lbs/day, then the SVETS will continue to operate.
- If the total VOC emission rate calculated using analytical results from the verification sample is greater than or equal to 25 lbs/day, then the SVETS will be shut down, and operating conditions and GAC loading re-evaluated. Following operational corrective actions, the SVETS will be restarted and resampled to verify compliance.

The SVETS is a new or modified source that has the potential to emit very low levels of carcinogenic toxic air contaminants or toxic air contaminants; therefore, emissions from the SVETS are subject to Rule 1000 (Air District, 2017). For each quarterly period, the hazard index (HI) and risk to a hypothetical receptor 50 meters away from the SVTU discharge point will be calculated using output from AERSCREEN modeling and the following analytic approach shall be used to determine whether discharge limits are being met at the SVTU effluent with respect to Rule 1000:⁷

- If the calculated HI is less than 1 and the excess cancer risk is less than 10 per million (1×10^{-5}), then the SVETS will continue to operate.
- If the calculated HI is greater than or equal to 1 or the risk is greater than 1×10^{-5} , then a confirmation sample will be collected from the SVTU effluent and analyzed with a 24-hour TAT.
- If the HI is greater than or equal to 1 or the risk is greater than 1×10^{-5} as calculated using analytical results from the confirmation sample, then the SVETS will be shut down, operating conditions and GAC loading evaluated, SVE well flow rates adjusted as necessary, and a variance report issued for any out-of-limits operation. Following operational corrective actions, which may include a GAC change-out, the SVETS will be restarted and resampled to verify compliance.
- If the HI is less than 1 and the risk is less than or equal to 1×10^{-5} as calculated using analytical results from the verification sample and calculated SVETS flow rates, then the SVETS will continue to operate.
- If the HI is greater than or equal to 1 or the risk is greater than 1×10^{-5} as calculated using analytical results from the verification sample, then the SVETS will be shut down, and operating conditions and GAC loading re-evaluated. Following operational corrective actions, the SVETS will be restarted and resampled to verify compliance.

2.9.5.5 GAC Change-out

The decision to do a GAC change-out will be made on a case-by-case basis taking into consideration the SVETS operating conditions, including, but not limited to:

- Online operational time into current GAC cycle;
- SVTU influent and effluent COC concentrations and concentration trends;
- SVETS operating flow rates;

⁷ The AERSCREEN will produce estimates of “worst-case” 1-hour concentrations for a single source and includes conversion factors to estimate “worst-case” 3-hour, 8-hour, 24-hour, and annual concentrations. See <https://www.epa.gov/scram/air-quality-dispersion-modeling-screening-models> for more information.

- Operating SVE well COC concentrations and concentration trends; and
- Historical, current and planned future SVETS operations.

Generally, if the calculated quarterly total VOC emission rate is greater than or equal to 22.5 lbs/day (90% of the maximum emission rate of 25 lbs/day), then the need for a GAC change-out will be determined by the USACE Technical Lead and the Ahtna Project Manager based on SVETS operating conditions.

2.9.5.6 Soil Gas Plume Remediation

Assessment of COC removal from the vadose zone resulting from operation of the SVETS is conducted through a soil gas monitoring program that evaluates plume migration and COC concentrations. SVE well and soil gas probe monitoring data will be used to evaluate the operational status of individual SVE wells and for evaluation of remediation progress. The analytic approach for determining the operational status of SVE wells with respect to soil gas plume remediation are:

- An SVE well will continue to be operated if any COC detected in the SVE well is greater than the corresponding SGCL (Worksheet #15).
- An SVE well will continue to be operated if any COC detected in a soil gas probe within the radius of influence (ROI) of the SVE well and within 20 feet of the groundwater interface has a concentration greater than the corresponding SGCL (Worksheet #15).
- An SVE well will continue to operate if its ROI and analytical data from nearby SVE wells and/or soil gas probes indicate operation of the SVE well is necessary for completion of the groundwater restoration remedial action.
- An SVE well will be shut off if COCs detected in the SVE well are less than or equal to the SGCL for two consecutive quarterly monitoring events, and if its ROI and analytical data from nearby SVE wells and/or soil gas probes indicate operation of the SVE well is no longer necessary for completion of the groundwater restoration remedial action.

Site closure is dependent on decision criteria for completion of the groundwater restoration remedial action as described in QAPP Appendix A (Ahtna, 2021). Soil gas monitoring is only relevant for determining whether soil gas may be a continuing source of COCs to groundwater; therefore, this analytic approach may be subordinated by the analytic approach for groundwater plume remediation described below.

2.9.5.7 Groundwater Plume Remediation

Assessment of groundwater cleanup resulting from operation of the Sites 2/12 groundwater treatment system is conducted through a groundwater monitoring program that evaluates plume migration and COC concentrations, as described in QAPP Appendix A (Ahtna, 2021). Soil gas remediation was established to prevent COCs in soil gas from partitioning into groundwater at concentrations exceeding ACLs. As operation of the SVETS reduces COC mass in soil gas, the process may be reversed and COCs will partition from groundwater into soil gas where they may be removed by the SVETS; therefore, groundwater well data will also be used to evaluate the operational status of individual SVE wells. The analytic approach for determining the operational status of SVE wells with respect to groundwater plume remediation are:

- An SVE well will be operated if it is located within an area where any groundwater COC detected in a monitoring well is greater than the corresponding ACL (Ahtna, 2021). However:
 - If analytical data from the operating SVE well and nearby groundwater monitoring wells indicate the SVE well is not facilitating groundwater remediation, then a recommendation will be presented for regulatory agency approval to discontinue operation of the SVE well.
 - If a groundwater monitoring well has COC concentration trends that are statistically decreasing but has adjacent soil gas probes with concentrations of COCs greater than SGCLs within 20 feet of the groundwater interface, then a recommendation will be presented for regulatory agency approval to discontinue operation of the SVE well.
- An SVE well will be operated if the SVE ROI and analytical data from nearby groundwater wells indicate operation of the SVE well may supplement groundwater plume remediation.
- An SVE well will be shut off if the SVE ROI and analytical data from nearby groundwater wells indicate operation of the SVE well is no longer necessary for groundwater plume remediation.

2.9.5.8 SVE Well Sampling Frequency

SVE wells will be sampled quarterly when operating as part of the SVETS; however, due to the density of soil gas probes in the Site 12 area, it is not necessary to sample an SVE well after its operation has been terminated in accordance with the analytic approach for plume remediation. If an SVE well is no longer needed for plume remediation, then it may be proposed for decommissioning.

2.9.6 Specification of Limits of Decision Errors

Because decisions pertaining to remediation will be based on sample collection and analysis, decision errors may result from the limitations of sampling or analytical techniques. To limit analytical errors, analytical method requirements have been established that include precision, accuracy and sensitivity goals that will produce data capable of supporting project decisions. To limit sampling errors, sample collection protocol specified in SOPs (Attachment A) will be strictly followed and sufficient samples will be collected to support project decisions. Sample volume and preservation requirements will be followed as described in Worksheet #19.

False positive and false negative decision errors are defined in the context of hypothesis testing, where the terms are defined with respect to the null hypothesis. A false positive decision error occurs when the null hypothesis is rejected when it is true. A false negative decision occurs when the null hypothesis is not rejected when it is false. The null hypothesis is COC concentrations in samples are greater than the SGCLs. Potential consequences of a false positive detection might include:

- Unnecessarily performing remediation activities where COC concentrations are lower than SGCLs.
- Increasing or maintaining sampling frequency when it is not necessary.

The potential consequence of a false negative result is COCs remaining in soil gas at levels that are potentially a source of groundwater COC concentrations above ACLs.

Decision errors are most likely to occur when the measured concentration is near the SGCL, or in the case of non-detects, when the limit of quantitation (LOQ) is near the SGCL. To control decision errors when the LOQ is near the SGCL, the laboratory is required to report any detections below the LOQ (but above the LOD), thereby giving the data user additional information regarding trace level contamination. This may be the case when sample dilution raises the LOQ to a level near the cleanup level.

False negatives or positives could occur due to laboratory error, contamination, or dilution. False negatives or positives could occur if ambient air containing, respectively, lower or higher concentrations of COCs compared to the soil gas is inadvertently introduced during sample collection. All soil gas probes and sampling assemblies are leak tested with helium in the field prior to sampling to mitigate the possibility of accidental ambient air contamination.

Definitive data are required for supporting project decisions. It is assumed that, if the precision, accuracy, and sensitivity requirements specified in the QAPP are met, the data will be usable for decision-making purposes.

2.9.7 Optimization of the Design

The sampling approach is non-random and based on professional judgment. To limit uncertainty in obtained environmental data, criteria for the precision, accuracy, representativeness, completeness, comparability parameters and reporting limits for the parameters have been developed and are presented in Sections 3 through 5 of this QAPP. Measurement errors will be controlled by using the appropriate sampling and analytical methods, adhering to the requirements of QSM Version 5.3 (DoD, 2019), and data validation/review to verify laboratory processes and measurement quality objectives. The data that meet these criteria will be of definitive quality.

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

Project quality objectives (PQOs), in terms of type, quantity, and quality of data determined using a systematic planning process, are described below. The PQOs in the form of qualitative and quantitative statements are provided.

2.10.1 Data Users

The data will be used by the USACE and its contractors, the regulatory agencies, property owners and occupants, citizen groups and members of the public.

2.10.2 Data Uses

The data collected will be used to continue to delineate the magnitude and extent of COCs in soil gas. The data will be used to assess soil gas concentrations compared to SGCLs and the progress of remediation toward site closure.

2.10.3 Data Types

Analytical data are needed to make decisions. Sample media and analytes for project samples will include soil gas and COCs by EPA Method TO-15, respectively. Sample type and location-specific analytes are included in Worksheet #15.

2.10.4 Data Validation

Data review and validation are described in Worksheets #34 – #37.

2.10.5 Data Collection

The analytic approach identified in Worksheet #10 will be followed to determine sampling frequency (quarterly or annually) and will also follow the schedule provided in Worksheet #17. Sample collection procedures are provided in the SOPs in Attachment A.

Soil gas probes will be purged of three probe volumes prior to sampling to ensure stagnant air is removed and a representative soil gas sample is collected from the desired sample location. The probe volume calculation methodology is depicted in Figure 4 and purge volumes for each soil gas probe are listed in Table 1. Soil gas probe purge volume calculations vary depending on tubing length, tubing diameter, borehole diameter, sand-pack thickness, and dry bentonite thickness. All Sites 2/12 soil gas probes were installed with 8-inch diameter boreholes, 0.19-inch inner diameter tubing, and varying thickness of dry bentonite (typically 0.5 feet) and sand-pack (typically 1.25 feet).

SVE wells and the SVTU influent and effluent do not need to be purged prior to sampling if they are operating.

2.10.6 Data Collectors

Data for the project will be collected by Ahtna field staff or qualified subcontractors.

2.10.7 Reporting

Data from investigation activities will be included in the Quarterly Sites 2/12 Groundwater and Soil Vapor Monitoring and Treatment System Report submitted to project stakeholders.

2.10.8 Archiving

Paper copies of the reports will be submitted to the Fort Ord Administrative Record, USACE, and regulatory agencies. Electronic media (e.g., CD) or online storage (e.g., Portable Document Format [PDF]) will be used to permanently archive data and reports. Final reports will be archived in the Fort Ord Data Integration System (FODIS).

2.11 Measurement Performance Criteria Tables (Worksheet #12)

The data quality indicators (DQIs), measurement performance criteria (MPC), and QC sample and/or activity used to assess the measurement performance for both the sampling and analytical measurement systems are included. QC samples will be prepared at the frequency specified below:

- **Method Blank (MB):** One per every batch or group of up to 20 samples extracted and analyzed sequentially on an instrument.
- **Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD):** One LCS/LCSD pair per batch or group of up to 20 samples extracted and analyzed sequentially on an instrument. All target analytes shall be spiked at a known concentration.
- **Surrogate:** Surrogate compounds are similar in chemical composition and behavior to the analytes of interest, but are not normally found in environmental samples. Surrogates will be added to all samples analyzed in accordance with EPA Test Method TO-15.
- **Laboratory Duplicate:** One laboratory duplicate per batch or group of up to 20 samples analyzed sequentially on an instrument.
- **Field duplicates:** Duplicate samples shall be collected during soil gas sampling at a frequency of 10 percent (%) per quarterly event. Duplicate samples shall be collected at the same time as the original sample using a duplicate “T” sample train to establish even flow of air to both SUMMA canisters. Duplicate samples should be analyzed for acceptance criteria of $\leq 30\%$ relative percent difference (RPD). Samples not meeting the acceptance criteria shall indicate an audit of field procedures if necessary to refine sampling precision.
- **Below Reporting Limit:** Results reported between the LOD and LOQ should be reported with an estimated result “J” data qualifier applied by the laboratory for all results between the LOD and LOQ.

2.11.1 VOCs

Matrix: Soil Gas

Analytical Group: EPA Method TO-15

Sampling Procedure	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S) Analytical (A) or Both (S&A)
SOPs 2 and 5	Representativeness	Presence or Absence of Leak Check Compounds	Leak Check Compound on sample containers	S
	Precision - Overall	≤ 30% RPD	Field Duplicate	S&A
	Precision – Lab	≤ 30% RPD	Lab Duplicate	A
	Bias	Laboratory in-house limits	Surrogates	A
	Accuracy/Precision	Refer to Worksheet #28	LCS/LCSD	A
	Accuracy/Bias/Contamination	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.	MB	A
	Analytical Completeness	90%	Evaluation of number of usable results out of total number of results reported	S&A
Field Completeness	95%	Evaluation of number of samples collected out of number of samples planned	S	

Notes:

A = analytical

LCS = laboratory control samples

LCSD = laboratory control sample duplicate

LOQ = limit of quantitation

DQI = Data Quality Indicator

MB = method blank

N/A = not applicable

RPD = relative percent difference

S = sampling

S&A = sampling and analytical

SOP = standard operating procedure

2.12 Secondary Data Criteria and Limitations (Worksheet #13)

Secondary data and information that will be used, including originating sources, are identified below. How the secondary data will be used and the limitations on their uses are specified. Data from these documents will be utilized as appropriate.

Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation/collection dates)	How Data Will be Used	Limitations on Data Use
USACE – Final Soil Gas Investigation Interim Report, June 2013	USACE, soil gas results at temporary probes five feet bgs, collected 2012-2013	Data will be used to evaluate magnitude and extent of soil gas COC concentrations and historical trends	None
AES – Final Remedial Investigation/Feasibility Study Addendum at Sites 2 and 12, February 2015	AES, soil gas results at permanent probes 10 to approximately 70 feet bgs, collected 2013-2014	Data will be used to evaluate magnitude and extent of soil gas and groundwater COC concentrations and historical trends	None
Sites 2/12 Quarterly and Annual Groundwater and Soil Gas Monitoring and Treatment System Reports	Ahtna, groundwater and soil gas wells sample results, quarterly data collection ongoing	Data will be used to evaluate magnitude and extent of soil gas and groundwater COC concentrations and historical trends	None

2.13 Summary of Project Tasks (Worksheet #14)

Sampling Tasks:
1. SVETS, soil gas as described in Worksheet #17
Analysis Tasks:
1. See Worksheet #15.
Quality Control Tasks:
1. Implement three phase QC process in Worksheet #31 to verify SOPs (Attachment A) for collection of samples, packing and transport, and post-field processing prior to analysis are being followed. Collect and submit QC samples as described in Worksheet #20. Analyses to be performed in accordance with this QAPP, the guidance provided in the published methods and analytical laboratory SOPs.
Secondary Data:
1. See Worksheet #13.

Data Management Tasks:
<ol style="list-style-type: none">1. Analytical data from the laboratory for each sample will be tracked, reviewed, and loaded into the Ahtna database and the FODIS database.2. Following receipt of analytical data packages, the data is validated and a qualifier file is created. The data qualifier file is verified against the hardcopy data validation reports at a rate of 10%. Once verified, the data qualifier file is loaded into the Ahtna database, the FODIS database, and data uploaded to the GeoTracker database for Sites 2/12 (Global ID: DOD100204800).
Documentation and Records:
<ol style="list-style-type: none">1. Sampling methods, times, field measurements, observations, and assessments will be documented in field notes or sampling forms. Chain of custody forms and courier/transportation bills will be prepared and retained in project files for each sample. A copy of laboratory records will be included in the final report. PDF copies of all validated laboratory data and electronic data deliverables (EDDs) will be delivered to USACE.
Assessment/Audit Tasks:
<ol style="list-style-type: none">1. Sampling activities will be evaluated for compliance with the applicable SOP by the Field Supervisor or Project Chemist.2. Field and Laboratory audits may be performed by the Project or Program Chemist.
Data Review Tasks:
Following data review performed by the analytical laboratory, 100% of the sample results will be subject to the equivalent of EPA Stage 2B review and 10% will be subject to the equivalent of EPA Stage 4 raw data review as described in Worksheets #34 through #37. The review will assess compliance with the requirements of this QAPP, the guidance provided in the QSM Version 5.3 (DoD, 2019), <i>General Data Validation Guidelines, Environmental Data Quality Workgroup, Revision 1</i> (DoD, 2019), and published analytical methods of analysis. The findings of the data review and validation will be used to assess the usability of the data for supporting project decisions, and to implement corrective actions with the laboratory, if appropriate. Data qualifiers will be applied to sample results as needed based on the findings of the data validation and uploaded with the analytical data to the Ahtna database, FODIS, and GeoTracker.

2.14 Reference Limits and Evaluation Table (Worksheet #15)

Matrix: Soil Gas (analyzed by TO-15 5&20)⁸

Analyte	Soil Gas Screening Level ¹ (SG-SL) $\mu\text{g}/\text{m}^3$	SG-SL Reference	Soil Gas Cleanup Level ² (SGCL) $\mu\text{g}/\text{m}^3$	Laboratory Limits ³		
				DL ($\mu\text{g}/\text{m}^3$)	LOD ⁴ ($\mu\text{g}/\text{m}^3$)	LOQ ($\mu\text{g}/\text{m}^3$)
Tetrachloroethene	603	CHHSL	1,800	11.01	20	34
Trichloroethene	888	Calculated	1,000	5.66	16	27

Matrix: Soil Gas at SVTU Influent and Treated Soil Gas at SVTU Effluent (analyzed by TO-15 Low-Level)⁹

Analyte	Discharge Limit Compliance (total VOCs)			Laboratory Limits ³		
	lbs/day	HI	Risk	DL ($\mu\text{g}/\text{m}^3$)	LOD ⁴ ($\mu\text{g}/\text{m}^3$)	LOQ ($\mu\text{g}/\text{m}^3$)
Tetrachloroethene	<25	<1	<1x10 ⁻⁵	0.32	0.54	0.68
Trichloroethene				0.07	0.43	0.54

Notes:

$\mu\text{g}/\text{m}^3$ = micrograms per cubic meter

Calculated = see RI/FS Addendum (AES, 2015)

CHHSL = California Human Health Screening Levels Table 3: Soil-Gas Screening Numbers for Volatile Chemicals below Buildings Constructed without Engineered Fill below Sub-slab Gravel (September 23, 2010; <https://oehha.ca.gov/chhsltable>).

DL = detection limit

LOD = Limit of Detection

LOQ = Limit of Quantitation

$\mu\text{g}/\text{m}^3$ = microgram per cubic meter

¹SG-SL data from the RI/FS Addendum (AES, 2015). SG-SLs are conservative and based on a 5-foot sample depth.

²SGCLs are based on soil gas as a source of groundwater contamination and are presented in the RI/FS Addendum (AES, 2015) and ESD No. 1 (Army, 2016).

³Laboratory limits listed are base values. The final laboratory limits are determined by applying a dilution factor calculated from the SUMMA[®] canister pressurization measured upon receipt by the laboratory (see Attachment A, SOP 1).

⁴The LODs listed are Eurofins instrument-specific.

⁸ See Worksheet #19 for descriptions of modified TO-15 methods.

⁹ There are no chemical-specific screening levels for treated soil gas at the SVTU effluent; discharge requirements are defined in Worksheet #10, Section 2.9.5.

2.15 Project Schedule/Timeline (Worksheet #16)

A general soil gas sampling schedule is shown in Table 2. The schedule will be updated with annual QAPP updates. Deliverables associated with soil gas and SVETS sampling are the quarterly and annual Sites 2/12 Groundwater and Soil Gas Monitoring and Treatment System Reports. Quarterly reports are issued within 60 days after completion of sampling for the first, second and fourth calendar quarters. Draft annual reports are issued within 90 days after completion of sampling for the third calendar quarter.

3.0 Measurement and Data Acquisition

3.1 Sampling Design/Rationale (Worksheet #17)

A total of 167 permanent soil gas probes were installed at Site 12 in 24 locations with seven nested probes at each location.¹⁰ The nested probes were installed at approximately 10-foot intervals from 10 feet bgs to a depth of approximately 70 feet bgs (Figure 1 and Table 1).¹¹ The installed soil gas probe locations were selected to delineate the PCE and TCE soil gas plumes identified initially in 1992 (HLA, 1995) and expanded upon the initial soil gas investigations (USACE, 2013). All 167 soil gas probes were sampled during the Remedial Investigation (RI) Addendum at Sites 2/12 and quarterly sampling was initiated in 2015.¹² Two nested probe locations (SG-12-10 and SG-12-21; 14 probes total) were decommissioned in 2016. Five other probes (SG-12-06-70, SG-12-07-10, SG-12-12-50, SG-12-13-70, and SG-12-17-75) are no longer functional and cannot be sampled due to an obstruction or the screen interval becoming submerged in groundwater. The sampling design rationale for soil gas probes is described in detail in the *Final Work Plan, Remedial Investigation/Feasibility Study Addendum at Sites 2 and 12* (AES, 2013).

Ten permanent SVE wells were installed during pilot study construction and SVETS construction. The SVE pilot study well locations were selected to provide data to support design and construction of the full-scale SVETS. The SVETS well locations were selected to maximize COC removal from the vadose zone based on the results of the pilot study. The SVTU was installed to apply vacuum to the SVE wells and process extracted soil gas for treatment through GAC. The sampling design rationale for SVE wells and the SVTU is described in detail in the RAWP Addendum (AEI, 2015).

All samples will be analyzed for TCE and PCE by EPA Method TO-15. The sample analytical results will be assessed via the analytic approach in Worksheet #10 to determine:

- The lateral and vertical soil gas plume extent;
- Modifications to the sampling design;
- Discharge limit compliance;
- The schedule for GAC change-outs; and
- Site closure.

Site 12 soil gas probes and SVETS sampling locations are listed in Worksheet #18. All 167 soil gas probes were sampled during the RI Addendum and the First Quarter 2015 soil gas monitoring event; however, in subsequent events the locations to be sampled either quarterly or annually are determined through application of the analytic approach presented in Worksheet #10 (Section 2.9.5) to the soil gas analytical results from the previous monitoring event. The quarterly and annual events also include 10% duplicate soil gas sampling. The initial SVETS sampling schedule after startup of the SVETS is described in RAWP Addendum (AEI, 2015), with subsequent sampling to be determined through application of the analytic

¹⁰ One location has 6 nested probes (SG-12-09) due to a shallower saturated soil depth.

¹¹ Several locations have different depths of the deepest two probes due to shallower or deeper saturated soil depth.

¹² RI soil gas analytical results are presented in the RI/FS Addendum report (AES, 2015).

approach presented in Worksheet #10 (Section 2.9.5) to analytical results from the previous sampling events. Based on sampling results and implementation of the analytic approach to date, the number of soil gas samples collected annually is summarized in Table 3.

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

Sample Location ID	Soil Gas Probe ID	Analytical Method for PCE & TCE	Sampling Methods/SOPs
SG-12-01	SG-12-01-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-01-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-01-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-01-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-01-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-01-58	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-01-65	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-02	SG-12-02-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-02-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-02-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-02-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-02-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-02-57	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-02-65	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-03	SG-12-03-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-03-20.5	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-03-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-03-39.5	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-03-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-03-58	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-03-65	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-04	SG-12-04-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-04-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-04-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-04-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-04-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-04-58	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-04-65	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-05	SG-12-05-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-05-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-05-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-05-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-05-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-05-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-05-70	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-06	SG-12-06-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-06-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7

Sample Location ID	Soil Gas Probe ID	Analytical Method for PCE & TCE	Sampling Methods/SOPs
	SG-12-06-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-06-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-06-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-06-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-06-70	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-07	SG-12-07-10	NA	See note *
	SG-12-07-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-07-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-07-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-07-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-07-57.5	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-07-65	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-08	SG-12-08-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-08-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-08-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-08-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-08-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-08-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-08-70	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-09	SG-12-09-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-09-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-09-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-09-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-09-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-09-59	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-11	SG-12-11-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-11-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-11-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-11-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-11-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-11-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-11-70	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-12	SG-12-12-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-12-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-12-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-12-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-12-50	NA	See note *
	SG-12-12-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7

Sample Location ID	Soil Gas Probe ID	Analytical Method for PCE & TCE	Sampling Methods/SOPs
	SG-12-12-70	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-13	SG-12-13-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-13-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-13-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-13-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-13-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-13-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-13-70	NA	See note *
SG-12-14	SG-12-14-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-14-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-14-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-14-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-14-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-14-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-14-70	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-15	SG-12-15-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-15-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-15-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-15-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-15-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-15-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-15-70	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-16	SG-12-16-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-16-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-16-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-16-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-16-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-16-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-16-70	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-17	SG-12-17-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-17-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-17-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-17-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-17-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-17-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-17-75	NA	See note *
SG-12-18	SG-12-18-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-18-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7

Sample Location ID	Soil Gas Probe ID	Analytical Method for PCE & TCE	Sampling Methods/SOPs
	SG-12-18-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-18-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-18-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-18-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-18-70	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-19	SG-12-19-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-19-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-19-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-19-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-19-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-19-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-19-70	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-20	SG-12-20-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-20-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-20-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-20-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-20-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-20-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-20-70	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-22	SG-12-22-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-22-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-22-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-22-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-22-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-22-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-22-70	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-23	SG-12-23-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-23-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-23-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-23-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-23-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-23-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-23-70	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
SG-12-24	SG-12-24-10	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-24-20	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-24-30	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-24-40	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-24-50	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7

Sample Location ID	Soil Gas Probe ID	Analytical Method for PCE & TCE	Sampling Methods/SOPs
	SG-12-24-60	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
	SG-12-24-70	TO-15 (5&20)	SUMMA®/SOPs 1-4 and 7
VE-12-01	N/A	TO-15 (5&20)	SUMMA®/SOPs 1, 5 and 7
VE-12-02	N/A	TO-15 (5&20)	SUMMA®/SOPs 1, 5 and 7
VE-12-03	N/A	TO-15 (5&20)	SUMMA®/SOPs 1, 5 and 7
VE-12-04	N/A	TO-15 (5&20)	SUMMA®/SOPs 1, 5 and 7
VE-12-05	N/A	TO-15 (5&20)	SUMMA®/SOPs 1, 5 and 7
VE-12-06	N/A	TO-15 (5&20)	SUMMA®/SOPs 1, 5 and 7
VE-12-07	N/A	TO-15 (5&20)	SUMMA®/SOPs 1, 5 and 7
VE-12-08	N/A	TO-15 (5&20)	SUMMA®/SOPs 1, 5 and 7
VE-12-09	N/A	TO-15 (5&20)	SUMMA®/SOPs 1, 5 and 7
VE-12-10	N/A	TO-15 (5&20)	SUMMA®/SOPs 1, 5 and 7
SVTU-212-INF	N/A	TO-15 (Low-Level)	SUMMA®/SOPs 1 and 5-7
SVTU-212-MID	N/A	TO-15 (Low-Level)	SUMMA®/SOPs 1 and 5-7
SVTU-212-EFF	N/A	TO-15 (Low-Level)	SUMMA®/SOPs 1 and 5-7

Notes:

*No longer functional and cannot be sampled due to an obstruction or the screen interval becoming submerged in groundwater.

Sampling schedule will be determined based on soil gas data analysis compared to the established analytic approach (Worksheet #10, Section 2.9.5).

SG = soil gas [probe]

VE = vapor extraction [well]

SVTU = soil vapor treatment unit

SVTU-212-INF = SVTU influent sampling location prior to treatment of extracted soil gas by GAC

SVTU-212-MID = SVTU sampling location between lead and lag GAC vessels (i.e., lead GAC vessel effluent and lag GAC vessel influent)

SVTU-212-EFF = SVTU effluent sampling location after GAC treatment

N/A = not applicable

3.3 Analytical Requirements – Sample Volumes/Preservation Requirements Table (Worksheet #19)

The analytical and preparation method/SOP and associated sample volume, container specifications, preservation requirements, and maximum holding time are listed.

Analysis	Method	Matrix	Container	Preservation	Holding Time (from sample date)
VOCs	EPA TO-15 (5&20) ¹	Soil Gas ³	1-liter SUMMA canister	None	30 days
VOCs	EPA TO-15 (Low-Level) ²	Soil Gas ⁴	1-liter SUMMA canister	None	30 days

Notes:

¹TO-15 (5&20): This method involves full scan gas chromatograph/mass spectrometer (GC/MS) analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for VOCs using EPA Method TO-14A/TO-15 protocols. An aliquot of up to 50 milliliters of air is withdrawn from the canister utilizing a volumetric syringe or mass flow controller. This volume is loaded onto a hydrophobic multi-bed sorbent trap to remove water and carbon dioxide and to concentrate the vapor sample. The focused sample is then flash-heated to sweep adsorbed VOCs onto a secondary trap for further concentration and/or onto a GC/MS for separation and detection. The 5&20 analytical configuration has base LOQs of approximately 34 µg/m³ for PCE and 27 µg/m³ for TCE. The methodology is described in more detail in Eurofins SOP #91 in Attachment A.

²TO-15 (Low-Level): This method involves full scan GC/MS analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for VOCs using EPA Method TO-14A/TO-15 protocols. An aliquot of up to 400 milliliters of air is withdrawn from the canister utilizing a volumetric syringe, volumetric loop, or mass flow controller. This volume is loaded onto a hydrophobic multi-bed sorbent trap to remove water and carbon dioxide and to concentrate the vapor sample. The focused sample is then flash-heated to sweep adsorbed VOCs onto a GC/MS for separation and detection. Compounds are detected using an MS operating in full scan mode. The Low-Level analytical configuration has base LOQs of approximately 0.68 µg/m³ for PCE and 0.54 µg/m³ for TCE. The methodology is described in more detail in Eurofins SOP #83 in Attachment A.

³Collected from soil gas probes and SVE wells.

⁴Collected from the SVTU influent and effluent.

3.4 Field Quality Control Sample Summary (Worksheet #20)

Matrix	Frequency -Field Duplicate Samples	Frequency – Trip Blanks	Frequency – Field Blanks
Soil Gas	10% per sampling year (4Q through 3Q)	N/A	N/A

Notes:

N/A = not applicable

Q = quarter

3.5 Project Field Sampling SOP Table (Worksheet #21)

SOPs associated with project sampling are listed. Copies of the SOPs are included in Attachment A. Sampling SOPs are numbered in the “Reference Number” column. The Reference Number is used throughout the QAPP to refer to a specific SOP.

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Sample Type	Modified for Project Work? (Y/N)	Comments
1	Guide to Air Sampling, June 27, 2014	Eurofins	Soil Gas	No	
2	Soil Gas Sampling, September 25, 2015	DTSC	Soil Gas	No	
3	Helium Shroud Spec Sheet, September 30, 2014	Eurofins	Soil Gas	No	
4	SVE Treatment System Sampling, September 25, 2015	Ahtna	Soil Gas	No	
5	DoD Environmental Field Sampling Handbook, Revision 1.0, April 2013, Chapter 3. “Common Sampling Procedures”	USACE	All	No	Includes sample documentation, packaging, shipping, and chain of custody
6	SVE Treatment System Flow Meter Use	Ahtna	NA	No	

3.6 Field Equipment Calibration, Maintenance, Testing, and Inspection Table (Worksheet #22)

Field equipment will be calibrated, maintained, tested, and inspected per the SOPs in Attachment A or manufacturer instructions. The following field equipment may be used during soil gas sampling and SVETS sampling:

Site Setup

- Personal protective equipment including high visibility vest, sun/wind protection (if necessary), steel-toe boots and nitrile gloves
- Tools to open soil gas probe vault if it is flush mount ($\frac{3}{4}$ -inch or $\frac{15}{16}$ -inch wrench)
- Traffic cones for delineation of exclusion zone

Soil Gas Probe Purging

- Vacuum pump
- Vacuum gauge
- Tubing (silicone and nylon)

Soil Gas Probe Integrity Test (Helium Test)

- Helium compressed gas cylinder (can be provided by laboratory)
- Helium cylinder regulator and tubing with connection to shroud (can be provided by laboratory)
- In-line helium detector (can be provided by laboratory)
- Shroud helium detector (can be provided by laboratory)
- Shroud assembly (provided by laboratory)
- Plastic sheeting to cover shroud
- Weights to hold shroud down

Sampling

- Sample containers – 1-liter SUMMA canisters (provided by laboratory)
- Flow regulator – 100-200 milliliters per minute (part of shroud sampling manifold assembly)
- Sample manifold (T-manifold for duplicate samples) (provided by laboratory)
- Vacuum gauge
- Tools ($\frac{9}{16}$ -inch wrench to remove canister caps and attach gauges, tubing, and/or flow regulators, tubing cutter)
- Tubing
 - $\frac{1}{4}$ -inch Nylaflow (nylon) or Teflon, approximately 1 foot per probe and
 - $\frac{3}{8}$ -inch silicone, approximately 3 inches per probe
- Polytetrafluoroethylene (Teflon) thread seal tape
- Small zip ties
- Ferrules and $\frac{1}{4}$ -inch compression caps (extras in case laboratory-provided ones do not work)

3.7 Analytical SOP References Table (Worksheet #23)

Laboratory SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Variance to QSM	Modified for Project Work?
SOP #91	EPA Method TO-14A/TO-15 Volatile Organic Compounds (5&20 ppbv), November 4, 2020, Revision 17	Definitive	Soil Gas VOCs	GC/MS	Eurofins	Yes	No
SOP #83	EPA Method TO-14A/TO-15 Volatile Organic Compounds (Low-Level), November 4, 2020, Revision 22	Definitive	SVTU Influent/ Effluent VOCs	GC/MS	Eurofins	Yes	No

Laboratory SOPs #83 and #91 are in Attachment A. Additional laboratory SOPs, LOD studies, in-house sample custody procedures, quality control acceptance limits, and the Quality Assurance Manual are maintained on-site by the laboratory and are included in Attachment A. Laboratories used for sample analysis maintain DoD Environmental Laboratory Accreditation Program (ELAP) certification (Attachment E). Laboratory SOPs are subject to revision and updates. For the duration of the project, the laboratory will use the most current version of the SOP at the time of analysis. Laboratory audits are performed as part of the DoD ELAP certification process and are beyond the scope of this QAPP. In the event a problem is encountered in the laboratory, the Army may request a special audit.

3.8 Analytical Instrument Calibration QC Table (Worksheet #24)

Analytical Group/Test Method: VOCs by EPA Method TO-15

Matrix: Soil Gas

SOP Reference: #83 and #91 (Attachment A)

QSM Version 5.3 Reference: Table B-21

Procedure	Frequency	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action
Demonstrate acceptable GC/MS performance	Prior to the analysis of any calibration standards, blanks, and samples, the GC/MS must meet the mass spectral abundance criteria for 4-bromofluorobenzene (BFB). BFB must be checked at the beginning of each 24-hour period.	BFB mass spectral ion abundance ratios must meet the following acceptance criteria: Mass Ion Abundance Criteria 50 8.0 - 40% m/e 95 75 30.0 – 66.0% m/e 95 95 Base Peak, 100% Relative Abundance 96 5.0 – 9.0% m/e 95 173 <2.0% of m/e 174 174 50.0 – 120.0% m/e 95 175 4.0 – 9.0% of m/e 174 176 93.0 – 101.0% of m/e 174 177 5.0 to 9.0% of m/e 176	If BFB acceptance criteria are not met, the instrument must be retuned or the ion source cleaned and/or the column changed.	GC/MS Analyst
Initial Calibration (ICAL)	At instrument setup, prior to sample analysis; after corrective action (e.g., ion source cleaning or repair, column replacement, etc.), and a CCV if ICAL passes after 24 hour period (per EPA Method TO-15 10.5.2).	Minimum of five points. % RSD ≤ 30 for the RRF of all target analytes. RRT for each target compound at each calibration level must be within 0.06 RRT units of the mean RRT for the compound (per EPA Method TO-15 10.5.5.2). The lowest calibration standard concentration at or below the LOQ.	Correct the problem then repeat initial calibration curve.	GC/MS Analyst

Procedure	Frequency	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action
Retention time (RT) window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	RT for blanks and samples must be within ± 0.33 min of the RT in the CCV and within $\pm 40\%$ of the area counts of the daily CCV internal standards. Position shall be set using the mid-point standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	Correct problem then repeat.	GC/MS Analyst
Initial Calibration Verification (ICV)	Once after the ICAL and before field samples.	Recoveries of compounds must be 70-130%. ICV will be prepared from a source independent of the source of the initial calibration standards.	Check the system and reanalyze the standard. Reprepare the standard if necessary, to determine the source of error. Recalibrate the instrument if the primary standard is found to be in error.	GC/MS Analyst
Continuing Calibration Verification (CCV)	Prior to the analysis of samples and blanks, but after tuning criteria have been met; after every 24 hours of analysis time; and at the end of the analytical batch.	Concentration the same as the mid-point calibration standard (or lower). All reported analytes within $\pm 30\%$ of true value. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Check system; two consecutive passing CCVs must be analyzed. Re-prepare standard if necessary. Recalibrate system if the criterion cannot be met. Reanalyze affected samples.	GC/MS Analyst

Notes:

BFB = 4-bromofluorobenzene	Rel = relative
m/e (or m/z) = mass/charge ratio	RRF = relative response factor
COD = coefficient of determination	RSD = relative standard deviation
N/A = not applicable	ICV = initial calibration verification
GC/MS = gas chromatography/mass spectrometry	CCV = Continuing Calibration Verification
ICAL = initial calibration	RT = retention time
RRT = relative retention time	

3.9 Analytical Instrument and Equipment Maintenance, Testing and Inspection Table (Worksheet #25)

Instrument/ Equipment	Maintenance Activity ¹	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP ² Reference
GC/MS VOC	Change Trap	Analyze ICV, CCV, or sensitivity check	Daily	When responses start to drop	ICV, CCV, or sensitivity check passes criteria	Re-bake trap, replace trap, reanalyze ICV, CCV, or sensitivity check, recalibrate	Analyst or Department Manager	#83 #91
	Backflush Purge and Trap Lines	Analyze ICV, CCV, or sensitivity check	Daily	ICV, CCV, or sensitivity check will not pass, high level sample analyzed	ICV, CCV, or sensitivity check passes criteria, Blank clean	Backflush lines again, replace lines, recalibrate	Analyst or Department Manager	#83 #91
	Change septa and liner, clean injection port, clip column	Analyze ICV, CCV, or sensitivity check	Daily	After high level sample analyzed	ICV, CCV, or sensitivity check passes criteria	Re-inspect injection port, cut additional column, reanalyze ICV, CCV, or sensitivity check, recalibrate instrument	Analyst or Department Manager	#83 #91

Notes:

¹When appropriate per method

²Laboratory SOPs are subject to revision and updates during the duration of the project. The laboratory will use the most current revision of the SOP at the time of analysis (Attachment A).

ICV = initial calibration verification

CCV = continuing calibration verification

GC/MS = gas chromatography/mass spectrometry

VOC = volatile organic compound

3.10 Sample Handling System (Worksheet #26)

This worksheet identifies components of the project-specific sample handling system. Personnel and their organizational affiliations, who are primarily responsible for ensuring proper handling, custody, and storage of field samples from the time of collection, to laboratory delivery, to final sample disposal, are listed.

Sample Collection, Packaging, and Shipment
Sample Collection (Personnel/Organization): Field Technician/Ahtna or Subcontractor
Sample Packaging (Personnel/Organization): Field Technician/Ahtna or Subcontractor
Coordination of Shipment (Personnel/Organization): Field Technician/Ahtna or Subcontractor
Type of Shipment/Carrier: Lab Courier, Federal Express or other overnight shipment
Sample Receipt and Analysis
Sample Receipt (Personnel/Organization): Laboratory Receiving Personnel
Sample Custody and Storage (Personnel/Organization): Laboratory Sample Custodian
Sample Preparation (Personnel/Organization): Laboratory Personnel
Sample Determinative Analysis (Personnel/Organization): Laboratory Personnel
Sample Archiving
Field Sample Storage: Released for cleaning after analyses
Sample Disposal
Personnel/Organization: Laboratory Personnel
Released for cleaning after analyses

3.11 Sample Custody Requirements (Worksheet #27)

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory).
Samples will be collected in laboratory-provided certified containers using methods described in Worksheet #19. Procedures for sample packaging, shipment, and delivery to the laboratory are included in SOP 5.
Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):
Samples will be received and logged into the laboratory information management systems (LIMS) for analysis as requested on the chain of custody form. Samples will be archived following analysis as described in Worksheet #26. Samples will be disposed of following archive period in accordance with state and local requirements.
Sample Identification Procedures:
Sample identification procedures will be performed in accordance with the QSM Version 5.3 (DoD, 2019) and Worksheet #29.
Chain of Custody Procedures:
Chain of custody procedures will be performed in accordance with DoD QSM 5.3 (DoD, 2019) and Worksheet #29.

3.12 Analytical QC Sample Table (Worksheet #28)

Matrix	Soil Gas		
Analytical Group	VOCs	Sampling Reference	See Worksheet #21
Concentration Level	All	Analytical Method	TO-15
Field Sampling Organization	Ahtna	Sampler	Ahtna
No. of Sample Locations	166	Analytical Organization	Eurofins

QC Sample:	Frequency/ Number	Acceptance Limits	Corrective Action (CA)	Person Responsible for Corrective Action	Data Quality Indicator
Laboratory Control Samples (LCS) containing analytes of interest and surrogate compounds	Once per preparatory batch of up to 20 samples.	QSM 5.3 Appendix C Limits will be used for batch control.	Results may not be reported without a valid LCS. Must contain all surrogates and all analytes to be reported. Check the system and reanalyze. Reprepare if necessary to determine the source of error. Recalibrate the instrument if the error is found.	Laboratory Manager/ Analyst	Laboratory Accuracy/Bias
Laboratory Control Sample Duplicate (LCSD); Initial and Closing CCV can serve as the LCS/LCSD	One per analytical batch.	LCS/LCSD - Use DoD QSM 5.3 published limits Table C-43; RPD <30% Initial and Closing CCV - see Worksheet #24	Investigate the cause and perform maintenance as required. If instrument maintenance is required, calibrate as needed.	Laboratory Manager/ Analyst	Laboratory Accuracy/ Precision
Sample Dilution	If the on-column concentration of compounds in any sample exceeds the initial calibration range, a dilution of	N/A	N/A	Laboratory Manager/ Analyst	N/A

QC Sample:	Frequency/ Number	Acceptance Limits	Corrective Action (CA)	Person Responsible for Corrective Action	Data Quality Indicator
	the sample must be analyzed.				
Method Blank (MB)	After analysis of standards and prior to sample analysis, or when contamination is present.	No analytes detected $> \frac{1}{2}$ LOQ or $> \frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit, whichever is greater. Common contaminants must not be detected $>$ LOQ.	Correct problem. If required, reprepare and reanalyze MB and all samples processed with the contaminated blank. Results may not be reported without a valid MB. Flagging is only appropriate in cases where the samples cannot be reanalyzed.	Laboratory Manager/ Analyst	Laboratory Accuracy/Bias/ Contamination
Surrogate Spike	All field and QC samples.	Current in-house laboratory limits (limits as of June 2018 listed below): 1,2-Dichloroethane-d4 (74-130%) Toluene-d8 (87-113%) 4-Bromofluorobenzene (76-119%) While surrogate compounds routinely demonstrate narrow recovery ranges, there are times where the surrogate recoveries in the samples are wider than the historical acceptance limits but within +/-30% of 100% for EPA method TO-15. In this case, the laboratory will default to the SOP surrogate control limits of 70-130%. Quantification achieved using a multipoint calibration at a single concentration, analogous to internal standards, detection limits (DLs) and LOQs are not established.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with a surrogate is present, reanalysis may not be necessary.	Laboratory Manager/ Analyst	N/A

QC Sample:	Frequency/ Number	Acceptance Limits	Corrective Action (CA)	Person Responsible for Corrective Action	Data Quality Indicator
Internal Standards	Every field sample, standard and QC sample.	RT for blanks and samples must be within ± 0.33 minutes of the RT in the CCV and within ± 40 percent of the area counts of the daily CCV Internal Standards.	For blanks: inspect the system and reanalyze the blank. For samples: reanalyze the sample. If the Internal Standards are within limits in the reanalysis, report the second analysis. If Internal Standards are out-of-limits a second time, dilute the sample until Internal Standards are within acceptance limits and narrate.	Laboratory Manager/ Analyst	N/A

Notes:

SOP = standard operating procedure
 BFB = 4-bromofluorobenzene
 N/A = not applicable
 QC = quality control
 LOQ = limit of quantitation

MB = method blank
 RPD = relative percent difference (absolute value of difference of two sample results divided by average result)
 LCS/LCSD = laboratory control sample/laboratory control sample duplicate
 RT = retention time

3.13 Project Documentation and Records (Worksheet #29)

Project data and information will be documented, tracked, and managed in a manner that ensures the data maintains integrity, defensibility, and retrievability. Project records will be generated from various aspects of the project, including 1) Sample Collection and Field Measurement Records, 2) Analytical Records, and 3) Data Assessment Records. Project data and information are stored in the Fort Ord Administrative Record located at Building 4463 Room 101, Gigling Road, Seaside, California. The Administrative Record is managed by the Army and will be maintained until site closure, at which time disposition of site records will be determined by the Army.

3.13.1 Sample Collection and Field Measurement Records

At a minimum, the following documentation will be used for sample collection and field measurement activities. Examples of field forms are presented in Attachment B.

- Field Activity Daily Logbook – A bound field activity daily logbook (FADL) with sequentially numbered pages will be used for field documentation of key sampling and analytical activities associated with Sites 2/12. The FADL will include:
 - Name and Company of sampling technician
 - Date, time, and location of sample collection
 - Site observations and remarks related to sampling activities
 - Field equipment calibration documentation
- Sampling Forms – are used to record collection of samples. The sampling forms will include:
 - Name of sampling technician
 - Date and time of sample collection
 - SUMMA canister volume (e.g., 1-liter)
 - SUMMA canister identification number
 - Flowmeter identification number
 - SUMMA beginning of sample vacuum reading
 - SUMMA end of sample vacuum reading
 - Depth of sample collection
 - Analysis requested
 - QC samples collected at the sampling station
- Sample Labels – Sample labels will be affixed to each sample container upon collection and prior to transfer to the laboratory. Each sample will be assigned a unique sample identification number. The sample label will include:
 - Project name, number, and location
 - Site name
 - Name of collector
 - Date and time of collection
 - Sample identification number
- Chain of Custody Forms – A chain of custody form will be completed for every sample collected and submitted to the analytical laboratory to document custody of the sample from the time of

collection to receipt at the laboratory. Chain of custody forms will be completed in duplicate (at a minimum) so that one copy is sent to the Task Lead or designee and one copy accompanies the samples submitted to the analytical laboratory. The laboratory will send the Project Chemist or designee a copy of the completed chain of custody form along with a completed sample receipt form and completed login information within 24 hours of sample receipt and log in. The chain of custody will include:

- Name, number, and location of project
- Project Manager or “Report to” contact
- Name and signature of sample collector, sampler or recorder
- Date and time of sample collection
- Sample type/matrix
- Number of containers submitted
- SUMMA canister identification number
- Final SUMMA vacuum reading after sampling
- Flowmeter identification number
- Analyses requested and turnaround time requirements
- Signature trail of persons relinquishing and receiving samples
- Date and time of sample receipt

3.13.2 Analytical Records

The analytical laboratory will maintain and submit the records listed below as part of the data deliverable for each sample. These records together make up the Comprehensive Certificate of Analysis (CCA), which is a required deliverable to report results and is used in the data validation process. The final CCA will be reported by the laboratory, with the EDD, to Ahtna within 21 calendar days of sample receipt. The CCA information is included in the full data package as an electronic validation package. The CCA items are bookmarked within the PDF file for easy navigation. The CCA will include:

- A case narrative (on laboratory letterhead and signed by the laboratory manager or designee) that addresses instances where exceedances of QC limits occurred and provides explanations for these exceedances and the corrective actions implemented. Samples not meeting QC criteria will be identified. If matrix effects are proposed as a cause for QC exceedances, a detailed justification for this assertion, including a summary of relevant data, will be provided. The case narrative will also present any other information relevant to the interpretation and usability of the analytical data.
- Copy of the chain of custody form signed by a representative of the laboratory.
- A summary of detected concentrations of target compounds indexed by method and sample ID will be provided in each CCA.
- Sample/Cooler Receipt Forms documenting the general condition of the samples upon receipt, including temperature, sample preservation, and number of containers received as well as any discrepancies or issues.
- Sample preparation and analysis forms/logbooks.

- Tabulated data summary forms and raw data for field samples, QC samples, and standards. If manual integration is performed on project samples, raw data to include chromatographs from before and after manual integration is applied. The case narrative will also address the reason manual integration was performed on each affected sample.
- Date and times of sample receipt, extraction, and analysis.
- Report by the contract laboratory for each analytical method on each target analyte. For VOC analysis, detected results above the LOD but less than the LOQ will be reported by the laboratory as estimated values and will be qualified using a J-qualifier. Dates of extraction and analysis, dilution factors, LOQs and any appropriate data qualifiers will be reported for each analyte. Results associated with spike recoveries outside of acceptance criteria shall be designated as such.
- Report on MB data for all analytical methods and target analytes, including values between the LOQ and the LOD. Analytical results for each sample will be clearly associated with a given MB.
- Report on surrogate spike recoveries for all applicable methods, accompanied by the control limits. Samples re-extracted and reanalyzed because surrogate recoveries were not within limits will be reported if the reanalysis meets the criteria, or the original analysis will be reported if the reanalysis confirms initial results. Re-extracted and reanalyzed samples will be clearly designated as such.
- Laboratory control sample for all analytical methods and the specified target analytes, accompanied by the control limits as specified in the QAPP. Analytical results for each sample will be clearly associated with a given LCS. Re-extracted and reanalyzed laboratory duplicates will be clearly designated as such.
- Corrective action reports.
- Definitions of laboratory qualifiers.
- Initial and continuing calibration data, including sample injection records, for all analytical methods and target analytes, accompanied by the control limits as specified in the QAPP. Analytical results for each sample will be clearly associated with the initial, continuing and bracketing calibration standard results as appropriate. Injection records for sample analyses shall be included with the calibration data.
- Signatures for laboratory sign-off.

3.13.3 Raw Data Packages

Full data packages (i.e., including raw data) will be requested from the laboratory for all samples. Analytical results to be prepared as full data packages will be selected to represent the matrices, analyses, locations and temporal characteristics of interest for the project. Full data packages will be delivered with the CCA.

The following additional information provides a summary of required contents for the full data package:

- Internal standard area counts and RTs will be reported for all applicable methods and will be accompanied by the control limits.

- Gas chromatography/mass spectrometry (GC/MS) tuning data will be reported for all applicable methods and will be accompanied by the control limits.
- All raw data, including chromatograms and other instrument printouts, will be reported.
- Injection logs for all instruments used for analysis of project samples, indicating the date and time of analysis of project samples and associated QA/QC samples, will be reported.
- A summary of the QA/QC data, including data for MBs, surrogate recoveries, LCS/LCSD recoveries and RPDs, initial calibration data, continuing calibration data and field duplicate RPDs. QA/QC data will be accompanied by the QAPP-specified control limit data.
- RT windows will be provided for GC/MS analyses. The RT window for each analyte for both primary and confirmation analyses will be reported.
- Compound identification verification will be provided for GC/MS analyses. The RTs and concentrations of each analyte detected in the environmental and QC samples are to be reported.

3.13.4 Electronic Data Deliverables

EDDs will be generated by the laboratory using the format and data elements as specified in Attachment C. This attachment contains file specifications developed for USACE EDDs that support use of a contract compliance screening software program and an Automated Data Review (ADR) program. EDDs generated by the lab will include the ADR EDDs for Ahtna upload into the FODIS database and the California Electronic Data Format (EDF) EDD for Ahtna upload into the CCRWQCB's GeoTracker database for Sites 2/12 (Global ID: DOD100204800). The ADR EDD will also be uploaded into the Ahtna database for archival and reporting purposes.

3.14 Analytical Services Table (Worksheet #30)

All laboratories or organizations that will provide analytical services for the project are identified.

Matrix	Analytical Groups	Sample Locations	Laboratory/Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number)
Soil Gas/ Treated Soil Gas	PCE and TCE – EPA TO-15	See Worksheet #18	Eurofins Air Toxics, LLC 180 Blue Ravine Rd, Suite B Folsom, CA 95630 (800) 985-5955 Brian Whittaker Project Manager (916) 605-3355 BrianWhittaker@EurofinsUS.com	Other laboratories to be determined as needed

4.0 Assessment and Oversight

4.1 Planned Project Assessments Table (Worksheet #31)

Planned project assessments will be completed for the work conducted using the Three Phase Quality Control Process, which consists of the following:

- **Preparatory Phase:** Activities and assessments during the preparatory phase are conducted prior to the start of a definable feature of work and are performed to ensure technical requirements and work prerequisites are completed prior to the start of the feature of work. Discrepancies will be resolved and corrective actions implemented and verified prior to the start of work.
- **Initial Phase:** Activities and assessments during the initial phase are performed during the first day of the definable feature of work and are conducted to verify compliance with the specifications and requirements described in this QAPP and approved project plans and procedures. Discrepancies will be resolved and corrective actions implemented and verified prior to work proceeding.
- **Follow Up and Reporting Phase:** Activities and assessments performed during the follow up and reporting phase are conducted to verify continued compliance with project requirements and to verify project reports meet client and regulatory requirements.

An overview of the Three Phase Quality Control Process and related forms used to document the process are provided in Attachment D. The activities and assessments conducted during each phase of the Three Phase Quality Control Process are described below.

Worksheet #31 (Continued)

Assessment	Frequency	Person(s) Responsible for Performing Assessment	Assessment Mechanism	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing Corrective Actions	Person(s) Responsible for Monitoring Effectiveness of Corrective Action
Phase I – Preparatory Phase						
Have planning documents been prepared in accordance with statement of work, regulatory requirements, and contract requirements?	Prior to sampling startup	Derek Lieberman, Project Manager, Ahtna Bruce Wilcer, QC Manager, Ahtna	Quality Control Review of document by Project Manager and QC Manager.	Document author	Document author	Derek Lieberman, Project Manager, Ahtna
Have planning documents been read by appropriate project personnel (including subcontractors) before work is conducted?	Prior to sampling startup and with all new field staff prior to assignment	Derek Lieberman, Project Manager, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Documentation (e.g., sign-off form, note to file, email acknowledgment) that document has been read and requirements are understood.	Mark Fisler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Mark Fisler, Field Supervisor, Ahtna Brian Whittaker, Project Manager, Eurofins	Derek Lieberman, Project Manager, Ahtna
Has required preliminary work (e.g., clearance activities, permits, site access) been completed in accordance with the project plan?	Prior to sampling startup	Mark Fisler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Comparison of information obtained from preliminary work completion assessment as specified in the project planning document(s).	Mark Fisler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Mark Fisler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Derek Lieberman, Project Manager, Ahtna Bruce Wilcer, QC Manager, Ahtna
Are staff and subcontractors prepared to implement project activities according to planning documents?	Prior to sampling startup and with all new project staff prior to assignment	Derek Lieberman, Project Manager, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Review and discussion of planned activities prior to implementation.	Mark Fisler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Mark Fisler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Derek Lieberman, Project Manager, Ahtna Bruce Wilcer, QC Manager, Ahtna

Assessment	Frequency	Person(s) Responsible for Performing Assessment	Assessment Mechanism	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing Corrective Actions	Person(s) Responsible for Monitoring Effectiveness of Corrective Action
Is necessary field equipment available and in acceptable working order?	Prior to sampling startup	Mark Fisler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Compare field equipment list with planned activities. Compare field equipment calibration documentation with project goals specified in the QAPP.	Andrew Mauck, Soil Gas Monitoring Task Lead Mark Fisler, Field Supervisor, Ahtna	Mark Fisler, Field Supervisor, Ahtna Brian Whittaker, Project Manager, Eurofins	Derek Lieberman, Project Manager, Ahtna Bruce Wilcer, QC Manager, Ahtna
Phase II – Initial Phase						
Is work being performed according to project plans?	Beginning of project activity	Derek Lieberman, Project Manager, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Perform field audits. Laboratory audits are performed as part of the DoD ELAP certification process and are beyond the scope of this QAPP. In the event a problem is encountered with the laboratory, the Army may request a special audit.	Mark Fisler, Field Supervisor, Ahtna Brian Whittaker, Project Manager, Eurofins	Mark Fisler, Field Supervisor, Ahtna Brian Whittaker, Project Manager, Eurofins	Derek Lieberman, Project Manager, Ahtna Bruce Wilcer, QC Manager, Ahtna
Have necessary audits been performed?	Early phase of project	Derek Lieberman, Project Manager, Ahtna Bruce Wilcer, QC Manager, Ahtna	Review project phase and check to see if required audits have been satisfactorily completed.	Derek Lieberman, Project Manager, Ahtna	Derek Lieberman, Project Manager, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Derek Lieberman, Project Manager, Ahtna Tom Ghigliotto, Field Oversight Inspector, Chenega
Phase III – Follow up and Reporting Phase						
Have data reports been prepared in accordance with project plans?	Reporting phase of project	Teri Farrell-Bage, Data Manager, Ahtna Eric Schmidt, Project Chemist, Ahtna	Compare data reports to specifications detailed in planning documents.	Brian Whittaker, Project Manager, Eurofins	Brian Whittaker, Project Manager, Eurofins	Derek Lieberman, Project Manager, Ahtna Eric Schmidt, Project Chemist, Ahtna Bruce Wilcer, QC Manager, Ahtna

Assessment	Frequency	Person(s) Responsible for Performing Assessment	Assessment Mechanism	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing Corrective Actions	Person(s) Responsible for Monitoring Effectiveness of Corrective Action
Are reports adequate to meet client and regulatory agency requirements?	After draft report submittal or project completion	Derek Lieberman, Project Manager, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead Bruce Wilcer, QC Manager, Ahtna	Review client and regulatory comments and prepare response to comments and revised reports.	Document Authors	Document Authors	Derek Lieberman, Project Manager, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead
Have other definable features of work been completed in accordance with project requirements?	Ongoing during all phases of work and upon work completion	Derek Lieberman, Project Manager, Ahtna	Compare definable features of work with project requirements.	Derek Lieberman, Project Manager, Ahtna	Derek Lieberman, Project Manager, Ahtna	Derek Lieberman, Project Manager, Ahtna Bruce Wilcer, QC Manager, Ahtna
Are daily Contractor Quality Control Reports (CQCRs) being prepared according to contract requirements?	Ongoing throughout project	Derek Lieberman, Project Manager, Ahtna	Review daily CQCRs from field supervisors.	Mark Fidler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Mark Fidler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Derek Lieberman, Project Manager, Ahtna
Do project plans adequately address any changes in project activities or goals?	Ongoing throughout project	Eric Schmidt, Project Chemist, Ahtna Derek Lieberman, Project Manager, Ahtna Bruce Wilcer, QC Manager, Ahtna	Compare data gathered to assess conformance to the project plan and conceptual site model, DQO, and project plan.	Document Authors	Document Authors	Eric Schmidt, Project Chemist, Ahtna Derek Lieberman, Project Manager, Ahtna

4.2 Assessment Findings and Corrective Action Responses (Worksheet #32)

For each type of assessment, the procedures for handling QAPP and project deviations encountered during the planned project assessments are described. The regulatory agencies will be notified within 30 days of the implementation of significant corrective actions or procedural changes to the QAPP.

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title and Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title and Organization)	Timeframe for Response
Phase I – Preparatory Phase						
Planning Document review	Internal Memo	Document Author	Prior to the start of field activities	Response to comments documentation and USACE approval of document as applicable	Derek Lieberman, Project Manager, Ahtna	One week
Planning document (QAPP) sign-off by field staff, subcontractors, and laboratory	Memo	Andrew Mauck, Soil Gas Monitoring Task Lead Mark Fisler, Field Supervisor, Ahtna Brian Whittaker, Project Manager, Eurofins	Prior to the start of field activities	Obtain sign-off that document has been read and understood by field and laboratory personnel	Derek Lieberman, Project Manager, Ahtna	One week
Preliminary work activities performed	Memo	Andrew Mauck, Soil Gas Monitoring Task Lead	Prior to the start of field activities	Provide clearance forms, permit forms, site access communications	Derek Lieberman, Project Manager, Ahtna	Prior to the start of field activities
Review of lab and field staff readiness	Memo	Mark Fisler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead Brian Whittaker, Project Manager, Eurofins	Prior to the start of field activities	Provide kick-off meeting notes from field and laboratory meetings	Derek Lieberman, Project Manager, Ahtna	One week
Review of field equipment	Memo	Mark Fisler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead Brian Whittaker, Project Manager, Eurofins	Prior to the start of field activities	Provide checklist documenting field equipment is available and in good working order	Derek Lieberman, Project Manager, Ahtna	Prior to the start of field activities.

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title and Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title and Organization)	Timeframe for Response
Phase II – Initial Phase						
Work performed according to project plans	Memo	Mark Fisler, Field Supervisor, Ahtna Andrew Mauck, SSHO/Soil Gas Monitoring Task Lead Derek Lieberman, Project Manager, Ahtna Bruce Wilcer, QC Manager, Ahtna	Within 24 hours of observation	Communications with USACE	Bridget Floyd, Technical Lead, USACE Duane Balch, Senior Project Manager, USACE Dana Gentry, Project Manager, USACE	One week
Field and laboratory audit	Field and Lab audit report	Mark Fisler, Field Supervisor, Ahtna Brian Whittaker, Project Manager, Eurofins Eric Schmidt, Project Chemist, Ahtna Derek Lieberman, Project Manager, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Within 48 hours of audits	Field and laboratory to issue formal response to audit findings requiring corrective action	Derek Lieberman, Project Manager, Ahtna Bruce Wilcer, QC Manager, Ahtna	One week
Review of CQCRs	Memo	Mark Fisler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Within 48 hours of review	Revision of CQCRs as needed	Derek Lieberman, Project Manager, Ahtna Bridget Floyd, Technical Lead, USACE Duane Balch, Senior Project Manager, USACE Dana Gentry, Project Manager, USACE	One week

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title and Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title and Organization)	Timeframe for Response
Review of project plans to reflect current site or lab activities	Memo	Mark Fisler, Field Supervisor, Ahtna Brian Whittaker, Project Manager, Eurofins Derek Lieberman, Project Manager, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Within 10 days of observations	Update project plans to reflect current conditions (may be addendum to existing document) or documentation of changes to field or laboratory protocol to be in accordance with project plans	Derek Lieberman, Project Manager, Ahtna Bridget Floyd, Technical Lead, USACE Duane Balch, Senior Project Manager, USACE Dana Gentry, Project Manager, USACE	Prior to next scheduled sampling event.
Field and laboratory audit	Field and Lab audit report	Mark Fisler, Field Supervisor, Ahtna Brian Whittaker, Project Manager, Eurofins Eric Schmidt, Project Chemist, Ahtna Derek Lieberman, Project Manager, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Within 48 hours of audits	Field and laboratory to issue formal response to audit findings requiring corrective action	Derek Lieberman, Project Manager, Ahtna Bruce Wilcer, QC Manager, Ahtna	One week
Phase III – Follow up and Reporting Phase						
Data reports prepared in accordance with project plans	Internal comments from staff	Document Author	Prior to issuance of report	Provide response to comments and revise report as needed	Commenting staff Derek Lieberman, Project Manager, Ahtna	Prior to issuance of report

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title and Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title and Organization)	Timeframe for Response
Report meets client and regulatory agency requirements	External comments from client and regulatory agencies	Document Author Derek Lieberman, Project Manager, Ahtna	Within 30 days of receipt of report	Provide response to comments and revise report as needed	Commenting Client and or Agencies Derek Lieberman, Project Manager, Ahtna Bridget Floyd, Technical Lead, USACE Duane Balch, Senior Project Manager, USACE Dana Gentry, Project Manager, USACE	30 days
Other definable features of work completed	Memo	Derek Lieberman, Project Manager, Ahtna	Before end of contract period	Complete definable features of work	Derek Lieberman, Project Manager, Ahtna Bridget Floyd, Technical Lead, USACE Duane Balch, Senior Project Manager, USACE Dana Gentry, Project Manager, USACE	Before end of contract period

4.3 Quality Assurance (QA) Management Reports Table (Worksheet #33)

The frequency and type of planned QA Management Reports, the project delivery dates, the personnel responsible for report preparation, and the report recipients are identified.

Type of Report	Frequency (daily, weekly, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (name, title and organization)	Report Recipient(s) (name, title and organization)
Daily CQCR Field Report	Daily	At the end of each day of fieldwork. Original field reports will be kept on-site in the project file.	Mark Fisler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Derek Lieberman, Project Manager, Ahtna Bruce Wilcer, QC Manager, Ahtna Duane Balch, Senior Project Manager, USACE Dana Gentry, Project Manager, USACE Bridget Floyd, Technical Lead, USACE Tom Ghigliotto, Field Oversight Inspector, Chenega
Field Work Variance Report	As needed	Prior to implementation of proposed change or immediately following a variance implemented in the field. A copy of the Field Work Variance will also be included in the final report.	Mark Fisler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Duane Balch, Senior Project Manager, USACE Dana Gentry, Project Manager, USACE Derek Lieberman, Project Manager, Ahtna Eric Schmidt, Project Chemist, Ahtna Bridget Floyd, Technical Lead, USACE
Non-Routine Occurrences Report	As needed	Within 48 hours of a Non-Routine Occurrence in the field or laboratory. A copy of this report will also be included in the final report.	Mark Fisler, Field Supervisor, Ahtna Andrew Mauck, Soil Gas Monitoring Task Lead	Duane Balch, Senior Project Manager, USACE Dana Gentry, Project Manager, USACE Derek Lieberman, Project Manager, Ahtna Eric Schmidt, Project Chemist, Ahtna Bridget Floyd, Technical Lead, USACE

5.0 Data Review

5.1 Verification (Stage 1) Process Table (Worksheet #34)

Verification Input	Description	Internal / External	Responsible for Verification (Name, Title, and Organization)
Chain of custody and shipping forms	Chain of custody forms will be reviewed upon completion and verified against the contents of the sample coolers they represent. After verification, the shipper's signature on the chain of custody form will be initialed by the reviewer. A copy of the form will be retained in the site file, and the original and remaining copies will be taped inside the cooler for shipment. Refer to Ahtna SOP (Attachment A) for further detail.	Internal	Mark Fisler, Field Supervisor, Ahtna
	The condition of canisters/sample IDs of the canisters will be documented upon receipt at the analytical laboratory through use of a receipt form. Each canister will be certified clean by the laboratory. The completed receipt form will be transmitted by email upon receipt of samples in the lab. The receipt form will also be submitted with the final analytical results from the laboratory.	Internal	Mark Fisler, Field Supervisor, Ahtna
	Receiving laboratory will verify chain of custody forms with contents of coolers. The Ahtna Task Lead and Project Chemist will be notified of discrepancies or issues within 24 hours of sample receipt. Resolution will be documented in writing and submitted with final data package.	External	Brian Whittaker, Project Manager, Eurofins
	Laboratory receipt/login report will be reviewed against chain of custody form internally.	Internal	Eric Schmidt, Project Chemist, Ahtna
Field Notes	Field notes will be reviewed internally by the field supervisor for consistency with the chain of custody forms and SOPs. The original field notes will be retained in the site file, and a copy of field notes will be forwarded to Task Lead for review.	Internal	Mark Fisler, Field Supervisor, Ahtna
Laboratory Data	Analytical data packages will be verified by the laboratory performing the work for completeness prior to submittal.	External	Brian Whittaker, Project Manager, Eurofins
	Received data packages will be verified according to the data validation procedures specified in Worksheet #35. Laboratory EDDs will be verified against the data package hard copy reports.	Internal	Eric Schmidt, Project Chemist, Ahtna

5.2 Validation (Stages 2A and 2B) Process Table (Worksheet #35)

Stage	Validation Input	Description	Responsible for Verification (Name, Title, and Organization)
2A	Methods used for sample collection	Field data notes will be reviewed for compliance with published methods and SOPs. Deviations from SOPs and methods described in this QAPP will be summarized and provided to the Project Manager in writing.	Eric Schmidt, Project Chemist, Ahtna
2A	Methods used for analysis	Laboratory data packages will be reviewed to verify that the methods specified in this QAPP and applicable published methods and laboratory SOP requirements were followed. Deviations shall be documented in writing.	Eric Schmidt, Project Chemist, Ahtna
2A	SOPs (field sampling)	Review field notes for compliance with SOPs.	Eric Schmidt, Project Chemist, Ahtna
		Review laboratory data deliverables for compliance with QAPP, laboratory SOP, and published methods.	Eric Schmidt, Project Chemist, Ahtna
2A	Documentation of method QC results	Review laboratory data packages to determine if QC parameters required by the referenced methods were performed and reported. The QC forms will be reviewed to determine if method acceptance criteria were met. Method QC outliers will be identified by the laboratory in the case narrative. Reviewer will determine if data will require qualification due to outliers.	Eric Schmidt, Project Chemist, Ahtna
2B	Documentation of QAPP QC sample results	Verify that QC samples specified in this QAPP were analyzed and reported. Reviewer will identify QAPP QC sample results in the data validation report.	Eric Schmidt, Project Chemist, Ahtna
2B	Laboratory data package documentation	Laboratory data packages will be reviewed to ensure documentation requirements specified in the QAPP have been met. If deficiencies are found, the data reviewer will document the issue in a memorandum to the laboratory. The laboratory will address deficiencies in writing or submit a revised data package addressing the deficiencies.	Eric Schmidt, Project Chemist, Ahtna
2B	Target analyte list	Laboratory report summary forms will be reviewed to verify that the target compounds and parameters specified in the QAPP were reported.	Eric Schmidt, Project Chemist, Ahtna
2B	LOD/LOQ	Determine that the quantitation limits were achieved, as outlined in the QAPP. Verify that the laboratory analyzed a low standard at the quantitation limit in the initial calibration.	Eric Schmidt, Project Chemist, Ahtna
2A and 2B	Data Validation Report	Summarize deviations from the referenced methods, SOPs, and QAPP-specific requirements. Include qualified data and explanations of all data qualifiers.	Eric Schmidt, Project Chemist, Ahtna
	Data Validation Report Review	Review validation reports and Summary Reports.	Eric Schmidt, Project Chemist, Ahtna

5.3 Validation (Stages 2B and 4) Summary Table (Worksheet #36)

The matrices, analytical groups, and concentration levels that each entity performing validation will be responsible for, as well as criteria that will be used to validate those data are identified below.

Stage 2B/4	Matrix	Analytical Group	Validation Criteria	Data Validator (name, title and organization)
2B	All	VOCs	General Data Validation Guidelines, Environmental Data Quality Workgroup, Revision 1 (DoD, 2019)	Eric Schmidt, Project Chemist, Ahtna
4	All	VOCs	EPA 540-R-08-005. "Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use" (EPA, 2009) and associated QAPP Worksheets	Eric Schmidt, Project Chemist, Ahtna

Data review and validation will be performed in accordance with the guidelines specified in Worksheets #34-37. Data review (Stage 2B) is a process where an evaluation of data is based on the review of QC only. Data qualifiers are shown in Table 4. Stage 2B data review will be performed on all (100%) samples. Stage 2B review comprises an evaluation of the following:

- Data package completeness
- Holding times and sample preservation
- Initial and continuing calibrations
- MB contamination
- Surrogate compound recoveries
- Laboratory control sample/ laboratory control sample duplicate (LCS/LCSD) recoveries and RPDs
- Field duplicate RPDs
- Internal standard areas, where applicable
- Target compound DLs

Data validation (Stage 4) is a process where QC, along with raw data, are evaluated to confirm calculations and analyte identifications. Data validation will be performed on a minimum of 10% of samples. Stage 4 review comprises an evaluation of the following:

- Stage 2B review
- Recalculation of reported sample results using instrument output results, dilution factors, and calibration factors
- Review of instrument outputs
- Verification of analyte identification

5.4 Usability Assessment (Worksheet #37)

The procedures, methods, and activities that will be used to determine whether data are of the right type, quality, and quantity to support project decisions are described below. Also described is how data quality issues will be addressed and how limitations on the use of the data will be handled.

The suitability of the environmental data collected for its intended use will be assessed by the Ahtna Project Chemist in consultation with the Project Manager. Data usability will comprise an evaluation of the quantity, type, and overall quality of the generated data against the project DQOs as presented in Worksheet #10. The usability of data associated with QC results outside of the established acceptance criteria is dependent on the degree of the exceedance, whether the potential bias is high or low, and whether the uncertainty implied by the exceedance is significant relative to project decisions and DQOs. Data usability will be assessed in accordance with the guidance provided in the QSM Version 5.3 (DoD, 2019) and additional applicable USACE and EPA guidance as well as the professional experience of the decision-maker during data validation. The following items will be assessed and conclusions drawn based on their results:

- **Precision** – Duplicate field and laboratory samples will be evaluated for precision based on the calculated RPD between detectable results between duplicate samples. RPDs exceeding MPC in Worksheet #12 will be identified and any limitations on the use of the data will be noted. RPDs within the MPC will demonstrate the data has acceptable precision and the data are usable.
- **Accuracy** – LCS will be evaluated for accuracy of the data by comparing laboratory results with MPC in Worksheet #12.
- **Sensitivity** – The sensitivity of the data will be verified by comparing MB results with MPC in Worksheet #12 and cross-checking analyte data with LOQs presented in Worksheet #15.
- **Bias** – Laboratory surrogates will be investigated for bias by comparing results with MPC in Worksheet #28.
- **Contamination** – MB data will be used to determine whether there are contamination issues based upon MPC in Worksheet #12. Helium shroud leak testing results (see SOPs 2 and 3) will also be used to determine whether there are contamination issues based on field sampling methods and/or conditions.
- **Representativeness** – Sampling procedures will be implemented in accordance with SOPs to eliminate or minimize sources of error. Compliance with SOPs will be confirmed through QC field audits. Analytical procedures will be implemented in accordance with laboratory SOPs (Attachment A), quality control acceptance limits, and the laboratory Quality Assurance Manual. Laboratories used for sample analysis will maintain DoD ELAP certification.
- **Completeness** – The completeness of the sample event will be determined based upon the number of field samples collected compared to the number of samples planned and the number of unqualified laboratory results compared to the total number of results. This information will be compared to MPC in Worksheet #12.
- **Comparability** – The data from each sampling event is comparable to past and future events as long as the same or similar sampling and analytical SOPs are utilized.

- **Reconciliation** – Each of the DQOs presented in Worksheet #10 will be examined to determine whether the objectives were met. This examination will include a combined overall assessment of the results of each analysis pertinent to an objective. Each analysis will be first evaluated separately in terms of the major impacts observed from the data validation, DQI and MPC assessments. Based on the results of these assessments, the quality of the data will be determined. Based on the quality, the usability of the data for each analysis will be determined. Based on the combined usability of the data from all analyses for an objective, it will be determined whether the DQO was met and whether action limits were exceeded.

In the event the data quantity or quality prove to be inadequate to meet project objectives, reanalysis or re-sampling may be required. Replacement samples may be collected when existing data is insufficient or inadequate to support project objectives. The decision to collect replacement samples will be made in coordination with the project team and may include USACE, the Ahtna Project Manager, and the Ahtna Program Chemist. Laboratory audits are performed as part of the DoD ELAP certification process and are beyond the scope of this QAPP. In the event a problem is encountered with the laboratory, the Army may request a special audit.

Usability of the data will be presented in validation reports, which will be attached to the final submitted reports.

6.0 References

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IDQTF, 2005b. *Uniform Federal Policy for Quality Assurance Plans, Part 2B: Quality Assurance/Quality Control Compendium: Minimum QA/QC Activities, Final Version 1*. March. Available at: <http://www2.epa.gov/fedfac/assuring-quality-federal-cleanups#ufp-qapp>.

¹³ At the end of references included in the Fort Ord Administrative Record are the Administrative Record Numbers (AR#s) (e.g. BW-1234). To find the referenced document, this number may be typed into the Online Search tool at: <http://www.fortordcleanup.com/documents/search/>. Please note the referenced documents were available in the Fort Ord Administrative Record at the time this document was issued; however, some may have been superseded by more current versions and were subsequently withdrawn.

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TABLES

Table 1. Soil Gas Probe Identification, Sample Schedule, and Purge Specifications

Soil Gas Location ID	Soil Gas Probe ID	Screen Depth (ft bgs)	Sample Schedule ¹	Purge Volume ² (mL)	Purge Rate ³ (mL/min)	Purge Time (min)
SG-12-01	SG-12-01-10	10	R	21,810	200	109
	SG-12-01-20	20	R	21,973	400	55
	SG-12-01-30	30	R	22,136	400	55
	SG-12-01-40	40	R	21,112	400	53
	SG-12-01-50	50	R	23,054	400	58
	SG-12-01-58	58	R	22,592	400	56
	SG-12-01-65 ⁵	65	A	23,892	400	60
SG-12-02	SG-12-02-10 ⁴	10	Q	22,403	200	112
	SG-12-02-20	20	A	22,566	400	56
	SG-12-02-30	30	A	22,729	400	57
	SG-12-02-40	40	A	22,892	400	57
	SG-12-02-50	50	A	23,054	400	58
	SG-12-02-57	57	A	23,168	400	58
	SG-12-02-65	65	R	23,299	400	58
SG-12-03	SG-12-03-10	10	R	22,403	200	112
	SG-12-03-20.5	20.5	R	22,574	400	56
	SG-12-03-30	30	R	22,729	400	57
	SG-12-03-39.5	39.5	R	19,918	400	57
	SG-12-03-50	50	R	22,461	400	58
	SG-12-03-58	58	R	23,185	400	58
	SG-12-03-65	65	R	23,299	400	58
SG-12-04	SG-12-04-10 ⁴	10	Q	21,810	200	109
	SG-12-04-20	20	A	21,973	400	55
	SG-12-04-30	30	R	22,136	400	55
	SG-12-04-40	40	R	22,892	400	57
	SG-12-04-50	50	R	22,461	400	56
	SG-12-04-58	58	R	22,592	400	56
	SG-12-04-65 ⁵	65	A	20,333	400	51
SG-12-05	SG-12-05-10	10	R	22,403	200	112
	SG-12-05-20	20	R	22,566	400	56
	SG-12-05-30	30	R	22,729	400	57
	SG-12-05-40	40	R	22,892	400	57
	SG-12-05-50	50	R	23,054	400	58
	SG-12-05-60	60	R	23,217	400	58
	SG-12-05-70	70	R	20,415	400	51

Table 1. Soil Gas Probe Identification, Sample Schedule, and Purge Specifications

Soil Gas Location ID	Soil Gas Probe ID	Screen Depth (ft bgs)	Sample Schedule ¹	Purge Volume ² (mL)	Purge Rate ³ (mL/min)	Purge Time (min)
SG-12-06	SG-12-06-10 ⁴	10	Q	22,403	200	112
	SG-12-06-20	20	R	22,566	400	56
	SG-12-06-30	30	R	22,729	400	57
	SG-12-06-40	40	R	22,892	400	57
	SG-12-06-50	50	R	23,054	400	58
	SG-12-06-60	60	R	23,217	400	58
	SG-12-06-70	70	Q	20,415	400	51
SG-12-07	SG-12-07-10	10	N	21,810	200	109
	SG-12-07-20	20	R	36,799	400	92
	SG-12-07-30	30	R	22,136	400	55
	SG-12-07-40	40	R	22,298	400	56
	SG-12-07-50	50	R	23,054	400	58
	SG-12-07-57.5	57.5	R	20,211	400	51
	SG-12-07-65	65	R	20,333	400	51
SG-12-08	SG-12-08-10	10	R	22,403	200	112
	SG-12-08-20	20	R	22,566	400	56
	SG-12-08-30	30	R	22,729	400	57
	SG-12-08-40	40	R	22,892	400	57
	SG-12-08-50	50	R	23,054	400	58
	SG-12-08-60	60	R	23,217	400	58
	SG-12-08-70	70	R	20,415	400	51
SG-12-09	SG-12-09-10	10	R	22,403	200	112
	SG-12-09-20	20	R	22,566	400	56
	SG-12-09-30	30	R	22,729	400	57
	SG-12-09-40	40	R	22,298	400	56
	SG-12-09-50	50	R	20,089	400	50
	SG-12-09-59	59	R	20,236	400	51
SG-12-11	SG-12-11-10	10	R	21,810	200	109
	SG-12-11-20	20	R	22,566	400	56
	SG-12-11-30	30	R	22,729	400	57
	SG-12-11-40	40	R	22,892	400	57
	SG-12-11-50	50	R	23,054	400	58
	SG-12-11-60	60	R	23,217	400	58
	SG-12-11-70	70	R	20,415	400	51
SG-12-12	SG-12-12-10	10	R	21,810	200	109
	SG-12-12-20	20	R	22,566	400	56
	SG-12-12-30	30	R	22,729	400	57
	SG-12-12-40	40	R	22,298	400	56
	SG-12-12-50	50	N	22,461	400	56
	SG-12-12-60	60	R	22,624	400	57
	SG-12-12-70	70	R	20,415	400	51

Table 1. Soil Gas Probe Identification, Sample Schedule, and Purge Specifications

Soil Gas Location ID	Soil Gas Probe ID	Screen Depth (ft bgs)	Sample Schedule ¹	Purge Volume ² (mL)	Purge Rate ³ (mL/min)	Purge Time (min)
SG-12-13	SG-12-13-10	10	R	22,996	200	115
	SG-12-13-20	20	R	23,159	400	58
	SG-12-13-30	30	R	23,322	400	58
	SG-12-13-40	40	R	25,857	400	65
	SG-12-13-50	50	R	22,461	400	56
	SG-12-13-60	60	R	26,183	400	65
	SG-12-13-70	70	N	20,415	400	51
SG-12-14	SG-12-14-10	10	R	21,810	200	109
	SG-12-14-20	20	R	21,973	400	55
	SG-12-14-30	30	R	22,136	400	55
	SG-12-14-40	40	R	22,892	400	57
	SG-12-14-50	50	R	23,054	400	58
	SG-12-14-60	60	R	22,624	400	57
	SG-12-14-70	70	R	20,415	400	51
SG-12-15	SG-12-15-10	10	R	25,368	200	127
	SG-12-15-20	20	R	25,531	400	64
	SG-12-15-30	30	R	25,694	400	64
	SG-12-15-40	40	R	25,857	400	65
	SG-12-15-50	50	R	22,461	400	56
	SG-12-15-60	60	R	26,183	400	65
	SG-12-15-70	70	R	23,973	400	60
SG-12-16	SG-12-16-10	10	R	25,368	200	127
	SG-12-16-20	20	R	21,973	400	55
	SG-12-16-30	30	R	22,136	400	55
	SG-12-16-40	40	R	23,485	400	59
	SG-12-16-50	50	R	22,461	400	56
	SG-12-16-60 ⁶	60	R	23,217	400	58
	SG-12-16-70	70	R	20,415	400	51
SG-12-17	SG-12-17-10	10	R	22,403	200	112
	SG-12-17-20	20	R	25,531	400	64
	SG-12-17-30	30	R	22,729	400	57
	SG-12-17-40	40	R	25,857	400	65
	SG-12-17-50	50	R	26,020	400	65
	SG-12-17-60	60	R	23,217	400	58
	SG-12-17-75	75	N	20,496	400	51
SG-12-18	SG-12-18-10	10	R	22,996	200	115
	SG-12-18-20	20	R	23,159	400	58
	SG-12-18-30	30	R	22,729	400	57
	SG-12-18-40	40	R	22,892	400	57
	SG-12-18-50	50	R	23,648	400	59
	SG-12-18-60	60	R	26,183	400	65
	SG-12-18-70	70	R	20,415	400	51

Table 1. Soil Gas Probe Identification, Sample Schedule, and Purge Specifications

Soil Gas Location ID	Soil Gas Probe ID	Screen Depth (ft bgs)	Sample Schedule ¹	Purge Volume ² (mL)	Purge Rate ³ (mL/min)	Purge Time (min)
SG-12-19	SG-12-19-10	10	R	21,810	200	109
	SG-12-19-20	20	R	21,973	400	55
	SG-12-19-30	30	R	22,136	400	55
	SG-12-19-40	40	R	23,485	400	59
	SG-12-19-50	50	R	23,648	400	59
	SG-12-19-60	60	R	22,624	400	57
	SG-12-19-70	70	R	20,415	400	51
SG-12-20	SG-12-20-10	10	A	22,996	200	115
	SG-12-20-20	20	A	21,973	400	55
	SG-12-20-30	30	R	22,136	400	55
	SG-12-20-40	40	R	23,485	400	59
	SG-12-20-50	50	R	23,648	400	59
	SG-12-20-60	60	R	23,810	400	60
	SG-12-20-70	70	R	20,415	400	51
SG-12-22	SG-12-22-10	10	R	21,810	200	109
	SG-12-22-20	20	R	23,159	400	58
	SG-12-22-30	30	R	23,322	400	58
	SG-12-22-40	40	R	22,298	400	56
	SG-12-22-50	50	R	23,648	400	59
	SG-12-22-60	60	R	23,810	400	60
	SG-12-22-70	70	R	21,601	400	54
SG-12-23	SG-12-23-10	10	R	22,996	200	115
	SG-12-23-20	20	R	23,159	400	58
	SG-12-23-30	30	R	23,322	400	58
	SG-12-23-40	40	R	22,298	400	56
	SG-12-23-50	50	R	22,461	400	56
	SG-12-23-60	60	R	21,438	400	54
	SG-12-23-70	70	R	22,787	400	57
SG-12-24	SG-12-24-10	10	R	22,966	200	115
	SG-12-24-20	20	R	23,159	400	58
	SG-12-24-30	30	R	23,322	400	58
	SG-12-24-40	40	R	23,485	400	59
	SG-12-24-50	50	R	23,648	400	59
	SG-12-24-60	60	R	23,810	400	60
	SG-12-24-70	70	R	20,415	400	51

Table 1. Soil Gas Probe Identification, Sample Schedule, and Purge Specifications

Soil Gas Location ID	Soil Gas Probe ID	Screen Depth (ft bgs)	Sample Schedule ¹	Purge Volume ² (mL)	Purge Rate ³ (mL/min)	Purge Time (min)
----------------------	-------------------	-----------------------	------------------------------	--------------------------------	----------------------------------	------------------

Notes:

- ¹ The soil gas probe sample schedule as of December 2020 and subject to change based on the analytic approach.
- ² Purge volume calculation is detailed in QAPP Section 2.10.5 and Figure 4.
- ³ Purge rates were determined based on guidance in the Advisory, Active Soil Gas Investigations (DTSC, 2015): shallow (10-foot) soil gas probes purged at approximately 200 mL/min due proximity to the surface where ambient air interference is more likely; deeper soil gas probes purged at 400 mL/min or less. Purge vacuums will not exceed maximum of 7.4 inches mercury (100 inches water).
- ⁴ Soil gas probe located vertically and laterally adjacent to the front of a building with historic results above soil gas cleanup level and sampled quarterly for the duration of the SGMP.
- ⁵ The soil gas probe is located near the groundwater interface in the tetrachloroethene groundwater plume footprint; subject to change based on recent groundwater analytical data.
- ⁶ This probe was removed from the sampling schedule as of this QAPP revision.

Acronyms and Abbreviations:

- A: Annual (third quarter event)
- ft bgs: feet below ground surface
- ID: identification
- min: minutes
- mL: milliliters
- N: Non-functional, cannot be sampled due to an obstruction or the screen interval submerged in groundwater
- Q: Quarterly
- R: Removed from sampling program
- SGMP: soil gas monitoring program

Table 2. Soil Vapor Extraction Treatment System Identification and Sample Schedule

SVETS Group	SVETS Sample Point ID	Screen Depth (ft bgs)	Top of Screen Elevation (ft MSL)	Sample Schedule ¹
Southern Well Field ²	VE-12-01	47.9-67.9	37.77	R
	VE-12-02	50-70	37.07	R
	VE-12-03	50.6-70.6	35.01	R
	VE-12-04	30-50	55.64	R
	VE-12-05	30.3-50.3	55.62	R
Northern Well Field ³	VE-12-06	45-65	29.69	R
	VE-12-07	45-65	32.1	R
	VE-12-08	45-65	34.01	R
	VE-12-09	45-65	27.95	QNO
	VE-12-10	45-65	26.99	R
SVTU	SVTU-212-INF	N/A	N/A	QNO
	SVTU-212-MID	N/A	N/A	R
	SVTU-212-EFF	N/A	N/A	QNO

Notes:

¹ The SVETS sample schedule as of December 2020; subject to change based on the analytical approach in Worksheet #10.

² These SVE wells are located near the historical trichloroethene soil gas plume footprint.

³ These SVE wells are located in the tetrachloroethene groundwater plume footprint.

Acronyms and Abbreviations:

ft bgs: feet below ground surface

ID: identification

N/A: not applicable

QNO: Quarterly currently not in operation (sampled if online)

R: Removed from sampling program (unless online)

SVE: soil vapor extraction

SVETS: soil vapor extraction treatment system

SVTU: soil vapor treatment unit

Table 3. Summary of Soil Gas and SVETS Samples Collected

Sample Location Type	Total Number of Sample Locations by Type	Number of Army-Owned Sample Locations	Total Number of Locations Currently Sampled	Number of Locations Sampled Quarterly	Number of Locations Sampled Annually	Number of Locations Not Sampled	Number of Samples Collected Annually ^{1,2}
Soil gas probe	153	153	18	4	10	135	29
Soil vapor extraction well	10	10	0	0	0	10	0
Soil vapor treatment unit	3	3	0	0	0	3	0
Total	166	166	18	4	10	148	32

Notes:

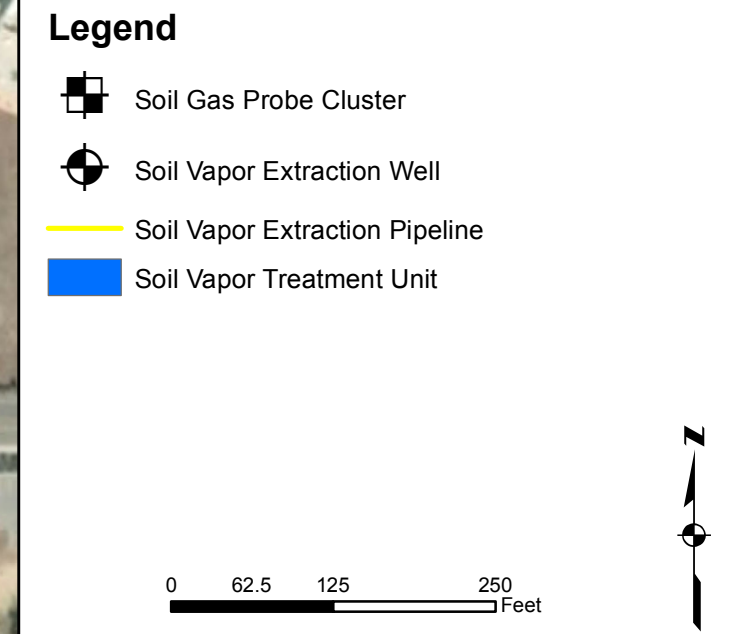
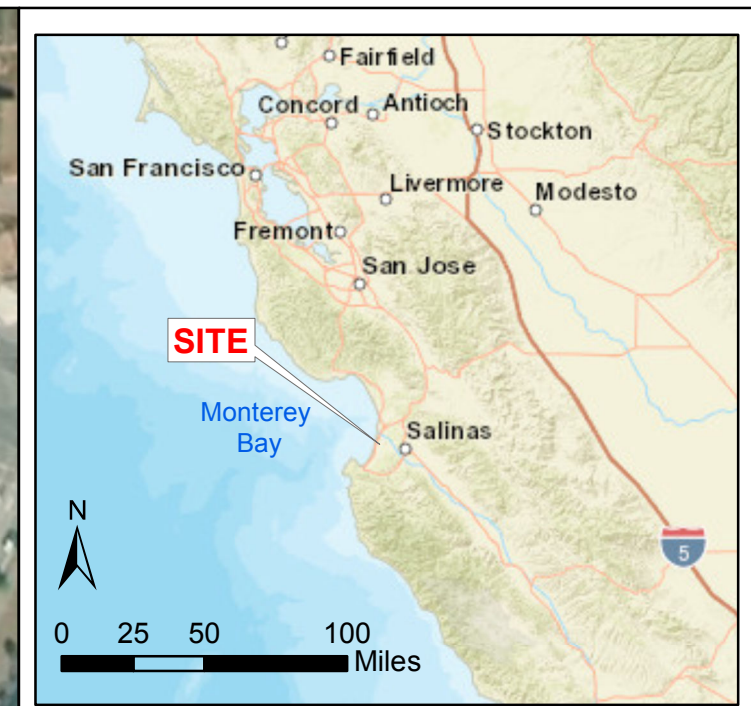
¹ Total Includes duplicate samples collected during soil gas sampling at a frequency of 10 percent (%) per quarterly event at soil gas probes.

² The soil gas probe and SVETS sample schedule as of December 2020 and subject to change based on the analytic approach in Worksheet #10.

Table 4. Qualifiers Applied During Data Validation

Qualifier	Definition
U	The analyte was not detected and was reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
J	The reported result was an estimated value with an unknown bias.
J+	The result was an estimated quantity, but the result may be biased high.
J-	The result was an estimated quantity, but the result may be biased low.
N	The analysis indicates the presence of an analyte for which there was presumptive evidence to make a "tentative identification."
NJ	The analyte has been "tentatively identified" or "presumptively" as present and the associated numerical value was the estimated concentration in the sample.
UJ	The analyte was not detected and was reported as less than the LOD or as defined by the customer. However, the associated numerical value is approximate.
X	The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Acceptance or rejection of the data should be decided by the project team (which should include a project chemist), but exclusion of the data is recommended.

FIGURES



Site 12 Location, Soil Vapor Treatment System and Soil Gas Monitoring Map

Quality Assurance Project Plan
Former Fort Ord, California
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Soil Gas Monitoring at Sites 2 and 12

AC-2330
AIR COOLER
HEAT EXCHANGER

DF-2104
DILUTION AIR FILTER
4 INCH

DS-2302
DISCHARGE SILENCER
8 INCH

DS-2321
DISCHARGE SILENCER
8 INCH

M/B-2320
BLOWER—POSITIVE DISPLACEMENT
W/SOUND ENCLOSURE
60 HP, 480V, 3Ø, 1,100 CFM

M/P-2220
TRANSFER PUMP
2 HP, 120V, 1Ø
25 GPM @ 26 FT

M/P-2420
TRANSFER PUMP
½ HP, 120V, 1Ø
2 GPM @ 26 FT

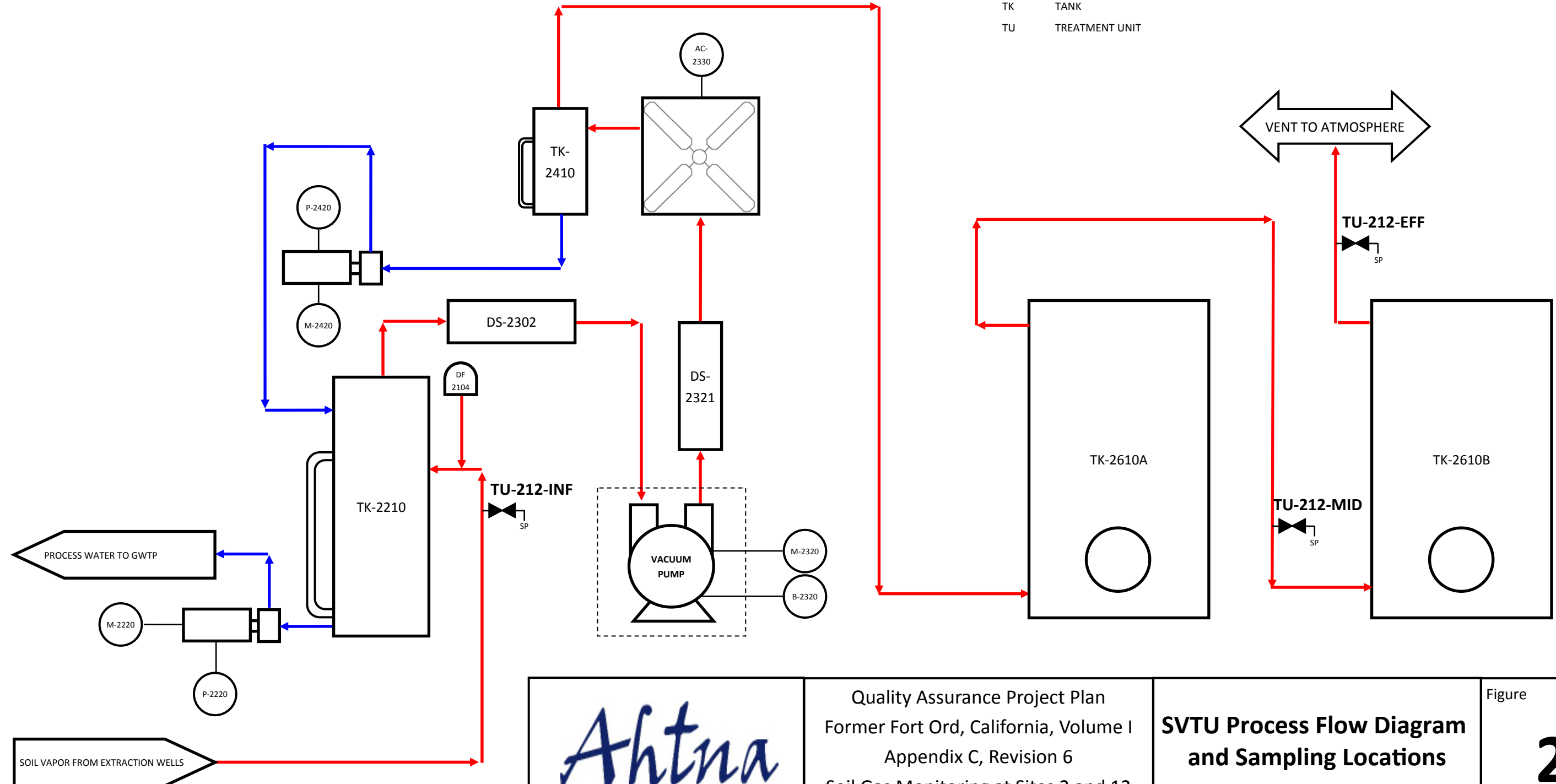
TK-2210
SEPERATOR—LIQUID
100 GAL

TK-2410
SEPERATOR—LIQUID
10 GAL

TK-2610A/B
VAPOR-PHASE CARBON VESSEL
3,000 LB GRANULAR ACTIVATED CARBON EACH

AC	AIR COOLER
B	BLOWER
DF	DILUTION AIR FILTER
DS	DISCHARGE SILENCER
EFF	EFFLUENT
GWTP	GROUNDWATER TREATMENT PLANT
INF	INFLUENT
M	MOTOR
MID	MID-POINT
P	PUMP
SP	SAMPLE PORT
TK	TANK
TU	TREATMENT UNIT

→ PROCESS AIR (SOIL VAPOR)
→ PROCESS WATER (CONDENSATE)
—| |— SAMPLE PORT

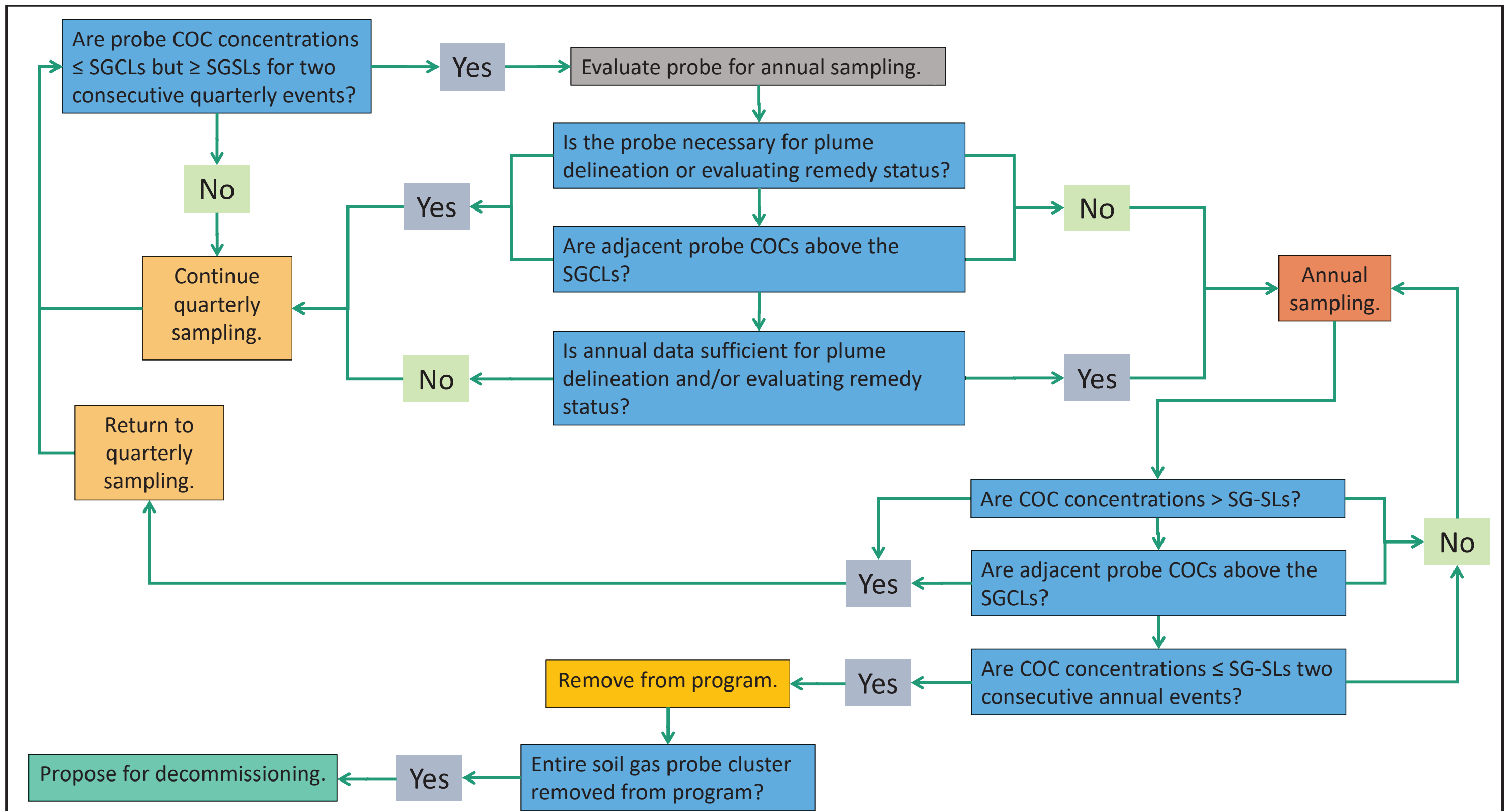


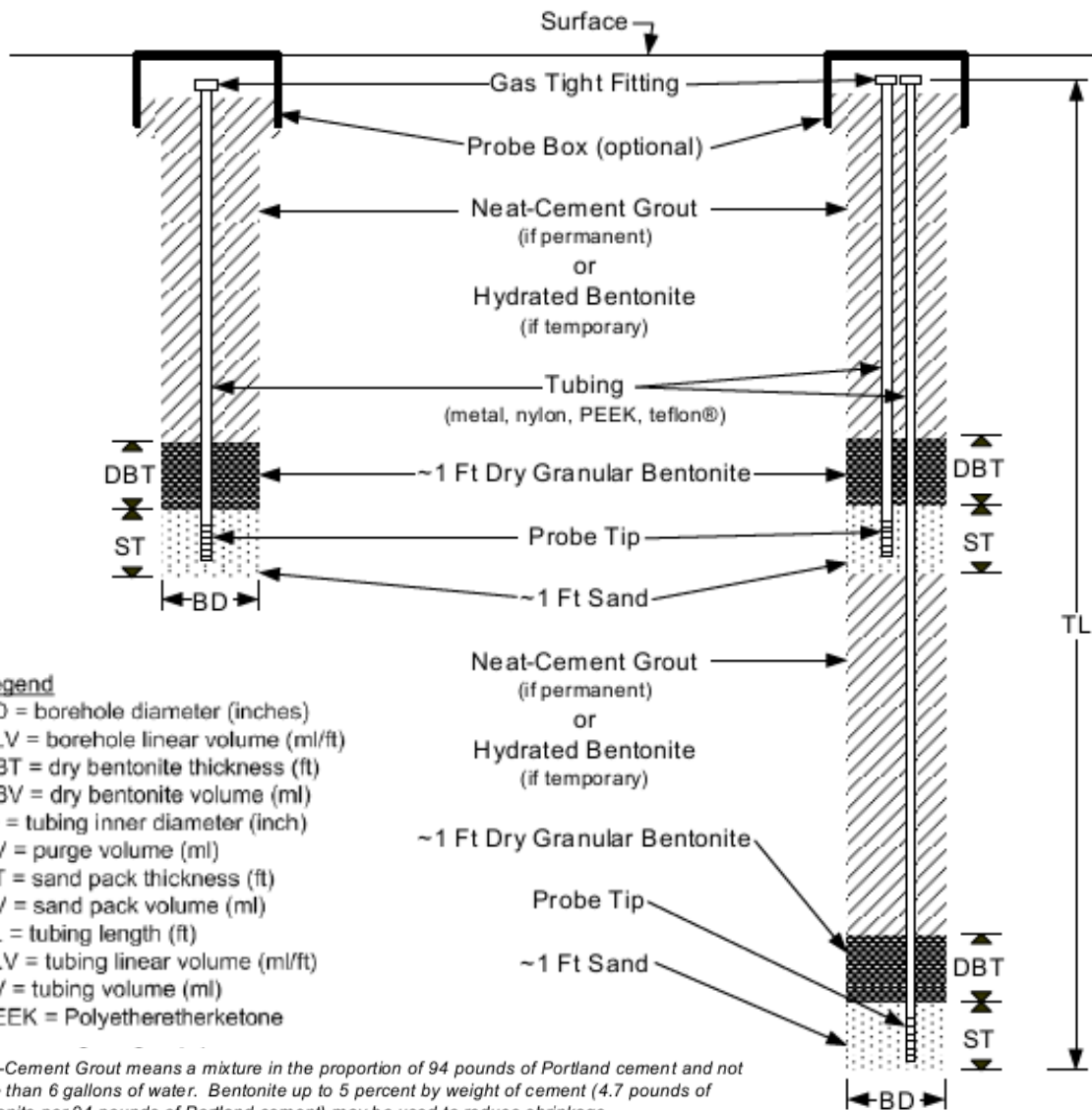
Ahtna

Quality Assurance Project Plan
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**SVTU Process Flow Diagram
and Sampling Locations**

Figure
2





Neat-Cement Grout means a mixture in the proportion of 94 pounds of Portland cement and not more than 6 gallons of water. Bentonite up to 5 percent by weight of cement (4.7 pounds of bentonite per 94 pounds of Portland cement) may be used to reduce shrinkage.

(1) TV = TL x TLV = (TL) _____	X 6 if tubing ID = 3/16" = _____ ml
	X 16 if tubing ID = 5/16" = _____ ml
	X ___ if tubing ID = ___" = _____ ml
(2) DBV = DBT x BLV = (DBT) _____	X 350 if BD = 2 1/8" = _____ ml
	X 820 if BD = 3 1/4" = _____ ml
	X ___ if BD = ___" = _____ ml
(3) SV = ST x BLV = (ST) _____	X 280 if BD = 2 1/8" = _____ ml
	X 660 if BD = 3 1/4" = _____ ml
	X ___ if BD = ___" = _____ ml
	1 PV = (1)TV + (2) DBV + (3) SV = _____ ml

Typical Soil Gas Probe Design & Purge Volume Calculation

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 Soil Gas Monitoring at Sites 2 and 12

Figure

4

Ahtna

ATTACHMENTS

ATTACHMENT A

Standard Operating Procedures (SOPs)

SOP No.	SOP Title	Author Organization
1	Guide to Air Sampling	Eurofins
2	Soil Gas Sampling	Ahtna
3	Helium Shroud Spec Sheet	Eurofins
4	SVE Treatment System Sampling	Ahtna
5	Common Sampling Procedures (Chapter 3 from DoD Environmental Field Sampling Handbook)	DoD
6	SVE Treatment System Flow Meter Use	Ahtna
#91	EPA Method TO-14A/TO-15 Volatile Organic Compounds (5&20 ppbv)	Eurofins
#83	EPA Method TO-14A/TO-15 Volatile Organic Compounds (Low-Level)	Eurofins



Soil Gas

Vapor Intrusion

Property Redevelopment

Ambient Air Monitoring

Indoor Air Quality

Waste-to-Energy



Air Toxics

Guide to Air Sampling

Canisters and Bags



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Guide to Whole Air Sampling – Canisters and Bags

Revision 6/27/14

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Section 1.0 Introduction

Eurofins Air Toxics Inc. presents this guide as a resource for individuals engaged in air sampling. Air sampling can be more involved than water or soil sampling due to the reactivity of chemical compounds in the gas matrix and sample interaction with the equipment and media used. Ensuring that air samples are collected properly is an important step in acquiring meaningful analytical results. This guide is not a substitute for experience and cannot sufficiently address the multitude of field conditions. Note that this guide is intended for projects involving whole air sampling of volatile organic compounds (VOCs) in canisters and Tedlar® bags. Eurofins Air Toxics provides the “Guide to Sorbent-Based Sampling - Volatiles and Semi-Volatiles” for other types of sampling.

1.1 Whole Air Sampling of VOCs

There are three general ways to collect compounds in a gas phase sample. A sampler may collect the gas sample in a container, actively pump the vapor through a sorbent tube, solution or filter, or rely on passive sample collection onto a sorbent bed. This guide focuses on collecting a sample in the most common air sampling containers, Summa canisters and bags. The sample may be collected in the container either passively, relying on an evacuated canister to drive the sample collection, or actively using a pump to fill the container. The container is subsequently sealed and transported to the laboratory for analysis. The sample is referred to as a “whole air sample” and the compounds remain in the gas matrix inside the container.

As a general rule, whole air sampling is appropriate when target compounds are chemically stable and have vapor pressures greater than 0.1 torr at 25°C and 760mm Hg (EPA standard ambient conditions). Performance of a given compound in a whole air sample is dependent upon its chemical properties, the matrix of the sample, and the degree of inertness of the sample container.

1.2 Choosing Between Canisters and Bags

Table 1.2 compares the features and performance of Summa canisters and bags. Summa canisters or similarly treated canisters are rugged containers designed to provide superior inertness and extended sample storage times. Evacuated canisters also do not require a sampling pump for sample collection. By contrast, bags require a sample pump, but can be purchased inexpensively in bulk, require little preparation or cleaning, and take up little space prior to use. Unlike canisters, bags are typically not appropriate for ppbv-level VOC measurements due to their background artifacts and short hold-times. Over time, low molecular weight gases can diffuse through the bag material while chemicals with lower vapor pressures can condense on the bag surface thereby compromising analyte recoveries. Call your Project Manager at 800-985-5955 if you have questions regarding the appropriate sampling media.

Table 1.2 Comparison of Canisters to Bags

	Canisters	Bags
Type of Sampling	Passive (vacuum)	Active (pump required)
Media Hold Time	Up to 30 days recommended	Indefinite
Hold Time to Analysis	Up to 30 days	Up to 3 days
Surface Inertness	Excellent	Fair
Cleanliness	Batch or 100% certified to ppbv/pptv levels	Some VOCs present in the ppbv range
Sampling Application	Ambient air, soil/landfill gas	Soil/landfill gas, stationary sources, SVE systems
Rule of Thumb	“ppbv device”	“ppmv device”
Advantages	Inertness, hold time, ruggedness, no pump	Purchase/shipping cost, availability, convenience

Section 2.0 Canisters and Associated Media

This section provides a description of air sampling canisters, practical considerations for sampling, and step-by-step instructions for collecting grab and integrated samples.

Photographs illustrate the correct way to assemble the various sampling components.

Tables provide detailed information on many operational factors that ultimately influence the quality of the data obtained from a canister sample.

2.1 Introduction to Canisters

An air sampling canister is a container for collecting a whole air sample. A canister may be spherical or cylindrical and is constructed of specially treated stainless steel. The canister is prepared for sampling by evacuating the contents to a vacuum of approximately 29.9 inches of Mercury (in Hg). Opening the stainless steel bellows valve allows the air sample to enter the canister. Flow controllers can be utilized to restrict the flow and allow for collection at a desired flow rate or over a desired range. When the sample has been collected, the valve is closed and the canister is returned to the laboratory. Canisters range in volume from less than 1 liter (L) to 6 L. In general, 6 L canisters are used to collect ambient air samples and samples requiring time integration greater than 2 hours. One liter canisters are typically used for taking high concentration (i.e., greater than 5 ppbv) samples not requiring time integration such as soil vapor.



2.1.1 Summa Canister

A Summa canister is a stainless steel container that has had the internal surfaces specially passivated using a “Summa” process. This process combines an electropolishing step with a chemical deactivation step to produce a surface that is nearly chemically inert. A Summa surface has the appearance of a mirror: bright, shiny and smooth. The degree of chemical inertness of a whole air sample container is crucial to minimizing reactions with the sample

and maximizing recovery of target compounds from the container. Eurofins Air Toxics maintains a large inventory of Summa canisters in 1 and 6 L volumes.

2.1.2 Canister Certification

Eurofins Air Toxics provides two types of canister cleaning certification, batch and 100%, depending upon the requirements of the project. The batch certification process is most appropriate for routine ambient air applications and high concentration applications such as soil vapor and landfill gas monitoring. The batch certification process begins by cleaning a set of canisters using a combination of dilution, heat and high vacuum. The cleaning batch is certified by analyzing a percentage of canisters for approximately 60 VOCs using GC/MS. The batch meets cleaning requirements if the target compound concentrations are below 0.2 ppbv. Alternatively, the 100% certification (i.e., individual certification) process is typically required for ambient and indoor air applications driven by risk assessment or litigation requiring pptv (parts per trillion by volume) sensitivity. If 100% certification is required, canisters are individually certified for a client-specific list of target compounds using GC/MS. When the 100% certified canisters are shipped, the analytical documentation demonstrating that they are free of the target compounds down to the project reporting limits is emailed to the client. When sampling with certified media, it is important to note that all media is certified as a train and must be sampled as such (i.e., a particular flow controller goes with a particular canister and is labeled as such).



Specify whether your project requires batch or 100% canister certification.

2.1.3 Canister Hold Time

Media Hold Time: Unlike water and soil environmental samples, which are collected in single-use, disposable vials and jars, air samples are collected in reusable summa canisters. Eurofins Air Toxics requires that canisters be returned within 15 days of receipt to effectively manage our inventory and to insure canisters meet performance requirements in the field. Evacuated canisters have a finite timeframe before the canisters naturally lose

vacuum during storage. Using canisters beyond 15 days increases the risk of having unacceptable initial vacuum at the start of sampling.

Sample Hold Time: EPA Method TO-15 cites a sample hold time of up to 30 days for most VOCs. Several non-routine compounds, such as bis(chloromethyl)ether, degrade quickly and demonstrate low recovery even after 7 days. Reactive sulfur compounds such as hydrogen disulfide and methyl, ethyl, and butyl mercaptan are not amenable to storage in stainless steel summa canister, and either fused silica lined (FSL) canisters or Tedlar bags are required for sample collection.

2.2 Associated Canister Hardware

Associated hardware used with the canister includes the valve, brass cap, particulate filter and vacuum gauge. (Flow controllers are covered in detail in section 3.2.)

2.2.1 Valve

An industry standard 1/4" stainless steel bellows valve is mounted at the top of the canister. The valve maintains the vacuum in the canister prior to sampling and seals the canister once the sample has been collected. No more than a half turn by hand is required to open the valve. Do not over-tighten the valve after sampling or it may become damaged. A damaged valve can leak, possibly compromising the sample. Some canisters have a metal cage near the top to protect the valve.

To protect the valve and provide secure connections in the field, a replaceable fitting is attached to all canisters. As threads wear and require replacement, new fittings can be installed at the laboratory prior to shipping to the field. You will need a 1/2" wrench to secure the fitting while connecting or removing the required equipment to the canister.

2.2.2 Brass Cap

Each canister comes with a brass cap (i.e., Swagelok 1/4" plug) secured to the inlet of the valve assembly. The cap serves two purposes. First, it ensures that there is no loss of vacuum due to a leaky valve or a valve that is accidentally opened during handling. Second, it prevents dust and other particulate matter from damaging the valve. The cap is removed prior to sampling and replaced following sample collection.



Always replace the brass cap following canister sampling.

2.2.3 Particulate Filter

Particulate filters should always be used when sampling with a canister. Separate filters are provided to clients taking a grab sample, and filters are built into the flow controllers for clients taking integrated samples. The 2 micron filter is a fritted stainless steel disk that has been pressed into a conventional Swagelok adapter. This device has a relatively high pressure drop across the fritted disk and restricts the flow into the canister even when sampling without a flow controller. Table 2.2.3 lists the typical fill time for a grab sample using a 2 micron particulate filter.



Table 2.2.3 Grab Sample Fill Times for Canisters

CANISTER VOLUME	2 micron filter
6 L	<5 minutes
1 L	<1 minute

2.2.4 Fittings

All fittings on the sampling hardware are 1/4" Swagelok, and a 9/16" wrench is used to assemble the hardware. A 1/2" wrench is also required to tighten fittings onto a union connector. Compression fittings should be used for all connections. Never use tube-in-tube connections. It is critical to avoid leaks in the sampling train. Leaks of ambient air through fittings between pieces of the sampling train will dilute the sample and cause the canister to fill at a faster rate than desired. Eurofins Air Toxics can provide the necessary fittings and ferrules if requested.

2.2.5 Vacuum Gauge

A vacuum gauge is used to measure the initial vacuum of the canister before sampling, and the final vacuum upon completion. A gauge can also be used to monitor the fill rate of the canister when collecting an integrated sample. Eurofins Air Toxics provides 2 types of gauges. For grab sampling, a test gauge checks initial and final vacuums only and is not to be sampled through. For integrated sampling a gauge is built into the flow controller and may be used for monitoring initial and final vacuums, as well as monitoring the fill rate of the canister. Both gauges are considered to be rough gauges, intended to obtain a relative measure of vacuum change. Accuracy of these field gauges are generally on the order of +/- 5 in Hg. Individuals with work plans that outline specific gauge reading requirements are strongly encouraged to purchase and maintain their own gauges in the field. In special cases, a laboratory-grade, NIST-traceable vacuum gauge can be provided upon request.



The vacuum gauges that are routinely provided are intended as a rough gauge measurement device (+/-5 in Hg accuracy).

Section 3.0 Sampling with Canisters

There are two basic modes of canister sampling: grab and integrated. A grab sample is taken over a short interval (i.e., 1-5 minutes) to provide a point-in-time sample concentration, while an integrated sample is taken over a specified duration or utilizing a specified flow rate. In both modes the canister vacuum is used to draw the sample into the canister. This is commonly referred to as passive canister sampling. Sections 3.1 and 3.2 detail procedures for grab and integrated sampling, and section 3.3 provides procedures specific to soil vapor collection.

Regardless of the type of canister samples collected, the following rules apply:

- DO NOT use canister to collect explosive substances, radiological or biological agents, corrosives, extremely toxic substances or other hazardous materials. It is illegal to ship such substances and you will be liable for damages.
- ALWAYS use a filter when sampling. NEVER allow liquids (including water) or corrosive vapors to enter canister.
- DO NOT attach labels to the surface of the canister or write on the canister; you will be liable for cleaning charges.
- DO NOT over tighten the valve, and remember to replace the brass cap.
- IF the canister is returned in unsatisfactory condition, you will be liable for damages.
- DO NOT make modifications to the equipment connections and/or use Teflon tape unless approved by the laboratory.
- AND, if you have any questions or need any support, our experienced project management team is just a phone call away at 800-985-5955.



Use a 9/16" and 1/2" wrench to tighten Swagelok connections on the canister sampling train.

3.1 Grab Sampling Using Canisters

The most common hardware configuration used to take a grab sample is to simply attach a particulate filter to the canister inlet. A particulate filter is shown in section 2.2.3 and is used to prevent particulate matter from fouling the valve and entering the canister.



3.1.1 Step-By-Step Procedures for Canister Grab Sampling

These procedures are for a typical ambient air sampling application; actual field conditions and procedures may vary.

Before you get to the field:

1. Verify contents of the shipped package (e.g., chain-of-custody, canister, particulate filter, and gauge – if requested).
 2. Make sure you include a 9/16" and 1/2" wrench in your field tool kit.
 3. Verify the gauge is working properly.
 4. Verify the initial vacuum of canister as described in the following section:
- **Verify Initial Vacuum of the Canister:** Prior to shipment, each canister is checked for mechanical integrity. However, it is still important to check the vacuum of the canister prior to use. Eurofins Air Toxics recommends doing this before going to the field if possible. The initial vacuum of the canister should be greater than 25 in Hg. If the canister vacuum is less than 25 in Hg, ambient air may have leaked into the canister during storage or transport and the sample may be compromised. Contact your Project Manager if you have any questions on whether to proceed with sample collection. If

sampling at altitude, there are special considerations for gauge readings and sampling (see Section 5.2). The procedure to verify the initial vacuum of a canister is simple but unforgiving.

1. Confirm that valve is closed (knob should already be tightened clockwise).
2. Remove the brass cap.
3. Attach gauge.
4. Attach brass cap to side of gauge tee fitting to ensure a closed train.
5. Open and close valve quickly (a few seconds).
6. Read vacuum on the gauge.
7. Record gauge reading on “Initial Vacuum” column of chain-of-custody.
8. Verify that canister valve is closed and remove gauge.
9. Replace the brass cap.



When ready to sample:

1. Confirm that valve is closed (knob should already be tightened clockwise).
2. Remove brass cap.
3. Attach particulate filter to canister.
4. Open valve 1/2 turn (6 L canister normally takes less than 5 minutes to fill).
5. Close valve by hand tightening knob clockwise.
6. Verify and record final vacuum of canister (repeat steps used to verify initial vacuum). For grab samples, the ending vacuum is typically close to ambient pressure (0 in Hg).
7. Replace brass cap.
8. Fill out canister sample tag (make sure the sample ID and date of collection recorded on the sample tag matches what is recorded on the COC exactly).
9. Return canister in box provided.
10. Return sample media in packaging provided.

11. Fill out chain-of-custody and relinquish samples properly (it is important to note the canister serial numbers on the chain-of-custody).
12. Place chain-of-custody in box and retain pink copy.
13. Tape box shut and affix custody seal (if applicable) across flap.
14. Ship accordingly to meet method holding times.



Return all equipment used or unused to the laboratory. Unreturned canisters and associated hardware will result in additional charges as outlined in the media agreement.

3.2 Integrated Sampling with Canisters and Flow Controllers

As an alternative to an “instantaneous” grab sample, an air sample collected at a controlled rate is referred to as an integrated sample. Flow controllers or flow restrictors are devices which provide sample collection at a desired flow rate and/or sampling interval. By using a flow controller at a specified flow rate, air samples can provide information on average compound concentrations over a defined period. For example, an 8- or 10-hour integrated sample can be used to determine indoor air quality in the workplace. Similarly, a 24-hour integrated sample may be collected to determine residential exposure to indoor or outdoor air sources. In addition to using a flow controller for time-integrated sample collection, a flow controller may be required for soil gas collection to restrict the vacuum applied to the soil and pore water and to collect a representative sample with minimal intrusion of ambient air.

Eurofins Air Toxics provides two general types of flow controllers: mass flow controllers and critical orifice devices. Both devices are driven by differential pressure between ambient conditions and vacuum in the canister.

3.2.1 Mass Flow Controller

A mass flow controller employs a diaphragm that actively compensates to maintain a constant mass flow rate over the desired time period. As the differential pressure decreases, the flow rate decreases and the diaphragm responds by opening up to allow more air to pass through to maintain a stable flow rate. Mass flow controllers are calibrated in the laboratory to provide flow rates suitable for durations up to 24 hours. Durations greater than 24 hours are possible, however, performance of the flow controller is less reliable due to the low flow rates required.



3.2.2 Critical Orifice Devices

Eurofins Air Toxics has two types of critical orifice controllers – “capillary column” and “frit pressed”. Both types restrict the flow rate and the canister fill rate decreases as the canister fills to ambient pressure.



These controllers are suitable for applications not requiring constant flow rate over the sampling period such as soil vapor collection or at sites in which temporal variability of VOCs is not expected. Critical orifice devices can cover intervals from 0.5 to 12 hours and flow rate from 10 to 250

ml/min. The “capillary column” device (also known as the Blue Body Flow Controller) restricts air flow by forcing the sample to enter a capillary column of minute radius. The flow rate is a function of the length of inert capillary column. The frit pressed device has a critical orifice machined to meet a set flow rate.



3.2.3 Sampling Interval and Flow Controller Setting

When you request canisters and flow controllers from Eurofins Air Toxics, you will be asked for the flow rate (soil vapor) or sampling interval (ambient air), and the flow controllers will be pre-set prior to shipment. The flow rate is set at standard atmospheric conditions (approximately sea level and 25°C). If samples will be collected at elevation or at ambient temperatures significantly different than 25°C, the canister will fill faster or slower depending on sample conditions. If you specify unusual sample conditions at the time of project set-up, we can set the flow controller accordingly. (See Section 5.2 for a discussion of collecting a sample at elevation.) Mass flow controllers should not be utilized for source or process samples in which the collection point is under vacuum or pressure. Please discuss these specific non-standard field conditions with your Project Manager at the time of project set-up.

Table 3.2.3 Flow Rates for Selected Sampling Intervals (mL/min)

Sampling Interval (hrs)	4 min.	0.5	1	2	4	8	12	24
6 L Canister	NA	167	83.3	41.7	20.8	11.5	7.6	3.8
1 L Canister	167	26.6	13.3	6.7	-	-	-	-

Note: Target fill volumes for 6 L and 1 L canisters are 5,000 mL and 800 mL, respectively.

3.2.4 Final Canister Vacuum and Flow Controller Performance

For time-integrated sample collection using a mass flow controller, the final vacuum of a canister should ideally be approximately 5 in Hg or greater. The flow rate will remain constant as the canister fills and will start to decrease as the canister vacuum approaches

5 in Hg. At this point, the differential pressure between the canister and ambient air is not sufficient to maintain the set flow rate. Because of normal fluctuations in the flow rate due to changes in field temperature and pressure, the final vacuum typically ranges between 3 and 10 in Hg.

- **If the residual canister vacuum is greater than 10 in Hg** (i.e., more vacuum), the actual flow rate is lower than the set point and less sample volume is collected. When the canister is pressurized prior to analysis, the pressurization dilution will be greater than normal. This will result in elevated reporting limits.
- **If the residual canister vacuum is near ambient pressure** for a time-integrated sample, the canister filled faster than calibrated. Once the vacuum decreases below 5 in Hg, the flow rate begins to decrease from its set point. This scenario indicates that the sample is weighted toward the first portion of the sampling interval. The sampler cannot be certain the desired sampling interval was achieved before the canister arrived at ambient conditions. Although the actual sampling interval is uncertain, the canister still contains a sample from the site.

Table 3.2.4 Relationship between Final Canister Vacuum, Volume Sampled, and Dilution Factor (6 L Canister)

Final Vacuum (in Hg)	0	2.5	5	7.5	10	12.5	15	17.5	20
Volume Sampled (L)	6	5.5	5.4	5	4	3.5	3	2.5	2
Dilution Factor*	1.34	1.46	1.61	1.79	2.01	2.30	2.68	3.22	4.02

*Canister pressurized to 5 psig for analysis

$$\text{Final Reporting Limit} = \text{Method Reporting Limit} \times \text{Dilution Factor (Canister Pressurization)} \times \text{Dilution Factor (Sample Analysis)}$$

$$\text{Dilution Factor (Canister Pressurization)} = \frac{\text{Final Pressure}}{\text{Receipt Pressure}} = \frac{14.7 \text{ psig} + \text{Final Pressure (psig)}}{14.7 \text{ psig}} \left[\frac{1 - \text{Receipt Vacuum (in Hg)}}{29.9 \text{ in Hg}} \right]$$

3.2.5 Considerations for Integrated Sampling with Canisters

Collecting an integrated air sample is more involved than collecting a grab sample. Sampling considerations include verifying that the sampling train is properly configured, monitoring the integrated sampling progress, and avoiding contamination.

- **Avoid Leaks in the Sampling Train:** A leak in any one of these connections means that some air will be pulled in through the leak and not through the flow controller. (Follow the leak check step #4 in 3.2.6).
- **Verify Initial Vacuum of Canister:** See Section 3.1.1 for instructions on verifying initial canister vacuum. A separate gauge is not necessary as both the mass flow controllers and critical orifice flow controllers have built-in rough gauges.
- **Monitor Integrated Sampling Progress:** When feasible, it is a good practice to monitor the progress of the integrated sampling during the sampling interval. The volume of air sampled is a linear function of the canister vacuum. For example, when using a 24-hour mass flow controller, at a quarter of the way (6 hours) into a 24-hour sampling interval, the canister should be a quarter filled (1.25 L) and the gauge should read approximately 6 in Hg lower than



the starting vacuum (~22 in Hg). More vacuum indicates that the canister is filling too slowly; less vacuum means the canister is filling too quickly. If the canister is filling too slowly, a valid sample can still be collected (see Section 3.2.4). If the canister is filling too quickly because of a leak or incorrect flow controller setting, corrective action can be taken. Ensuring all connections are tight may eliminate a leak. It is possible to take an intermittent sample; the time interval need not be continuous.

- **Avoid Contamination:** Flow controllers should be cleaned between uses. This is done by returning them to the laboratory.
- **Caution When Sampling in Extreme Temperatures:** Field temperatures can affect the performance of the mass flow controllers. Laboratory studies have shown that flow rates can increase slightly with decreasing temperatures. A flow rate increase of approximately 10% is expected when sampling at field temperatures of 5 to 10°C.

3.2.6 Step-by-Step Procedures for Integrated Sampling

These procedures are for a typical ambient air sampling application; actual field conditions and procedures may vary.

Before you get to the field:

1. Verify contents of the shipped package (e.g., chain-of-custody, canister, and flow controller)
2. Make sure you include a 9/16" and 1/2" wrench in your field tool kit.
3. Verify the gauge is working properly
4. Verify the initial vacuum of canister (section 3.1.1)

When ready to sample:

1. Confirm that valve is closed (knob should already be tightened clockwise).
2. Remove brass cap from canister.

3. Attach flow controller to canister. The flow controller is securely attached if the flow controller body does not rotate.
4. Place the brass cap at the end of the flow controller creating an air tight train, and quickly open and close the canister valve in order to check for leaks. If the needle on the gauge drops, your train is not airtight. In this case, try refitting your connections and/or tightening them until the needle holds steady.
5. Once the sample train is airtight remove the brass cap from the flow controller and open the canister valve a ½ turn.
6. Monitor integrated sampling progress periodically.
7. Verify and record final vacuum of canister (simply read built-in gauge).
8. When sampling is complete, close valve by hand tightening knob clockwise.
9. Detach flow controller and replace brass cap on canister.
10. Fill out canister sample tag (make sure the sample ID and date of collection recorded on the sample tag matches what is recorded on the COC exactly).
11. Return canisters and associated media in boxes provided. **Failure to return all of the provided equipment will result in a replacement charge as outlined in the media agreement.**
12. Fill out chain-of-custody and relinquish samples properly (it is important to note the canister serial numbers on the chain-of-custody).
13. Place chain-of-custody in box and retain pink copy.
14. Tape box shut and affix custody seal at each opening (if applicable).
15. Ship accordingly to meet method holding times.

3.3 Soil Gas Sample Collection

Canisters can be used for the collection of soil vapor by attaching the sampling train to the soil gas probe. Typically, a critical orifice flow controller is used to minimize the applied vacuum in order to minimize partitioning of VOCs from the soil or pore water to the soil vapor. Additionally, lower flow rates help to minimize the intrusion of ambient air into the soil vapor probe. In general, time-integration is not required for soil gas samples; however, there may be exceptions to this rule of thumb. For example, some regulatory guidance documents recommend concurrent indoor air and sub-slab soil vapor collection over a

24-hour period. This means that a mass flow controller calibrated for a 24-hour sample would be required for the sub-slab as well as the indoor air sample.

3.3.1 Canister to probe connection – Tubing

Collection of a soil gas sample requires the use of tubing to connect the soil gas probe to the sample train. Teflon FEP tubing is recommended based on its low background and its inertness. Alternative tubing can be used if shown to meet data quality objectives. Please note that Low Density Polyethylene or flexible Tygon tubing is not recommended due to VOC adsorption during sample collection. Teflon tubing is provided by the laboratory upon request at the time of order. A charge based on the length will be assessed. It is important to store the tubing away from VOC sources during storage and transport to the site to minimize contamination.

3.3.2 Canister to probe connection –Fittings

To connect the tubing to the canister sampling train, a Swagelok fitting and a pink ferrule are used. The position of the ferrule is key to ensure the fitting is securely connected to the canister. See the figure below for the correct positioning and connection. The pink ferrule is flexible and cannot be over-tightened.



3.3.3 Leak Check Compounds Considerations

To determine whether ambient air is introduced into soil gas sample, a leak check may be used. Leak check compounds may be liquid or gaseous tracers. Liquid compounds are challenging to use effectively in the field and can be introduced into the sample due to improper handling in the field, erroneously indicating a leak in the sampling train. Liquid tracers such as isopropanol should never be directly applied to connections in the sampling train. Rather, the liquid is carefully applied to a cloth and placed near the connection or on the ground next to the probe. Great care must be used in the field to insure the liquid tracer is not handled during sampling train assembly or disassembly. Even a trace amount of a liquid tracer on a glove used to replace a canister brass cap can contaminate the sample. Liquid leak check compounds can interfere with the analytical runs, and even small leaks may result in analytical dilution and raised reporting limits when measuring ppbv target compound levels.

Gaseous tracers such as helium are typically used with shroud placed over the sampling equipment and/or borehole. To quantify the leak, the concentration of the tracer gas in the shroud should be measured.



Specify the leak check compound planned for your soil gas sampling event and record on the COC.

3.3.4 Step-by-Step Procedures for Soil Vapor Sampling

These procedures are for a typical soil vapor sampling application; actual field conditions and procedures may vary. Please consult your specific regulatory guidance for details.

Before you get to the field:

1. Verify contents of the shipped package (e.g., chain-of-custody, canister, tubing, fittings, and flow controller).
2. Make sure you include a 9/16" and 1/2" wrench in your field tool kit.
3. Verify the gauge is working properly.
4. Verify the initial vacuum of canister.

Prior to vapor collection:

- **Purge tubing adequately.** A long length of tubing has significant volume of "dead air" inside. Without purging, this air will enter the canister and dilute the sample. Consider using a handheld PID/FID to confirm that you have purged the tubing and are drawing sample air through the tubing. A standard rule of thumb is to utilize 3 purge volumes prior to sample collection. However, under certain circumstances, purge volumes of 1 to 10 may be appropriate. Please review your regulatory guidance and your site specific conditions in determining the appropriate purge volumes.
- **Don't sample water.** If moisture is visible in the sample tubing, the soil gas sample may be compromised. Soil gas probes should be at an appropriate depth to avoid reaching the water table. Additionally, subsurface vapor should not be collected immediately after measurable precipitation.

When ready to sample:

1. Confirm that valve is closed (knob should already be tightened clockwise).
2. Remove brass cap from canister.
3. Attach flow controller to canister if needed. The flow controller is securely attached if the flow controller body does not rotate. (Note: The frit-press flow controller and 1 L canister may be pre-assembled by the laboratory.)
4. Place the brass cap at the end of the flow controller creating an air tight train, and quickly open and close the canister valve in order to check for leaks. If the needle on the

gauge drops, your train is not airtight. In this case, try refitting your connections and/or tightening them until the needle holds steady.

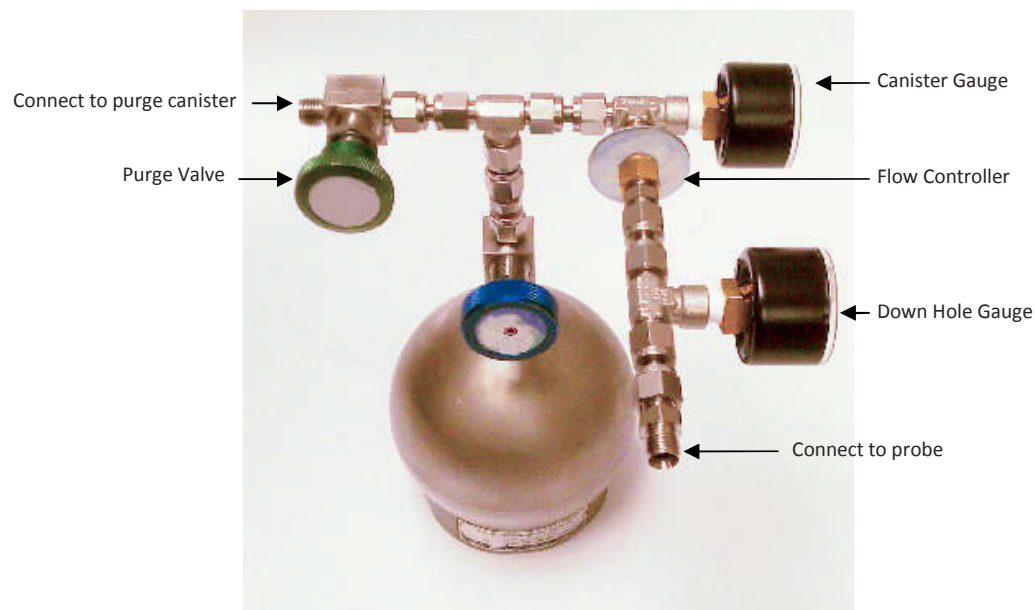
5. Once the sample train is airtight remove the brass cap from the flow controller and attach the probe tubing to the flow controller using the pink ferrule and Swagelok nut. (See 3.3.2 for proper positioning of the ferrule.)
6. Once the probe line has been purged and appropriate leak check measures have been implemented, open the canister valve a ½ turn.
7. Verify and record final vacuum of canister (simply read built-in gauge).
8. When canister fills to the desired end vacuum, close valve by hand tightening knob clockwise.

Please note: Some projects require residual vacuum of approximately 5 in Hg at the end of sample collection even if time-integrated samples are not required. The residual vacuum serves to provide a check of the integrity of the canister during transport to the laboratory to insure no leaks occurred during shipment. A field vacuum reading similar to the lab receipt vacuum reading demonstrated that no leak occurred.

9. Detach tubing and flow controller and replace brass cap on the canister.
10. Fill out canister sample tag (make sure the sample ID and date of collection recorded on the sample tag matches what is recorded on the COC exactly).
11. Return canisters and associated media in boxes provided. **Failure to return all of the provided equipment will result in a replacement charge as outlined in the media agreement.**
12. Fill out chain-of-custody and relinquish samples properly (it is important to note the canister serial numbers on the chain-of-custody).
13. Place chain-of-custody in box and retain pink copy
14. Tape box shut and affix custody seal at each opening (if applicable)
15. Ship accordingly to meet method holding times

3.4.4 Collecting Soil Gas Samples with Sampling Manifolds

If required, Eurofins Air Toxics can provide a sampling manifold to assist with leak checking the sampling train, purging the sampling line, and monitoring the vacuum applied to the soil gas bore hole during sample collection. The manifold is shown below:



The ‘Down Hole Gauge’, located prior to the flow restrictor, is a vacuum gauge that monitors the vacuum applied to the soil gas probe. Because this is not a flow meter but a measure of pressure/vacuum, the gauge should read at zero if there is sufficient flow from the soil. If the gauge begins to read a vacuum, then the flow is being restricted. Low flow, high vacuum conditions can be encountered when sampling in low permeability soil. The ‘Canister Gauge’, in line after the flow controller and prior to the purge canister, is a vacuum gauge that indicates to the sampler whether or not the canister is filling properly at the expected rate. This setup enables the sampler to evaluate the lithologic conditions at the site and determine if a valid soil gas sample is being taken. Finally, when duplicate

samples are required, the manifold can be used as a duplicate sampling “T” by simply replacing the purge canister with another sample canister.

There are several options to use as a purge vacuum source to attach to the purge valve connection – a Summa canister, sampling pump or sampling syringe. The below instructions assume a Summa canister will be used as a purge volume source since other sources are generally provided by the client.

When ready to sample:

Leak Check Test

1. Confirm that canister valves are closed (knob should already be tightened clockwise).
2. Remove brass caps from both the sample canister and the purge canister. (Unless using certified media, there is no difference between the two).
3. Attach manifold center fitting to sample canister.
4. Attach purge canister to the Purge Valve end of the manifold by attaching provided Teflon tubing and compression fittings.
5. Confirm that there is a brass cap secured at the inlet of the manifold creating an air tight train, make sure the manifold valve above the purge canister is open, and quickly open and close the purge canister valve in order to check for leaks. If the needle on the gauge drops, your train is not airtight. In this case, try refitting your connections and/or tightening them until the needle holds steady.

Purging

6. Once the sample train is airtight remove the brass cap from the manifold inlet, connect the tubing from the sample port using a compression fitting and open the purge canister valve, 1/2 turn.
7. Monitor integrated sampling progress periodically. *Please note, because the purge canister is inline after the flow restrictor the line will not purge faster than at a rate of 167 ml/min.

8. Once the desired purge volume is met close both the manifold valve and the purge canister valve by hand tightening the knobs clockwise.
9. If sampling at multiple locations, the purge canister can be disconnected from the manifold and used to begin purging the next sample location without compromising the sample train.

Sampling

10. The line is now ready to be sampled. Open the sample canister valve and monitor sampling progress periodically.
11. When the sampling is complete close the valve and replace the brass cap on the canister; record final vacuum of canister (simply read built-in gauge).
12. Fill out canister sample tag (make sure the sample ID and date of collection recorded on the sample tag matches what is recorded on the COC exactly).
13. Return canisters in boxes provided and all parts of the soil gas manifold. **Unreturned media will result in a replacement charged assessed as described in the media agreement.**
14. Fill out chain-of-custody and relinquish samples properly (it is important to note the canister serial numbers on the chain-of-custody).
15. Place chain-of-custody in box and retain pink copy.
16. Ship accordingly to meet method holding times.

Section 4.0 Sampling with Bags

This section provides a description of the types of air sampling bags, selecting the right bag for your application, practical considerations for sampling, and step-by-step instructions for collecting a grab sample. Photographs illustrate the correct way to assemble the various sampling components.

4.1 Introduction to Bags

Air sampling bags are containers used to collect whole air samples for landfill gas, soil gas and stationary source applications. Bags can be constructed from various materials which can differ in terms of stability characteristics and cleanliness. In general, air sampling bags are best suited for projects involving analysis of compounds in the ppmv range. They can be used to collect sulfur compounds, but only if the fittings are non-metallic (e.g., polypropylene, Teflon®, or Nylon).

Air sampling bags are equipped with a valve that allows for filling. Sample collection requires a pressurized sampling port, a low flow rate pump or a lung sampler. The bag expands as the vapor sample is pulled in. When the target volume of the sample is collected, the valve is closed and the bag is returned to the laboratory. Bag materials should be selected based on the specific application. Common air sampling bags include Tedlar film and FlexFoil. Eurofins Air Toxics maintains a limited inventory of air sampling bags in 1 L, 3 L and 5 L volumes.

4.1.1 Tedlar® Film

Tedlar® is a trade name for a polyvinyl fluoride film developed by DuPont Corporation in the 1960's. This patented fluoropolymer has been used in a wide variety of applications including protective surfacing for signs, exterior wall panels and aircraft interiors. Tedlar® film is tough yet flexible and retains its impressive mechanical properties over a wide range

of temperatures (from well below freezing to over 200°F). Tedlar® exhibits low permeability to gases, good chemical inertness, good weathering resistance and low off-gassing.

Tedlar® bags may be used to collect samples containing common solvents, hydrocarbons, chlorinated solvents, sulfur compounds, atmospheric and biogenic gases and many other classes of compounds. Compounds with low vapor pressures such as Naphthalene are not appropriate for Tedlar bags as recovery is very low even under short sample storage times. Low molecular compounds such as Helium and Hydrogen can diffuse through the Tedlar bag material resulting in poor storage stability.



4.1.2 Tedlar® Bag Suppliers and Re-use

Compounds commonly detected from analyzing new Tedlar® bags include methylene chloride, toluene, acetone, ethanol, 2-propanol, phenol, and dimethylacetamide. While levels of these common artifacts are typically in the ppbv range, the cleanliness of bags can vary significantly between vendors, and purchasing bags directly from an unknown vendor should be avoided. Once the Tedlar® bag is used for sample collection, the surface has been exposed to moisture and possible VOCs. It may irreversibly adsorb many VOCs at the low ppbv level. A series of purges with certified gas may not remove the VOCs from the surface. Consider your data quality objectives to determine whether re-using Tedlar® bags is appropriate.

4.1.3 Hold Time for a Tedlar® Bag

The media hold time for a Tedlar® bag is indefinite if stored out of sunlight in a cool, dry location.

The sample hold time to analysis varies by method and compound. See Table 4.1.3 for recommended sample storage times for commonly requested parameters.

Table 4.1.3 Recommended Maximum Sample Storage Times for Tedlar® Bags

Analytical Method	Chemical Class	Storage Time
ASTM D5504	Suite of sulfur compounds including Reactive Sulfur compounds (Hydrogen sulfide, Methyl mercaptan)	24 hours
ASTM D1946 ASTM D1945	Atmospheric and natural gases: CO, CO ₂ , CH ₄ , C ₂ -C ₅ hydrocarbons (He and H ₂ not recommended)	Up to 3 days
Modified TO-14A, TO-15, TO-3, TO-12	Volatile Organic Compounds (VOCs)	Up to 3 days

4.1.4 FlexFoil Bags

FlexFoil bags are made from an opaque and flexible material with 4-ply construction resulting in high physical strength to minimize rupture and leakage and low permeability to provide good stability for low molecular weight compounds. FlexFoil bags are ideal for target compounds such as Hydrogen and Helium and can be used for the suite of atmospheric and natural gas components. While the reactive sulfur compounds, Hydrogen Sulfide and Methyl Mercaptan, show good stability over 24 hours in FlexFoil bags, other sulfur compounds demonstrate low recovery. Table 4.1.4 summarizes the compounds and the hold times amenable to FlexFoil bags.

Table 4.1.4 Recommended Maximum Sample Storage Times for FlexFoil Bags

Analytical Method	Chemical Class	Storage Time
ASTM D5504	Hydrogen sulfide, Methyl mercaptan only Not recommended for full sulfur list.	24 hours
ASTM D1946 ASTM D1945	Atmospheric and natural gases Full List	Up to 3 days

4.2 Air Bag Sampling

Using a bag to collect an air sample normally involves “active” sampling, unlike an evacuated canister that can be filled “passively” by simply opening the valve. There are two methods commonly used to fill a bag: a pump or a lung sampler.

- Sampling with a Pump:** The most common method for filling a bag is to use a small pump with low flow rates (50-200 mL/min) and tubing to fill the bag. Eurofins Air Toxics, Inc. does not provide pumps but pumps may be rented from equipment providers or purchased from manufacturers such as SKC or Gilian.
- Sampling with a Lung Sampler:** A “lung sampler” may be used to fill a bag. Although a little more complicated than simply using a pump, the main advantage to using a lung sampler to fill a bag is that it avoids potential pump contamination.



A bag with attached tubing is placed in a small airtight chamber (even a 5-gallon bucket can work) with the tubing protruding from the chamber. The sealed chamber is then evacuated via a pump, causing the bag to expand and draw the sample into the bag through the protruding tube. The sample air never touches the wetted surfaces of the pump. Eurofins Air Toxics does not provide lung samplers, but they can be rented from equipment suppliers or purchased by manufacturers such as SKC Inc.

4.2.1 Considerations for Bag Sampling

Some considerations for collecting a bag sample:

- **Fill the bag no more than 2/3 full:** Allow for possible expansion due to an increase in temperature or decrease in atmospheric pressure (e.g., the cargo hold of a plane)
- **Keep the Tedlar® bag out of sunlight:** Tedlar® film is transparent to ultraviolet light (although opaque versions are available) and the sample should be kept out of sunlight to avoid any photochemical reactions
- **Protect the bag:** Store and ship the bag samples in a protective box at room temperature. An ice chest may be used, but DO NOT CHILL
- **Fill out the bag label:** It is much easier to write the sample information on the label before the bag is inflated. Make sure to use a ball-point pen, never a Sharpee or other marker which can emit VOCs.
- **Provide a “back-up” bag:** Consider filling two bags per location in the rare occasion that a defective bag deflates before analysis. The “hold” sample does not need to be documented on the Chain-of-Custody and should have an identical sample ID to the original sample indicating that it is the “hold” sample
- **Avoid Contamination:** Care should be taken to avoid contamination introduced by the pump or tubing. Begin sampling at locations with the lowest compound concentrations (e.g., sample the SVE effluent before the influent). Decontaminate the pump between uses by purging with certified air for an extended period; better yet, use a lung sampler. Use the shortest length possible of Teflon® tubing or other inert tubing. DO NOT REUSE TUBING. If long lengths of tubing are used, consider purging the tubing with several

volumes worth before sampling. If you are concerned about sampling for trace compounds, you shouldn't be using a Tedlar® bag (see Section 1.2)

- **Don't Sample Dangerous Compounds in a Bag:** Do not ship any explosive substances, radiological or biological agents, corrosives or extremely hazardous materials to Eurofins Air Toxics. Bag rupture during transit to the laboratory is possible and the sampler assumes full liability.

4.2.2 Step-by-Step Procedures for Bag Sampling (Pump)

Note: These procedures are for a typical stationary source (e.g., SVE system) sampling application; actual field conditions and procedures may vary.

Before you get to the field:

1. Verify contents of the shipped package (e.g., chain-of-custody, bag, and tubing/fittings – if requested).
2. Verify pump cleanliness and operation (Eurofins Air Toxics does not provide pumps).

When ready to sample:

3. Purge sample port.
4. Attach new Teflon® tubing from sample port or probe to low flow rate pump.
5. Purge tubing.
6. Fill out bag sample tag.
7. Attach additional new Teflon® tubing from the pump outlet to the bag valve.
8. Open bag valve.
9. Collect sample (FILL NO MORE THAN 2/3 FULL).
10. Close bag valve by hand tightening valve clockwise.
11. Return filled bags in a rigid shipping container (DO NOT CHILL).
12. Fill out chain-of-custody and relinquish samples properly.
13. Place chain-of-custody in box and retain pink copy.

14. Tape box shut and affix custody seal (if applicable) across flap.
15. Ship first overnight or priority overnight to meet method holding times.



Expedite delivery of air sampling bags to the laboratory for analysis.

Section 5.0 Special Sampling Considerations

This section provides recommendations for the collection of field QC samples such as field duplicates. Considerations for sampling at altitude, sampling SVE ports and using sample cylinders are presented.

5.1 Field QC

To measure accuracy and precision of the field activities, project plans often include field duplicates, field blanks, ambient blanks, trip blanks and/or equipment blanks.

5.1.1 Field Duplicate

A field duplicate is a second sample collected in the field simultaneously with the primary sample at one sampling location. The results of the duplicate sample may be compared (e.g., calculate relative percent difference) with the primary sample to provide information on consistency and reproducibility of field sampling procedures. Due to the nature of the gas phase, duplicate samples should be collected from a common inlet. The configuration for collecting a field duplicate includes stainless steel or Teflon® tubing connected to a Swagelok “T”. If integrated samples are being collected and the sample duration is to be maintained, the sample train should be assembled as follows: each canister should have a flow controller attached, then the duplicate sampling T should be attached to the flow controllers. If the collection flow rate from the sample port is to be maintained then the

duplicate sampling T should be connected to the canisters; then the flow controller is connected to the inlet of the sampling T.

Alternatively, if the project objective is to assess spatial or temporal variability, then field duplicates may be deployed in close proximity (ambient air sampling) or samples may be collected in succession (soil vapor).

5.1.2 Field Blank

A field blank is a sample collected in the field from a certified air source. Analysis of the field blank can provide information on the decontamination procedures used in the field. Clean stainless steel or Teflon® tubing and a certified regulator should be used. It is imperative that individually certified canisters (the sample canister and the source canister/cylinder, if applicable) be used to collect a field blank.

5.1.3 Ambient Blank

An ambient blank is an ambient air sample collected in the field. It is usually used in conjunction with soil gas or stationary source (e.g., SVE system) sampling. Analysis of the ambient blank can provide information on the ambient levels of site contaminants. It is recommended that an individually certified canister be used to collect an ambient blank.

5.1.4 Trip Blank

When sampling for contaminants in water, the laboratory prepares a trip blank by filling a VOA vial with clean, de-ionized water. The trip blank is sent to the field in a cooler with new sample vials. After sampling, the filled sample vials are placed back in the cooler next to the trip blank and returned to the laboratory. Analysis of the trip blank provides information on decontamination and sample handling procedures in the field as well as the cleanliness of the cooler and packaging.

When sampling for compounds in air, a trip blank provides little, if any, of the information above. A trip blank canister can be individually certified, evacuated, and sent to the field in a box with the sample canisters. Since the valve is closed and the brass cap tightened, it is questionable if the trip blank canister contents are ever “exposed” to sampling conditions. The trip blank VOC concentrations essentially provide information regarding the cleanliness and performance of the trip blank canister. Results cannot necessarily be applied to the associated field sample canisters accompanying the trip blank. **Eurofins Air Toxics does not recommend collecting a trip blank for air sampling.**

5.2 Considerations for Sampling at Altitude

Sampling at altitudes significantly above sea level is similar to sampling a stationary source under vacuum in that target fill volumes may be difficult to achieve. The figure to the right illustrates the relationship between increasing altitude and decreasing atmospheric pressure. Ambient conditions in Denver at 5,000 ft altitude are quite different from ambient conditions at sea level. Canister sampling is driven by the differential pressure between ambient conditions and the vacuum in the canister.

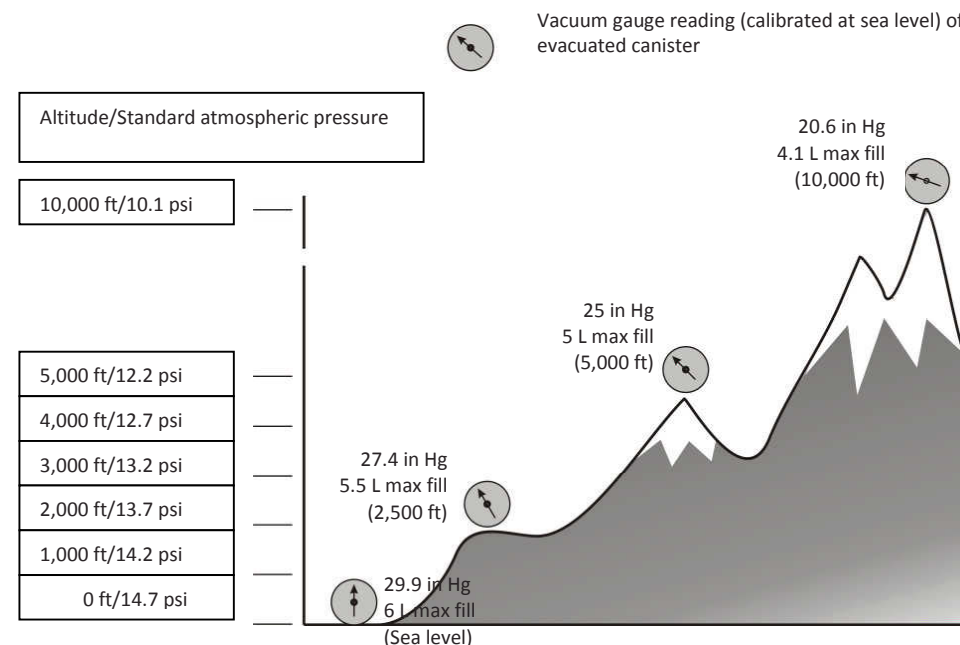
There is less atmospheric pressure in Denver and 5 L is the maximum fill volume of standard air assuming the canister is allowed to reach ambient conditions (i.e., final gauge reading of 0 in Hg). Theoretically, if you sample high enough (e.g., in space), no sample would enter the canister because there is no pressure difference between the evacuated canister and ambient conditions. To fill a canister to 6 L in Denver, you would need to use an air pump.

Sampling at altitude also affects gauge readings. The gauges supplied by Eurofins Air Toxics, Inc. (see Section 2.2.4) measure canister vacuum relative to atmospheric pressure and are calibrated at approximately sea level. Before sampling at altitude, the gauges should be equilibrated (see Section 3.1). But even after equilibrating the gauge, verifying the initial vacuum of a canister at altitude is misleading. In Denver at 5,000 ft, expect the gauge to read 25, not 29.9 in Hg. You do not have a bad canister (i.e., leaking or not evacuated properly). The canister is ready for sampling and the gauge is working properly.



Rule of Thumb: For every 1,000 ft of elevation, the gauge will be off by 1 in Hg and the fill volume will be reduced by 1/5 L.

If you have questions about sampling at altitude, please call your Project Manager at 800-985-5955.



5.3 Considerations for SVE/LFG Collection System Sampling

There are some additional sampling considerations for collecting grab samples (canister or bag) from a Soil Vapor Extraction (SVE) system or landfill gas (LFG) collection system. The general challenge with these samples arises from the need to employ a length of tubing to direct the landfill gas or process air to the canister or bag. Tubing introduces the potential for contamination and diluting the sample.

- **Use inert tubing.** Teflon® tubing is recommended. Tubing with an outer diameter of ¼” works best with the fittings on the particulate filter. (See Section 3.3.1).
- **Do not reuse tubing.**
- **Purge tubing adequately.** A long length of tubing has significant volume of “dead air” inside. Without purging, this air will enter the canister and dilute the sample. Consider using a handheld PID/FID to confirm that you have purged the tubing and are drawing sample air through the tubing.
- **Avoid leaks in the sampling train.** Leaks of ambient air through fittings between pieces of the sampling train (e.g., tubing to particulate filter) will dilute the sample.
- Always use compression fittings for all connections; never use tube in tube connections.
- **Purge the sample port.** A sample port on an SVE system or LFG collection system can accumulate solids or liquids depending upon the location of the port in the process and the orientation of the port. An influent sample port located upstream of a filter or moisture knock-out can be laden with particulates or saturated with water vapor. Heavy particulate matter can clog the particulate filter and foul the canister valve. It is important to prevent liquids from entering the canister. A sample port oriented downward may have liquid standing in the valve. Purge the sample port adequately before connecting the sampling train.
- **Consider the effects of sampling a process under vacuum or pressure.** When collecting a grab sample from a stationary source such as an SVE system or LFG collection system, some sample ports may be under vacuum or pressure relative to ambient conditions. When the sample port is under vacuum, such as the header pipe from the extraction well network, it may be difficult to fill the canister with the desired volume of sample. A vacuum pump may be used to collect a canister grab sample from a sample port under considerable vacuum. See the related discussion on sampling at altitude in Section 5.2. When the sample port is under pressure, such as the effluent stack downstream of the blower and treatment system, you may inadvertently pressurize the canister. Only a DOT-approved sample cylinder should be used to transport pressurized air samples (see Section 5.4). Under no circumstances should a Summa canister be pressurized more than 15 psig. Bleed off excess pressure by opening the valve temporarily while monitoring the canister with a pressure gauge.

5.4 Considerations for Sample Cylinder Sampling

Sample cylinders, also known as “sample bombs”, are DOT-approved, high pressure, thick-walled, stainless steel cylinders with a valve at each end. They were intended for collecting a pressurized sample for petroleum gas applications. Sample cylinders differ from sample canisters in that they do not have a Summa-passivated interior surface and are not evacuated prior to shipment. Sample cylinders are not suitable for analysis of hydrocarbons at ppbv levels. Sample cylinders can be used for analysis of natural gas by ASTM D-1945 and calculation of BTU by ASTM D-3588. Eurofins Air Toxics assumes that clients requesting a sample cylinder have a pressurized process and sample port with a built-in gauge and 1/4” Swagelok fitting to attach to the sample cylinder. Eurofins Air Toxics has a limited inventory of 500 mL sample cylinders that are particularly suited for landfill gas collection systems (i.e., LFG to energy applications). This section provides step-by-step procedures for sampling with a sample cylinder.



Inform the lab during project set up if hazardous samples (e.g. high Hydrogen Sulfide concentrations) will be collected to verify the lab can safely handle the samples.

Step-by-Step Procedures for Sample Cylinder Sampling

These procedures are for a typical stationary source sampling application and actual field conditions; procedures may vary. Follow all precautions in the site Health and Safety Plan when dealing with a pressurized sample port and sample cylinder. Follow required DOT guidelines for packaging and shipping.

1. Verify contents of the shipped package (e.g., chain-of-custody, sample cylinder, particulate filter).
2. Verify that gauge on sample port is working properly.
3. Purge sample port.

4. Remove brass caps on either end of cylinder.
5. Attach particulate filter to upstream valve.
6. Attach filter/cylinder assembly directly to the sample port.
7. Open both valves 1/2 turn.
8. Allow sample air to flow through sample cylinder (approximately 10 L for a 500 mL cylinder).
9. Close downstream valve of sample cylinder by hand tightening knob clockwise.
10. Allow sample cylinder to pressurize to process pressure (max 100 psig).
11. Close upstream valve of sample cylinder and sample port.
12. Detach filter/cylinder assembly from sample port and remove particulate filter.
13. Replace brass caps.
14. Fill out sample cylinder sample tag.
15. Fill out chain-of-custody and relinquish samples properly.
16. Include the chain-of-custody with the samples and retain pink copy.
17. Pack, label, and ship according to DOT regulations.



Follow DOT regulations for packaging and shipping hazardous samples.

Standard Operating Procedure (SOP) #2

Soil Gas Sampling

Introduction

Reference guidance provided in the main Quality Assurance Project Plan (QAPP). This SOP was developed using the following guidelines as references:

- California Department of Toxic Substances Control (DTSC) and California Environmental Protection Agency (Cal EPA), 2011. *Final Guidance for the Evaluation and Mitigation of Subsurface Vapor Intrusion to Indoor Air (Vapor Intrusion Guidance)*. October. Available from: https://dtsc.ca.gov/wp-content/uploads/sites/31/2018/01/Final_VIG_Oct_2011.pdf;
- DTSC/Cal EPA, 2015. *Advisory Active Soil Gas Investigations*. April. Available from: https://dtsc.ca.gov/wp-content/uploads/sites/31/2018/01/VI_ActiveSoilGasAdvisory_FINAL.pdf;
- and
- Department of Defense (DoD), 2009. *DoD Vapor Intrusion Handbook*. January. Available from: <http://www.clu-in.org/download/char/dodvihdbk200901.pdf>.

Since Site 12 is located in a public retail location, the likelihood of sampling site visitors is high. Visitors to the sampling site may include shareholders such as project personnel, clients, regulators, property owners or managers, property employees, property customers, other temporary site workers (e.g., construction workers, delivery drivers, painters, landscapers, etc.), or members of the public. If there are visitors to the site during sampling, who ask questions, direct them to the BRAC Office Community Relations department or fortordcleanup.com. Unauthorized visitors may not enter a safety exclusion zone. Notify project manager of any visitor encounters.

Materials

The following materials should be utilized during soil gas sampling:

- Site Setup
 - Tools to open well if it is a flush mount well box (3/4" or 15/16" wrench usually)
 - Cones for delineation of exclusion zone
- Soil Gas Probe Purging
 - Vacuum pump
 - Vacuum gauge
 - Tubing (small piece of Silicone and long piece of Nylon tubing)
- Soil Gas Probe Integrity Test (Helium Test)
 - Helium compressed gas cylinder (can be provided by lab)
 - Helium cylinder regulator and tubing with connection to shroud (can be provided by lab)
 - In-line helium detector (can be provided by lab)
 - Shroud helium detector (can be provided by lab)
 - Shroud assembly (provided by lab) Plastic sheeting to cover shroud
 - Weights to hold shroud down

- Cardboard cover for stickup wells
- Sampling
 - Sample containers – 1.0 Liter (L) SUMMA canisters provided by laboratory
 - Flow regulator – between 100 and 200 milliliters per minute (mL/min) (usually a part of the shroud sampling manifold assembly)
 - Sample manifold (T-manifold for duplicate samples) provided by laboratory
 - Vacuum gauge
 - Tools (9/16" wrench to remove canister caps and attach gauges, tubing, and/or flow regulators, tubing cutter)
 - Tubing
 - 1/4" Nylon or Teflon approximately 1 foot (ft) per probe and
 - 3/8" Silicone tubing approximately 3" per probe
 - Small zipties
 - Ferrules and 1/4" compression caps (extras in case lab provided ones don't work)
- Documentation
 - Chain of Custodies (COCs)
 - Sample Logbook
 - Soil Gas Sample Collection Log
 - SUMMA Sample Train Shut-In Test Log
 - Soil Gas Probe Integrity Testing Log
 - Sample labels (typically attached to sample canisters provided by laboratory)
- Sample Handling
 - Shipping boxes
 - Shipping labels
 - Custody seals
 - Laboratory contact information

Sample Locations and Schedule

Soil gas sampling shall be conducted in the Site 12 area at soil gas probes installed below ground surface according to the sampling schedule provided in the QAPP Table 2. Soil gas sampling should not be conducted during a significant rain event, which is defined as 1/2 inch or greater of precipitation in a 24 hour period unless infiltration has not occurred beneath high-integrity pavement. Shallow soil gas probes (10-foot probes) should not be sampled within five days of a significant rain event.

Sampling Procedures

The following procedures will be followed for each soil gas probe sample collected:

1. Gather materials listed above.
2. Once onsite, follow safety guidelines in the Accident Prevention Plan. Wear high visibility safety vest, steel toe boots, and eye protection as necessary. Setup safety cones around the site to delineate pedestrians and vehicles around the sampling site. Do not block building entrances or exits or parked vehicles from exiting.

3. Install a vacuum gauge on the SUMMA canister to check for proper vacuum (minimum -25 inches mercury [”Hg]).
4. Before sampling begins, attach the SUMMA canister to the sample manifold fitting, making sure the sample manifold assembly flow valve is turned off.
5. Perform the Equipment Test (Shut-In Test) by observing the starting vacuum does not drop over 1”Hg after 15 minutes of observation.
6. After the Shut-In Test passes, setup shroud in soil gas sample location. If the Shut-In Test fails, re-assemble connections, and retry test. If the initial vacuum is now less than the minimum - 25”Hg, do not use the canister. Try a different flow regulator or different ferrules and compression caps if necessary. Contact the laboratory for further guidance if Shut-In Test failures become a consistent problem.
7. Once shroud is in place, attach soil gas probe tubing to Silicone and Nylon tubing (securing tubing connections with small zipties) and attach to sample manifold inlet compression fitting with compression cap and ferrule. Tighten connections with appropriate wrench to a ¼ turn after it is hand tight, do not over tighten.
8. Each soil gas probe must be purged for a calculated volume of soil gas prior to sampling by the purge volumes listed in QAPP Table 1. Attach the vacuum pump and gauge assembly to the outlet of the sample manifold and turn the sample manifold valve to purge. Make sure flow rates are at or below 200 mL/min¹ for 10 foot soil gas probes and at or below 400 mL/min² for 20 foot or deeper soil gas probes.
9. During the soil gas probe purging, the Soil Gas Probe Integrity Test (SGPI Test or Helium Test) may be completed. This SGPI Test will take 15 minutes to complete if it passes. Attach the helium compressed gas cylinder to the regulator and connect it to the helium inlet on the shroud. Insert the shroud helium detector in the shroud and turn it on, it should read 0 percent helium (% He). Turn on the inline helium detector and record the %He reading on the SGPI Test form. Attach the inline helium detector to the sample manifold outlet tubing before the vacuum pump.
10. Windy conditions can cause leaks in the shroud and use more helium than desired, as possible block any gaps and weigh down the shroud to prevent helium leaks.
11. Begin applying helium to the shroud at approximately 30% He (as measured by the shroud helium detector). Do not to let the helium go below 20% He in the shroud during the 15 minute SGPI Test, so apply helium during test as necessary.
12. Record the shroud and inline helium detector readings every 5 minutes for 15 minutes. A 5% ambient air leak is acceptable, therefore when the shroud helium detector reads 20% He, a 1% He reading from the inline detector is acceptable. If all measurements pass, then the SGPI Test is passed. If one of the measurements does not pass, then corrective action is necessary prior to

¹ 200 mL/min was selected for 10-foot soil gas probes based on guidance (DTSC, 2012) suggesting 100 to 200 mL/min purge rates and because these probes are closest to the surface where ambient air is more likely to dilute soil gas.

² 400 mL/min was selected for deeper soil gas probes based on guidance (DTSC, 2012) suggesting flow rates greater than 200 mL/min may be used when purging times are excessive for deeper wells or larger diameter tubing. A vacuum of 100” water or 7.4” Hg should not be exceeded.

sampling similar to the Shut-In Test corrective action. Sometimes laboratory provided shroud assemblies have a leak that may not be remedied in the field, in this case a new shroud assembly should be attempted.

13. If the SGPI test cannot be completed, the sample may be analyzed for He by the laboratory to determine if there were leaks during sampling. If analyzing for He in the lab, the shroud must be filled with He to approximately 20% He during the time the sample is being collected.
14. After the allotted soil gas purge time is completed, sampling may begin by turning the sample manifold valve to the sample position, record the sample collection start time and vacuum. Make sure sampling flow rates are between 100 and 200 mL/min and vacuum rate is not above 100 inches water ("H₂O) or 7.4 "Hg. The laboratory provided flow regulators are usually already set for this, check with lab for compliance.
15. During soil gas sampling, periodically monitor the sample for vacuum remaining to make sure to turn it off at approximately between -4 and -8"Hg or as recommended by the laboratory. Depending on the formation of the subsurface conditions, sampling may take longer at different probes. Calculated sample time for a 200 mL/min flow rate and a 1.0L canister is 5 minutes.
16. Once sampling is completed, shut off shroud valve, record sample collection end time and vacuum.
17. Disassemble sample manifold and shroud and cap SUMMA canister (if necessary) and replace well cover.
18. Follow documentation and sample handling procedures below.

Duplicate Samples

Duplicate samples will be collected at a frequency of 10% for each quarterly event. Duplicate samples must be collected at the same soil gas probe as the original sample and at the same time using a duplicate "T" splitter sample manifold provided by the lab.

Documentation

Documentation includes the sampler's logbook, COCs, sample labels, and sampling forms. Each type of documentation will include at a minimum for each soil gas sample taken:

- Sampler's name (first initial and last name);
- Sample date;
- Sample time (in military time format, start time of collection);
- Sample identification (as described below);
- Canister identification (number should be labeled or stamped on canister by lab)
- Analytes (PCE and TCE); and
- Analysis method (EPA TO-15).

The sampler's logbook will also have additional information including:

- Project name (Site 12 Soil Gas Monitoring);
- Sampling event (e.g., 2015-3Q);

- Weather conditions (e.g., Sunny low 70's, wind approx. 15 mph from the NW, overcast and upper 60s after 1400);
- Sample location description;
- Probe ID
- Canister ID (number should be labeled or stamped on canister by lab);
- Canister equipment test (shut-in test) results (pass or fail);
- Flow meter ID (number should be labeled or stamped on canister by lab);
- Canister volume (typically 6L);
- Sample volume (start and end vacuum in inches mercury ["Hg]);
- Sample duration (start and end sampling times); and
- Identify any conditions that may affect the sample representativeness (e.g., proximity of vehicles to the sample).

Sample form Soil Gas Sample Collection Log should be filled out accordingly, one per soil gas sample. One line on a COC for each soil gas sample should also be filled out accordingly. The SUMMA Sample Train Shut-In Test Log should also have a record for the passed soil gas sample train equipment test. The Soil Gas Probe Integrity Testing Log should also have a record for the passed soil gas probe helium test.

Sample numbering shall follow the same convention as the Groundwater QAPP numbering system which is described below:

Example: 1535M212204F

- 15: This is a two digit reference to the year the sample is collected in
- 35: This is a two digit reference to the week of the year the sample is collected in
- M: This is a one letter reference to the sampler's identity
- 212: This is a three digit reference to the sample site (Sites 2/12)
- 204: This is a three digit reference to sample number collected by that sampler for the year (e.g., this is sampler M's 204th sample in 2015)
- F: This is a one letter reference to the type of sample as described below:
 - F: Field Sample: a regular soil gas sample
 - D: Duplicate Sample: a duplicate soil gas sample

Sampling Handling

Once the sample has been collected and it is secured with all documentation in place, notify the lab of expected sample transfer method. EPA Method TO-15 has a hold time of 30 calendar days from the time of sample collection (stop time), however laboratories recommend returning SUMMA canisters within 15 days of receipt for quality assurance. Samples are typically collected by a laboratory courier. No preservation or ice is required, so a cooler is not necessary. Make sure the canister compression cap is securely tightened and the finish vacuum is recorded on all documentation. Include the COC with the samples. The laboratory will send a sample receipt confirmation with the identity and conditions of the sample received along with an estimated date the results will be available.



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Eurofins Air Toxics Helium Shroud Spec Sheet

Items included:

- Two hand, zipper lock closure inflatable glove box bag. Dimensions: 32 in. × 42 in. average inflated height 12 in. Zippered opening diameter is 19.5 in.
- Port to cover soil vapor well 12 cm diameter at ground level.
- 2 additional ports allow for ¼" tubing to be inserted for charging the shroud with helium and purging with an external canister or pump.
- Zip ties to hold tubing in place

Instructions for use:

1. Open the zippered end of the shroud bag and place port 1 over the soil vapor point. Seal to the ground as needed.
2. Reach into bag and Push port 2 outwards, and snip a hole in the end. Place the tubing from your Helium tank or canister into this port, and zip tie securely.
3. Push port 3 outwards, and snip a hole in the end. Place the purge line through port 2, and zip tie securely.
4. Place your sample canister and manifold into the shroud through the zippered opening. Attach the purge tubing to the purge valve on the manifold. Attach the sample tubing to the manifold inlet.
5. Proceed with purging the lines according to your workplan.
6. Seal the shroud zipper. Charge the shroud with Helium.*
7. If necessary, Helium concentration can be confirmed by portable meter or additional summa canister sample collected inside the shroud.
8. Place your hands into the shroud gloves, and open the sample canister valve to begin sampling.
9. Close the canister valve when sampling is complete.
10. Open the shroud to remove the canister. Shroud can be moved to the next sampling point.
11. Return all shroud components to the laboratory with the samples. Full replacement cost will be charged for any unreturned items.

* The shroud can be charged using a 6L summa filled with helium to 15 psi (resulting shroud atmosphere is 10-15% Helium). Alternately the shroud can be charged with a pressurized cylinder fitted with a regulator set to 20 psi (resulting shroud atmosphere is 70-75% Helium).

Special features:

1. Inflatable shroud can be reused at multiple points, the sample does not come into contact with shroud components
2. Minimizes Helium use, easily remains inflated for 10 minutes without recharging.

3. Canister and flow controller are inside the shroud; consistent with the California DTSC Advisory.
4. "Glove Box" design allows for sampling to be carried out without disturbing the shroud environment, consistent with California DTSC Advisory.



Standard Operating Procedure (SOP) #4

SVE Treatment System Sampling

Since Site 12 is located in a public retail location, the likelihood of sampling site visitors is high. Visitors to the sampling site may include shareholders such as project personnel, clients, regulators, property owners or managers, property employees, property customers, other temporary site workers (e.g., construction workers, delivery drivers, painters, landscapers, etc.), or members of the public. If there are visitors to the site during sampling who ask questions, direct them to the BRAC Office Community Relations department or fortordcleanup.com. Unauthorized visitors may not enter a safety exclusion zone. Notify project manager of any visitor encounters.

I. Equipment List

- Personal protective equipment including safety vest, sun/wind protection (if necessary), steel-toe boots and nitrile gloves
- 1-Liter SUMMA® canisters, vacuum gauges, and fittings. Order extra equipment and materials in the event of failure during checkout and use.
- 1/4" Nylaflow® tubing and PTFE tape
- Clamps and tools
- Field logbook, indelible ink pen, field forms and camera.

II. Procedure

- 1) Wear new disposable gloves to prevent contamination during leak testing, and dispose of gloves after each test, in a trash bag for proper disposal. Wear new gloves for each VOC sampling event and location.
- 2) Confirm the valve on the SUMMA® canister is closed (knob should be tightened clockwise).
- 3) Remove the brass cap and attach the closed-loop vacuum gauge with the appropriate size wrenches.
- 4) Open the SUMMA valve to verify there is sufficient vacuum greater than -25 inches mercury (inHg) and that the vacuum does not decrease within 15 minutes, which would indicate a leak in the sampling train of the SUMMA® canister. If a leak is detected at any time, resample using all new sampling equipment; re-used canisters may have been contaminated (use professional judgement). Record the results of this shut-in test on the field logbook and sampling forms.
- 5) Record the SUMMA® canister vacuum after a passed shut-in test is completed as "Initial Vacuum" on the Chain of Custody form, field forms, and in the field logbook.
- 6) Close the SUMMA® valve and remove the brass cap on the manifold.
- 7) Attach an approximately 1-foot long piece of 1/4" Nylaflow® tubing with the compression fitting and Swagelok®. Use wrenches to assure a tight seal, but do not over-tighten.

- 8) Wrap the Swagelok®- Nylaflow® tubing union with PTFE tape.
- 9) Insert the tubing into the sample port at the SVE wellhead with the sample valve off.
- 10) Open the SUMMA® canister valve to make sure there are no leaks in the system. A leak will be identified by any vacuum drop on the canister gauge, whereas a leak free system will exhibit a stable vacuum reading for a period of 2 minutes.
- 11) If a leak is detected, close the SUMMA® canister valve. Re-tighten all fittings then repeat the above step with a new SUMMA® canister.
- 12) If no leak is noted, open the sample port valve and allow at least 3 minutes for the SUMMA® canister to fill.
- 13) Verify the final vacuum of the SUMMA® canister (repeat steps 2 through 4 above). For grab samples, the ending vacuum may be close to ambient pressure (0 inHg).
- 14) Record the SUMMA® canister final vacuum after sampling is completed as “Final Vacuum” on the Chain of Custody form, field forms, and in the field logbook.
- 15) Only perform VOC monitoring on the SVE wells and SVE system influent and effluent monitoring points while the SVE System is in operation to ensure proper purging.
- 16) Tubing associated with VOC sampling may be designated to one location or disposed of and new tubing used at the next VOC sampling event based upon event conditions.

Chapter 3. Common Sampling Procedures

3.1. Purpose

This chapter presents specific sampling procedures and items common to the sampling events covered in Chapters 4 – 12. Section 1.5 and Figure 1-1 of this handbook illustrate the relationships among these chapters. Additional references include facility standard operating procedures (SOPs) and special requirements contained in regulatory programs and site permits.

3.2. Preparations for Field Sampling

The success of a field sampling program depends on the level of preparation prior to entering the field. Implementation of the SAP begins with preparing for the field sampling operation. The following preliminary steps are vital to the success of the project:

- **Preliminary Off-Site Evaluation.** Prior to implementing the SAP, the Program Manager and Health and Safety Supervisor should review any Historical Overview and Site Description sections of the SAP. This review may result in the decision for an on-site evaluation to assess the sampling procedures, relevant safety equipment, and PPE.
- **Equipment Verification.** The SAP should specify an equipment list, including sampling equipment, sample containers, and PPE. This list should be reviewed in detail by the entire sampling team and the Health and Safety Supervisor to verify that necessary items are included and appropriate for the site being sampled.
- **Inventory.** The Equipment Technician (however named) shall gather all the specified equipment and containers into one place and verify that it is on hand. Reagents, supplies, and quality control materials shall be checked and verified as appropriate. The designated technician shall notify the Program Manager that equipment preparations are complete.
- **Sign-Over of Materials.** The designated individual shall check the equipment inventory, and sign for custody if required.
- **Staffing and Scheduling.** The Program Manager shall consider the impact of specified sampling requirements on staff and schedule.
- **Screening or Field Measurements.** Sample screening or field testing for pH, dissolved oxygen (DO), sulfite, conductivity, disinfection chemicals, and temperature require additional field time. The need for additional personnel is based on time demands, training requirements and degree of difficulty. Significant field testing requirements may justify the procurement of a field laboratory and a trained field chemist to relieve other team members of this responsibility.
- **Preservation.** Preservation, either chemical or thermal, is required for most water samples. Thermal preservation usually requires icing the samples after collection and storing samples at $\leq 6^{\circ}$ Celsius (C). For chemical preservation, two practices exist for adding preservative: 1) addition of the chemicals to the samples in the field, and 2) addition of the chemicals to the sampling containers prior to sending the containers to the field. Adding the reagents to the sample containers at the time the samples are collected requires the sampler to maintain records of addition and quality of the reagents and to follow proper chemical handling techniques. In some cases it may be advisable to have the laboratory add the reagents to specially labeled sample containers before they are sent to the field. This may reduce the fieldwork required and the possibility of field error resulting from contaminating the preservatives. Addition of the correct amount of preservative can be estimated for samples collected on a routine basis having little to no outside environmental or process effects.

WARNING: When using containers filled with preservative, use caution when filling the bottles to ensure the preservative is not released to the environment and the correct amount of preservative has been added to adequately fix the sample.

- **Time.** Many samples have short holding times prior to analysis. Review the holding time requirements and coordinate the schedule with the laboratory so the samples are analyzed within the required holding times. Holding times are dictated by the regulatory program and data may be invalidated if holding times are not met.

Note: Refer to Appendix B of this handbook for specific information on hold times, preservation, and containers.

3.2.1. Preparing for a Sampling Event

Preparing for a sampling event requires planning and a thorough knowledge of the regulatory program. The key elements for such preparation include:

- **Objectives.** The objectives should be thoroughly understood by all sampling personnel prior to sample collection. Knowledge of the compliance scope, boundaries, geography, and area roads and bridges will facilitate sampling.
- **Map of Study Area.** A map of the study area is essential for sampling. The map should be detailed enough so that sample locations and landmarks are clearly identifiable.
- **Permits and Regulations.** The person collecting samples should have a working knowledge of applicable permits, required monitoring, and other specified conditions. Regulations that potentially impact the sampling area, such as right of entry, should be reviewed by the sample collector.
- **Waste Sources.** When the objective of a project is to determine the nature, extent, or impact of a waste source upon an environmental medium, knowledge of waste source(s) within the area, as well as those sources upstream or upgradient that may impact the area, is essential. This knowledge entails knowing waste source discharge points or areas, type of waste, volume of discharge,

and constituent concentration. When this information is not readily available, it may be necessary to collect background information.

- **Environmental Medium Characteristics.** If the study is of a waterway, the physical characteristics of the waterway should be known prior to sample collection. These important physical characteristics include whether the receiving waterway resembles a lake, reservoir, pond, small stream, or a river. Average and maximum recorded flow, width and depth, type of benthic substrate, and type of predominant aquatic vegetation also should be noted.
- If the study area is limited to land, it is important to have knowledge of the terrain, soils classification, geology, terrestrial vegetation, industrial and residential development, predominant land use, and wildlife.
- **Sampling Information.** A sampler must know the types of samples to be collected, (e.g., water, wastewater, soil, or solid waste). The sampler also must know whether the samples are to be collected nocturnally or during the daytime, and where within the environmental medium the samples are to be collected (including both horizontal and vertical collections) as well as the preferred method of collection.
- **Laboratory Arrangements.** Arrangements must be made with the analytical laboratory to ensure that the laboratory is expecting the samples when they arrive and has a description of the types of samples (e.g., liquid, semi-solid, solid, or biological), an approximation of the number of samples for each sample type, and the analyses requested on each sample type. Arrangements must be made for the appropriate number of sample containers and preservatives where required. Regulations on transportation of samples from the point of collection to the laboratory must be considered, and the COC record must be traceable, as detailed in the SAP.

- **Equipment.** Prior to going to the sampling location, the sampling gear should be examined to ensure that it is appropriate for the task and in good working order. Verify that any preventative maintenance has been completed according to the SOPs. Label, mark, and otherwise identify all equipment, instruments, reference materials, and associated supplies for measurement processes to indicate calibration or standardization status. Expiration dates of reagents and solutions should be checked and verified as to usability. If a boat is required, an appropriate boat, motor, and life jackets must be available, and preliminary boat launch locations should be known before going to the sampling site. All equipment should be examined prior to starting the sampling event.

Note: When in use, sampling equipment should be anchored to prevent loss in the event the rope or equipment slips through the hands of the sample collector.

- **Safety.** The safety of sampling personnel is paramount. During wading operations, a rope should be attached to the sampler and extended to an anchored person on shore. In boating operations, at least two people should be present, one to collect the samples and another to operate the boat motor. Boat personnel are required to wear life preservers and take care to avoid overloading the craft. When collecting samples, beware of snakes, stinging insects, ticks, or other animals that may cause injury to the sample collectors.
- **Personnel Transportation and Lodging.** The Program Manager must consider arrangements for transporting sampling personnel and equipment to the sampling site, and for lodging when the sampling extends beyond a working day.

3.2.2. Preliminary On-Site Evaluation

When sampling for the first time at a new location, a preliminary on-site evaluation should be conducted prior to the sampling event to ensure that all aspects of the sampling process are addressed.

Upon arrival at the site, the Program Manager (or designee) and the Health and Safety Supervisor shall check with facility personnel to determine whether there have been any recent changes at the sampling locations that would influence the SAP or modify the expected hazards.

3.2.3. Preliminary Site Safety Evaluation

After a preliminary hazard analysis, sampling locations should be inspected to develop the Safety Plan or HASP as appropriate to the scope of the project. PPE information specified may not be completely reliable, and additional air monitoring may be required. When air monitoring activities are needed, focus first on identifying conditions that present an acute health hazard, and then evaluate exposure to chemicals such as carcinogens that could create long-term health problems.

If samples are to be collected in a confined spot, testing the air within the space for oxygen content should be a priority. Tests for explosive levels of flammable vapors should be conducted next, followed by testing for the presence of hazardous concentrations of specific toxic agents (depending upon the nature of the space and its contents or previous contents).

Note: Real-time instrumentation is available for making these measurements. Air samples should be collected to evaluate the levels of other chemicals in the air that may require respiratory protection. Some organic chemicals such as gasoline vapors can be monitored with standard field instruments. However, monitoring for carcinogens will normally require the use of a field gas chromatograph or the collection of test samples for laboratory analysis.

In general, the air monitoring program to evaluate worker exposures to toxic chemicals should be designed by an industrial hygienist familiar with the facility and potential hazards to which the field sampling team will be exposed.

Review physical hazards that may be present at the site, such as unstable footing near river embankments, water safety practices, first aid supplies, equipment safety practices, and other physical hazards.

3.2.4. Explosive Safety Evaluation

The possibility of encountering explosive hazards must be considered in all sampling plans. When the presence of energetic materials is suspected from the history of a site, appropriate precautions can be incorporated during the planning stages.

Consideration should also be given to situations that can lead to the formation of unstable materials from constituents that are not originally energetic compounds. Formation of peroxides in ethers and metal picrates are two examples that have been known to create safety hazards.

3.2.5. Preliminary Sampling Evaluation

Sampling locations should be inspected to ensure the information in the SAP is correct. All equipment should be checked prior to mobilization and the day before the sampling event to ensure proper equipment operation, parts, and records are available for the sampling operation. If needed, preventative maintenance should be performed.

Reagents, supplies, reference materials, and consumable materials should be verified as to expiration dates, quality, and applicability to the assigned equipment.

Locate all the sample locations during the on-site evaluation to determine site accessibility with the designated equipment, sample location, and possible background contamination for the contaminants of interest. Electromagnetic interferences, volatile air pollutants from locations off site, weather, and climate may affect the sampling event and should be planned for, as much as practical, to avoid delays in sampling.

3.3. The Sampling Event

A typical sampling event should include the following sequential activities:

- Complete all preparation and preliminary evaluation activities as needed
- Arrive at the sampling site with appropriate equipment, supplies, materials, and sample containers
- Set up equipment, work areas, and safety areas, as described in the SAP

- Collect samples at the locations specified in the SAP or reference procedure
- Immediately following sample collection, ensure that each sample container is labeled as described in the SAP. The sample label must be traceable to the sample number, date/time sampled, sampler's name, preservative, and site name, location or unique project identifier.
- Document the exact location of the collected sample(s) in the field logbook or field notebook (FLB/FN). Also, record in the FLB/FN other observations of environmental conditions that could affect or contribute to knowledge of the sampling area and the environment where the sample is collected. Prevailing weather conditions at the time of sampling should also be recorded.
- Preserve or ice samples as appropriate and record preservation method
- Perform field tests or field screening measurements and record all observations in the FLB/FN
- Complete the COC record and other field records
- Pack and seal the shipping container with collected samples, and transport the shipping container with the COC record and any laboratory required forms to the laboratory. Retain copies of all transmitted forms.
- Return all forms and copies of relevant FLB/FN pages to the Program Manager or designee
- Clean sampling equipment for the next sampling event or storage
- Breakdown all work area and safety areas as required and return the site to the condition found at the start of the sampling event
- Dispose of all waste materials using appropriate procedures.

3.4. Sampling Procedures

The SAP refers to detailed sampling procedures or includes the details of the sampling operation. A standard SOP format should be used to incor-

porate the following items for each type of sampling operation:

- Sampling locations, sample numbers or identifiers
- Type, volume, and number of sample containers to be filled at each sampling location and the records to be maintained
- Contaminants to be measured and special handling procedures to ensure proper collection
- Safety, health, and hazard cautions
- Sampling equipment (construction material, type, etc.) and records to be maintained for status, maintenance, and corrective action
- Step-by-step sample collection procedures (grab, composite, continuous for specified period, etc.)
- Sampling frequency for repeated sampling at the same sample location
- Special sampling requirements (e.g., the collection of initial runoff samples after a rain for contamination)
- Sample handling procedures for each sample container (e.g., screened, filtered, sequence for filling groundwater sample containers, etc.)
- Preservatives required for each sample container and contaminant
- Reagents, supplies and support services quality, verification and validation criteria to ensure materials are used properly
- Equipment decontamination procedures to be used between sample locations and between sampling events
- Recordkeeping requirements, documentation handling, and retention requirements
- Sample, equipment, and materials storage requirements
- Provisions for storage or disposal of wastes generated during field sampling.

3.4.1. Sampling Strategies

See Appendix A for sampler and sampling recommendations and strategies for waste materials. Sampling strategies for drinking water, wastewater,

groundwater and TSCA materials are permit or compliance dependent. The Scope or Purpose section of the sampling procedures should describe the rationale for the sampling strategy to ensure that all personnel involved with the project have an understanding of the sampling event.

3.4.2. Sampling Procedure Checklist

Following is a checklist of the minimum steps to address in SOP format.

- **Sampling Approach**
 - Objective
 - Design of sampling plan
 - Statistics
- **Material to be Sampled**
 - Physical state
 - Volume
 - Hazardous properties
 - Composition
- **Site**
 - Accessibility
 - Waste generation and handling
 - Transitory events, startup, shutdown
 - Maintenance
 - Climate
 - Hazards
- **Equipment**
 - Maintenance
 - Preparation and cleaning
 - Operation
 - Calibration and standardization
- **Sample Handling, Transportation, Storage and Preservation**
 - COC
 - Seals
 - Forms
 - Containers
 - Preservatives, reagents, and supplies
- **QA/QC**
 - Controls on process
 - Audits
 - Training
 - Samples, blanks, duplicates, and spikes
- **Health and Safety**
 - Personnel protection

- Safety procedures
- Emergency procedures
- **Laboratory**
 - Document transfer
 - Sample arrival schedule, transfer
 - Sample preservation, handling and storage
 - Analytical methods and QC
 - Reporting format and schedule.

3.5. Sample Documentation and COC Procedures

Thorough documentation of a sample's custody is required to support sample validity. The documentation must verify that the samples are representative, were collected in accordance with the requirements of the SAP, and are not vulnerable to tampering before being received by the laboratory. The COC begins when the sample is taken and ends when the sample is disposed of. Sample documentation and COC procedures include the following.

- A completed sample collection label attached to all sample containers
- Records of sampling operations written in FLB/FN or related forms as designated for the operation in the SAP. Records include sample type, sample matrix, sampling method, field test methods, and QC procedures. A table may be used to present this information.
- Identification of every sample container on a COC record and all custody transfers documented
- Custody of the samples with all discrepancies in the field operations resolved or duly recorded.

The following should be used to generate the required sample documentation.

Note: EPA's "Field Operations and Records Management System II Lite (FORMS II Lite™)" software is an electronic COC and may also be used to simplify and accelerate the sample documentation process.

3.5.1. Pre-Assigned Sample Numbers

Each sample consists of all of the material collected for analysis at one place, at one time, and of one

matrix, except for composite samples, which may contain components collected at different locations or time.

The Program Manager shall establish a system for assigning a unique sample number to each sample collected in the field. The numbering system will be defined in the SAP, in case additional samples are generated in the field. The number for each sample will be used to identify the sample in the FLB/FN, on the sample container, and on the COC record. The number may be used on other forms and reports presenting measurements, test data, or evaluations.

The sample numbers of field QC samples like a field duplicate should be transparent to the laboratory. The sample numbers should not reveal whether a sample is a blank sample or two field samples are duplicate/split pairs to avoid potential biasing of analytical results.

The sample number provides a common identifying code for all of the analytical results for a single sample. This is particularly useful when the results are entered into a computer database, which should include:

- Sample number
- Sample container number
- COC record number
- Matrix
- Location
- Sample type
- Sample date and time
- Sampler's name
- Parameter
- Analytical result
- QC data
- Compliance limit and
- Data qualifier code (optional).

Results from analysis of trip blanks, field blanks, equipment decontamination blanks, split samples and MS/matrix spike duplicate (MSD) samples may be entered into a computer database. In some testing programs, these results are used to

generate the data qualifier code for the analytical results from test and duplicate samples.

It is recommended that the information associated with each sample number consists of elements describing the sample type, matrix, location, and the time and date of sample collection as required.

Note: If the sampling and analytical data are to be added to an existing database, sample numbers should be consistent with database requirements.

3.5.2. Sample Container Labeling

Sample labels are an important part of proper documentation to reduce the possibility of confusing sample containers, and to provide the necessary handling information. Sample containers should be pre-labeled as much as practical before sample collection. The labels may be protected from the sample matrix with a clear tape covering. For volatile samples, check with the laboratory to ensure that any labels being used do not interfere with their auto-samplers. Sample labels should include sample number, date and time sampled, location, sample type, preservative and the sampler’s initials or signature.

Sample numbers may be unique to the sample location, to the sample type or to the container. In some labeling processes, a unique sample number is written on the container label, and all information recorded on the accompanying form(s) is traceable to the unique sample number.

Some number schemes uniquely number each sample container. All data reported for the sample includes the sample container number for traceability to the container measured. This is useful when sample containers are cleaned and lot controlled, and traceability from container preparation, preservation, sampling, and testing is required.

A designated Field Sample Custodian or sampler should label the sample containers when they are filled. Preprinted, adhesive, multiple part labels formatted as shown in Figure 3-1 may be used. Each part includes the unique sample container number that may be pre-numbered to avoid duplication.

Note: Because 40-mL volatile organic analysis (VOA) vials may be stuck in an autosampler, the field sampling team leader needs to contact the laboratory to make sure if applying a clear tape over a sample label of 40-mL VOA vials is acceptable. Use waterproof ink to make label entries. FLB/FN notations should provide an explanation if a pencil was used to fill out the sample container label due to field weather conditions. Because waterproof ink may contain target VOAs such as xylene, toluene, or alcohols as a solvent, great care is needed to prevent VOA samples from contamination by the solvent of waterproof ink or permanent marker.

Figure 3-1. Multiple Part Container Label

PROJECT NAME	
Sample #: XXXXX	
Container #: XXXXX	
Sample #: _____	
Date: _____	Time: _____
Location: _____	
Cont. Size: _____	
Cont. Type: _____	
Matrix: _____	
Type of Sample: _____	
Preservative: _____	
Signature: _____	

3.5.3. FLB/FN

The FLB/FN is the written record of all field data, observations, field equipment calibrations, and sample collection activities. Potential for future legal actions dictates that the FLB/FN be site-specific and that they be hardbound (e.g., ledger, composition book, diary, etc.). All pages (front and back) shall be serially numbered so removal will be apparent. Samplers shall adhere to the following guidelines when using FLB/FN.

- The FLB/FN shall be assigned to the QA/QC Coordinator or designee. Additional log books may be assigned by the Program Manager or designee to the Field Chemist and the Health and Safety Supervisor. The QA/QC Coordinator or designee shall note in each FLB the individual to whom it was assigned. The FLBs may be controlled by the QA/QC Coordinator or the Program Manager.

- Each FLB shall be annotated with the sampling program name or number.
- Key personnel and telephone numbers shall be listed on the first page.
- Entries shall be written in waterproof blue or black ballpoint pen. Avoid felt tip pens. *Do not use pencil.*
- Start a new page at the beginning of each day.
- Entries should be chronological – a time notation should introduce each entry.
- Sketch or obtain a map of the area or facility. Include sketches of layout, structural features, and points of interest or contamination. Include a north arrow and a rough scale. If possible, obtain a site map (reduced if necessary) and permanently place it in the FLB/FN.
- Language should be objective, factual, and free of personal feelings or other inappropriate terminology. Speculation or personal observations may be included if they are clearly identified as such.
- Do not erase or scratch out errors. Draw a single line through the error, then insert corrected material. The person who made the error shall initial and date the correction as well as clearly state the reason for the error.
- Entries or corrections made by individuals other than the person to whom the FLB/FN was assigned shall be signed and dated by the individual making the entry or correction. An explanation for the correction should be noted.
- The last entry for each day should include a short summary of the day's activities, weather conditions and the time the site was left. As appropriate, the last entry for each week should be a summary of the week's activities. Weekly summaries should be thorough and descriptive.
- The FLB/FN shall be signed at the end of each day. Signatures shall be written on a single diagonal line drawn across the blank portion of the page following the day's last entry.
- All FLBs/FNs shall be returned to the individual designated for review and final storage when sampling is completed as described in the SAP.
- FLB/FN entries will contain a variety of information. Information to be entered at the start of each day of sampling includes the following:
 - Date of the sampling event
 - Time sampling started and approximate time for set up of equipment
 - Weather conditions
 - Level of PPE being used
 - Names of field sampling team members and others present during the sampling.
- Fully document all deviations from the SAP or changes in sampling procedures. Problems, delays, or any unusual occurrences such as improper equipment or breakdowns should be included, along with resolutions and recommendations. Summarize the content and conclusions of all relevant meetings, discussions, and telephone conversations in which you are involved. Include the names and affiliations of all personnel involved. Thoroughly document all directives and/or guidance from EPA or other government personnel. Directives that give personnel specific authority to make critical decisions must be documented in the FLB/FN.
- Whenever a sample is collected or a measurement is made, a detailed description of the location must be recorded. The source from which the sample is collected should be clearly identified to maintain traceability and allow another person to locate the exact sampling location. The ability to relocate the sample site ensures duplication of future sampling events. Measurements from permanent features (e.g., center line of road, numbered utility pole, etc.) to the sample point must be made and entered into the FLB/FN. Coordinates on a map, or an accurate site sketch with distance measurements to known locations are other options to ensure the exact location of each sample is recorded.

- Describe the site thoroughly so another person will be able to locate the exact sample location. Note signs of contamination such as oily discharges, discolored surfaces, unusual odors, dead or distressed vegetation including types of plants, if possible. Photographs may be taken to provide evidence of visual observations, record site conditions, and assist with locating the sample site in the future. Photographs taken of sample locations should be noted along with the picture number. Log the record in the FLB/FN to identify which sampling site is depicted in the photograph.

Note: A series of photographs can be identified by taking the first photograph of an informational sign with the sampling program name, number, and the project number on it.

- Each time a sample container is filled and labeled, a copy from the multiple part form of the sample container label or reference number label with all information recorded shall be put into the FLB/FN.
- All equipment used to make measurements must be identified by type, manufacturer, and serial number, along with the date of calibration. Details of field calibration procedures and results shall also be included in the FLB/FN.
- Note decontamination or disposal procedures for all equipment, samples, protective clothing, and personnel decontamination procedures.
- For each delivery or shipment of samples to a laboratory, record the following information in the FLB/FN:
 - Custody procedures and serial numbers
 - Packing and shipping procedures (record air bill numbers)
 - Name, address, telephone number, and contact of the laboratory performing the analysis.

Note: The laboratory address should be the sample receipt address, which may not be the same as the mail receipt address.

3.5.4. Field Notes/Field Sampling Forms

Field notes or field sampling forms are used in addition to or in lieu of field log notes. When field notes are used in lieu of an FLB, the record keeping practices presented in Section 3.5.3 should be followed. The field form provides a place for the sampler to record the information required for the project. Field forms are specially designed for any given project and may be completed one per sample or one per sampling event. The forms include blank lines for recording the information necessary for the project to ensure the proper information is recorded. All blanks must be completed on a field form to ensure proper documentation. The sampler completes the field form for all samples collected including QC samples. An example of a field form for a well sampling activity is presented in Figure 3-2 below.

Note: A review of the regulatory program's specific requirements must be conducted to ensure that all documentation requirements are met. Some programs do not allow the use of loose field forms as the sole documentation vehicle and require hardbound logbooks.

The field form lists the sample number, location, matrix, the type and number of sample containers filled (including QC samples), any chemical preservatives added and checked for each sample container, sampling procedure reference, deviations to the procedures and all field measurements and observations.

The Field Sample Custodian indicates acceptance of the information on the field form by signing and dating the form. In cases where multiple part forms are used for the sample label, for each sample container filled, one part of the multiple part adhesive sample container label is placed on the field form at the appropriate location. The completed field forms are returned to the Program Manager as soon as possible and by the means indicated in the SAP. Deviations or problems encountered during the sampling event must be communicated promptly in writing to the Program Manager or designee. This may be completed by sending the field form by facsimile or other means to communicate the deviations, as

well as allow for continuation of the project and ensure sample holding times are not jeopardized.

Note: The field form becomes part of the permanent project records, but is not usually sent to the laboratory.

3.5.5. Chain of Custody (COC)

An overriding consideration for environmental measurement data is the ability to demonstrate that samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Documentation of security, field handling criteria, shipment, laboratory receipt, and laboratory custody until disposal, provides evidence of proper processing. The degree of custody documentation is dependent on the regulatory program, data use, and needs. Many state programs for sampling wastewater and drinking water do not require “legal custody,” but recommend legal custody whenever data is known to be used for evidence. A review of data use and risk of legal proceedings will dictate the type of custody procedure to be employed. Documentation consists of a COC record that is completed by the Sample Custodian.

3.5.5.1. Field Custody Procedures

The Field Sample Custodian or sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples. A sample is considered to be “in custody” for legal proceedings if it is:

- In a person’s actual possession
- In view after being in physical possession
- Locked up so that no one can tamper with it after having been in physical custody
- In a secured area, restricted to authorized personnel only.

If any one of these is not in place at all times, the COC is broken.

The Program Manager or designee shall review all field activities to determine whether proper custody procedures were followed during the fieldwork and whether additional samples are required. The sampler or Sample Custodian shall notify the Program Manager of any breach or irregularity of COC procedures described in the SAP.

Figure 3-2. Field Form

Sheet <u> 1 </u> of <u> 1 </u>	MICROPURGE/LOW-FLOW SAMPLING LOG									
PROJECT:					WELL ID:					
EPA ID NO:					Well Condition:					
Proj./Task No.					Well Riser Dia. (ID):					
Date:					Screened Interval:					
Weather:					S.W.L. Measuring Pnt:					
Samplers:					Well Bottom Depth:					
Purge Method: Micropurge										
Sample Collection Method:										
Sampling Device:					STATIC WATER LEVEL:					
Tubing:					Initial Purge Volume:					
Pump Intake Depth:					Approximate Pump Throttle Setting:					
Total Recovered Purge Water This Well:										
Groundwater Sample Data:										
Sample ID	Analysis	Primary	QC	MS/MSD	Blank	Sample ID	Analysis	Primary	QC	MS/MSD
Instrumentation/Equipment Data:						Calibration Date:				
Field Test Results:						Hach Kit Tests:				
						D.O.		mg/L		
						Total Iron:		mg/L		
						Ferr. Iron:		mg/L		
						CO2:		mg/L		
Comments:										
Observations: Clarity:			Odor:			Floating Product:			Sheen:	
<i>PURGE WATER DATA TABLES</i>										
Stabilization Parameters	Units	Minutes	Minutes	Minutes	Minutes	Minutes	Minutes	Minutes	Minutes	
Temperature	C									
Sp. Cond.	ms/cm									
D.O.	ppm									
pH										
ORP (Eh)	mV									
Turbidity	NTU									
Clock Time										
Static W.L.										
Flow Rate	ml/min.									
Stabilization Parameters	Units	Minutes	Minutes	Minutes	Minutes	Minutes	Minutes	Minutes		
Temperature	C									
Sp. Cond.	ms/cm									
D.O.	ppm									
pH										
ORP (Eh)	mV									
Turbidity	NTU									
Clock Time										
Static W.L.										
Flow Rate										

3.5.5.2. COC Records

From time of collection through final sample disposal, there are many transfers of custody during the course of a sampling program. All sample containers must be accompanied by a COC record to document these transfers. A separate COC record shall be prepared by the Field Sample Custodian or sampler for each sampling event. In some programs, a COC record accompanies each shipping container and includes a pre-numbered COC record. This record lists the sample containers that are in the shipping container, and serves as the packing list for the container. The serial number on the form becomes the identifying number for the shipping package.

Figures 4-3 provides an examples of a sample COC record. The example has been used for a wide variety of regulatory programs and meets legal COC requirements. It tracks the samples from sample collection to disposal. All sampling, preservative, and test information is included. The SAP will indicate the individual responsible for completing each section. The following information relates to the numbered blocks:

COC Record—Figure 3-3.

- (1) The **company/command** name and **code** for the source of the funding.
- (2) The **contact name** for the Program Manager or designee indicated in the SAP.
- (3) The **job order number (J.O. #)** is entered to trace the information to the specific job.
- (4) The **signature** of the Program Manager or designee authorizing the funds.
- (5) The **permit number (No.)**, if applicable, for the samples collected. The number is issued by the regulatory agency for specific compliance reporting.
- (6) The **sample ID/location** based on permit designations or actual site location name.
- (7) The **sample taken date and time** are recorded for grabs on the start line only and for


composites on the start date and time and stop date and time.

- (8) The code for **sample type** such as grab, composite flowing and composite time (see Section 18).
- (9) The initials for the **person sampled by**.
- (10) The code for **sample matrix** such as liquid, solid, and gas (see Section 18).
- (11) The code for **preservative** (see Section 18).
- (12) The **# of samples** and **container** type are entered as “4-P” for four plastic containers (See Section 18).
- (13) The **analysis** to be performed - may reference descriptions in the SAP.
- (14) The field reading for **pH** for the sample containers indicated.
- (15) The field reading for **temperature** with the unit of measure for the sample containers indicated. The SAP may indicate the temperature to be recorded in the outfall temperature and not the sample temperature.
- (16) The field reading for **other** required measurements may be entered with the unit of measure. The SOP and name of the test must be indicated on the custody form.
- (17) After the samples are preserved, the **preservation is verified**. The verification is noted per the SAP. This verification may be temperature, pH, or if all is correct an indication is made as “OK.”
- (18) This section of the custody form contains **common codes** to be used by the sampler when completing the custody record. When situations arise that do not match the code designations, alternates may be added for the one time use on the custody form.
- (19) The expected **turnaround** for sample request is placed in this area. The reason is presented to determine if the turnaround time is regulatory, project specific, or based on holding time requirements.

- (20) **Special instructions** or comments may be entered in this space.
- (21) The **regulation applied** to the project is checked.
- (22) The **sample collection/charge, possible sample hazard** and other **comments** relate to the command in charge of sampling, special sample hazards, or to other sample comments. Reference may be made to code or specific sections of the SAP.
- (23) The **delivery order number** is entered.
- (24) The **contract lab** and **contract number (No.)** are entered for testing work performed by a designated contract laboratory.
- (25) The **sample disposal** method and the date completed.
- (26) The signature and **company/command** of the person relinquishing custody (**relinquished by**).
- (27) The signature of the person custody is **received (rec'd) by**.
- (28) The **date/time** custody is transferred.

Figure 3-3. COC Record

CHAIN-OF-CUSTODY RECORD & ANALYSIS REQUEST FORM



PWC ENVIRONMENTAL LABORATORY
 CODE 990 BLDG 2:140
 9142 MARYLAND A VENUE
 NORFOLK, VA 23511-3095
 PH: (0044450951) FAX: (0044450952)

ENVIRONMENTAL

CLIENT INFORMATION

COMPANY/COMMAND: 1 CODE: _____

CONTACT: 2 EXT: _____ FAX: _____

PHONE: _____

J.O. #: 3

SIGNATURE: 4

PERMIT NO.: 5

LAB USE ONLY LINE ITEM #	SAMPLE NO	SAMPLE ID/LOCATION	SAMPLE TAKEN	ON DATE	AT TIME	TYPE	SAMPLED BY	MATRIX	PRESERVATIVE	# OF SAMPLES / CONTAINERS	ANALYSIS	FIELD READINGS		PRESERVATION	
												pH	TEMPERATURE	OTHER	VERIFIED BY
	<u>6</u>		START	<u>7</u>		<u>8</u>	<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>	<u>13</u>	<u>14</u>	<u>15</u>	<u>16</u>	<u>17</u>
			STOP												
			START												
			STOP												
			START												
			STOP												
			START												
			STOP												
			START												
			STOP												
			START												
			STOP												

TURNAROUND: 19 (FOR RUSH TURNAROUND STATE REASON BELOW)

SPECIAL INSTRUCTIONS: 20

SAMPLE DISPOSAL: 25 () RETURN TO CLIENT () DISPOSAL BY LAB

REGULATION APPLIED:

RCRA () HSD ()

SDWA () TSCA ()

CWA () PHOTO ()

CAA () OTHER ()

CONTAINER: A. AMBER (glass) B. BAG C. CARTRIDGE D. CAPTIONED LID E. HEAVY THINSED

P. PLASTIC S. GLASS T. BOTTLE V. VIAL

B. BAG C. CARTRIDGE D. CAPTIONED LID E. HEAVY THINSED

SAMPLING/COLLECTION CHANGE: 22

POSSIBLE SAMPLE HAZARDS: _____

COMMENTS: _____

D.O. NUMBER: 23

CONTRACT LAB: 24

CONTRACT NO.(S): _____

RELINQUISHED BY: 26 COMPANY/COMMAND: _____

REC'D BY: _____ DATE/TIME: 28

RELINQUISHED BY: _____ COMPANY/COMMAND: _____

REC'D BY: _____ DATE/TIME: _____

RELINQUISHED BY: _____ COMPANY/COMMAND: _____

REC'D BY: _____ DATE/TIME: _____

1. CUSTOMER IS RESPONSIBLE FOR ALL CHARGES NECESSARY FOR THE PROCESSING AND ANALYSIS OF SAMPLE(S).
 ALL RUSH SAMPLES ARE SUBJECT TO SURCHARGE

2. SAMPLES RECEIVED AFTER 3:00PM MON. - THURS. AND 2:00PM ON FRIDAY WILL BE REPROCESSED THE NEXT BUSINESS DAY (7:30 AM - 4:00 PM).

The COC record identifies which pairs of sample containers were collected for the same analysis, and identifies the sample containers that were filled with sample for use as the MS/MSD QC samples. Based on the needs and data use, the COC record may not list any information as to the exact sample location or whether a sample is a field duplicate, field blank, trip blank or an equipment decontamination blank. This information is kept as blind information from the laboratory to ensure objective reporting. When this process is used, records must be maintained that trace the sample collected in the field with the sample as identified to the laboratory. Compliance data for drinking water or wastewater testing do not require blind submissions. The QC sample information is provided to the laboratory to ensure prompt notification when the QC data does not meet the SAP specifications.

Whenever samples are split with a second laboratory or government agency, a separate COC record may be prepared for the second set of samples. The additional set of COC records must be noted. Copies of the original may be sent with the split samples noted, or a separate form may be prepared by copying the appropriate information for the samples onto the additional form. In all cases, the use and need of the additional form should be noted.

Upon completion of the packing of each shipping container, the Field Sample Custodian shall confirm the completeness of the COC record by signing the COC record. If a multiple-part form is used:

- The original copy is put into the shipping container
- The first copy is sent immediately (preferably by fax) to the Program Manager or designee
- The second copy is kept with the FLB/FN or copy of the field form.

If a single part form is used, photo copies should be made for the Program Manager and the FLB.

After the COC record is completed and all samples are packaged and shipped to the appropriate locations, the person relinquishing the samples to

the laboratory or agency shall request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses to sign, this is noted in the "received by" space.

Field COC terminates upon laboratory receipt of the samples. The laboratory should complete the "received by" sections and if appropriate, the "preservative checks" sections on the COC record and return the original signed record to the Program Manager. If there are any discrepancies between the COC record, the contents of the shipping container, and the SAP or contract requirements provided to the laboratory, the samples in question shall be segregated from normal sample storage, and the laboratory shall immediately notify the Program Manager. In some cases, the laboratory checks the sample submittal and recordkeeping to ensure adherence to the SAP. This added check is often used in drinking water and wastewater testing programs for compliance monitoring. Recordkeeping and information checks should be performed by the laboratory to ensure the samples received meet the requirements of the SAP.

3.5.5.3. Custody Seals (Optional)

Custody seals are narrow strips of adhesive paper used to indicate whether a shipping container has been opened during shipment. The seals are placed along the edges of the most exterior container in which samples are enclosed. It is not always necessary to place seals on individual sample containers in the shipping container.

Paper custody seals should be signed and applied before the shipping container is shipped to the laboratory. The preferred procedure includes use of a custody seal attached to the front-right and back-left of the container. Custody seals are covered with clear plastic tape. Another way to use custody seals, is to put all sample containers with packing and ice in a large garbage bag and seal the garbage bag with a signed custody seal.

3.5.5.4. Custody Transfer

Transfer of custody and shipment procedures are as follows.

- Each sample shipping container shall be accompanied by a properly completed COC record. The original of the record shall be included in the container. The Field Sample Custodian shall keep a copy of the completed form as part of permanent documentation and will send a copy of the COC record to the Program Manager.
- When transferring possession of samples, individuals relinquishing and receiving shall sign, date, and note the time of the transfer. This record documents custody transfer from the Field Sample Custodian to another person, to a mobile laboratory, to the permanent laboratory, or to a secure storage area.
- If the sample container is sent by common carrier, a bill of lading shall be used. Bill of lading receipts shall be sent to the Program Manager for permanent retention. If sent by mail, the package shall be registered with return receipt requested. Commercial carriers and the U.S Postal Service are not required to sign off on the COC record as long as it is sealed inside the package with the sample container and the custody seals remain intact (if used).

3.5.6. Request for Analysis

The Request for Analysis form is often incorporated into the COC record since the chain must accompany the samples. In more complex sampling programs, an additional form may be used to request testing.

When contracting for laboratory services and prior to submitting the samples, the laboratory should be contacted and the following information presented. The Request for Analysis form can be used as a preliminary contact mechanism to ensure that the scope of work is understood. This form:

- Specifies the analyses, procedures, and QC data to be performed on each sample container and the compliance protocols to be followed
- Specifies the laboratory accreditation/certification required to be maintained during the period of the contract

- Authorizes the payment for the analyses
- Alerts the laboratory to any anticipated hazards associated with the samples and custody procedures to be followed while the samples are in the possession of the laboratory
- Specifies the reporting requirements and content for the final report from the laboratory
- Instructs the laboratory as to the disposition of the samples after the completion of the analyses.

3.6. Sample Packaging, Handling, and Transportation

The Field Sample Custodian is responsible for the proper field storage, security, packing, and shipping of the samples from the field to the laboratory or designated holding location. The packaging, labeling, and shipment of samples by common carrier are regulated by the DOT and the International Civil Aviation Organization (ICAO)/International Air Transport Association (IATA), when appropriate. Instructions for classification, labeling, and packaging of hazardous materials are contained in DOT regulations (49 CFR 172 and 173, and subsequent Parts). Overnight couriers generally accept materials shipped under these regulations. However, some couriers have additional restrictions for hazardous shipments. EPA also regulates the shipment of hazardous waste and hazardous material by requiring labeling on certain packages.

The procedure for determining whether a sample is hazardous under DOT regulations is complex, as is the determination of the proper shipping name, packaging requirements, and labeling requirements for DOT hazardous materials. A summary of specific requirements are addressed below. Should questions arise, assistance is available from the DOT (1-202-366-4000) and Federal Aviation Administration (FAA) (1-866-835-5322) hotlines.

Samples obtained at sites are classified for shipping purposes as either environmental (non-hazardous) samples or hazardous samples. If a material is being shipped for testing to determine its hazards, a tentative hazard class assignment

should be made based on knowledge of the material. Samples requiring special packaging or labeling are those containing chemicals that are listed as hazardous materials in:

- 49 CFR 172.101
- CERCLA RQ Hazardous Substances
- DOT CLASS 9 listed in 49 CFR 172.101 Appendix A, Poison DOT Class 6.1 and Flammable Liquids.

Environmental (non-hazardous) samples are those that are not classified as Hazardous Materials under DOT regulations, are packaged in quantities less than the CERCLA RQ, *and* for which a Hazardous Waste Manifest is not required by EPA. These samples require careful packing, but no special shipping procedures. In general, samples of groundwater, surface water (other than leachate or lagoons), and soil may be shipped as environmental samples (non-hazardous) to an analytical laboratory for testing if each of the sample containers contains less than 1 pound of soil or 1 gallon of water, and the entire shipping package weighs less than 66 pounds. Eventual analysis for a hazardous constituent does not necessarily classify a sample as a DOT hazardous material, nor does the classification of a material as a hazardous waste under EPA regulations. DOT regulations forbid the shipping of non-hazardous materials as hazardous. However, if any doubt exists as to whether the sample might be classified as a hazardous material, the sample should be treated as hazardous.

Note: For details on the shipping of non-hazardous waste, refer to *ASTM D6911-03: Standard Guide for Packaging and Shipping Environmental Samples for Laboratory Analysis*. This standard provides guidance in determining the most appropriate procedures for packaging and shipping environmental samples.

The storage and disposal of hazardous waste is regulated by the EPA. Hazardous waste, as specified in 40 CFR 262, is not exempted from EPA manifesting requirements. However, EPA RCRA regulations exempt samples collected for analysis or treatability testing from the RCRA requirements that otherwise apply to hazardous waste (including the requirement for a Hazardous

Waste Manifest). The definitions for these exemptions are:

- **Samples for Analysis.** 40 CFR 261.4(d): Samples of solid waste, water, soil, or air, which are collected for the sole purpose of testing to determine their characteristics or composition, when samples are being sent to the laboratory for testing or are being returned to the collector after testing.
- **Samples for Treatability Testing.** 40 CFR 261.4(e): Samples collected for the purpose of conducting treatability studies when they are being transported to the testing facility provided they meet criteria for the quantity of material, packaging, and permit status of the receiving facility.

3.6.1. Sample Packaging Requirements

The Field Sample Custodian is responsible for the packing and shipping of the samples from the field to the laboratory. Samples shall be properly packaged for shipment and dispatched to the laboratory for analysis with a signed custody record enclosed in the shipping container box or cooler. Shipping containers shall be locked or secured with strapping tape in at least two locations. Shipments that are sent to an on-site laboratory or one in close proximity that does not require the use of a common carrier shall be transferred in accordance with local regulations. Table 3-1 below lists sample packaging procedures that will ensure samples arrive at the laboratory with the COC record intact.

The following major issues must be addressed in preparing environmental samples for shipment to the laboratory by common carrier:

- Compliance with EPA regulations, so the samples are not classified as hazardous waste
- Compliance with transportation regulations, including use of the proper shipping containers, use of warning labels, and completion of the required paper work
- Packing, to assure that the samples do not break or leak during shipping. This includes:

- Using approved containers meeting DOT drop test specification
- Lining coolers or containers with plastic bags
- For glass containers, wrapping each in bubble wrap and placing in a clear plastic resealable food bag
- For plastic containers, placing each in a clear plastic resealable food bag
- Never stacking glass containers or laying glass on its side.

3.6.1.1. Samples Classified as Flammable Liquid

Table 3-2 Column 1 lists packaging procedures that apply to those flammable and combustible liquids that do not meet the definitions of another hazard class except DOT Class 9, and for which exceptions under 49 CFR 173.150 are allowed. This includes Flammable Liquids Not Otherwise Specified (NOS), toluene, gasoline, and many of the other flammable liquids that are commonly encountered on hazardous waste sites.

Note: The DOT definition of “liquid” is different from that used by EPA. For purposes of transportation, liquid means a material that has a vertical flow of over 2 inches (50 mm) within a 3-minute period, or a material having 1 gram or more liquid separation, when determined in accordance with the procedures specified in *ASTM D4359-90, Standard Test Procedure for Determining whether a Material is a Liquid or Solid*, (49 CFR 171.8).

3.6.1.2. Samples Classified as Poison — DOT Class 6

Table 3-2 Column 2 lists packaging procedures that apply to those poisonous liquids and solids for which exceptions under 49 CFR 173.153 are allowed. This includes 1,1,1-trichloroethane,

trichloroethylene, trichlorobenzene, PCB transformer oil, and many of the other poisonous materials commonly encountered.

3.6.1.3. CERCLA Reportable Quantities — DOT Class 9

Table 3-2 Column 3 lists packaging procedures for substances (liquids and solids) where the waste material is not otherwise classified as a DOT Hazardous Material because of hazardous properties *and* for which the entry in Column 8a of 49 CFR 172.101 Table is 155. For the shipment of larger quantities of EPA hazardous waste and DOT Class 9 hazardous substances where the quantity of material in each container *exceeds* the CERCLA RQ and no other DOT Hazardous Material classification applies, the following packaging requirements apply:

- Label each container with a separate container number
- Seal each drum or pail with a Security Seal
- Prepare one COC record for each group of containers that is being shipped at the same time to the same destination. List the container numbers on the COC record.

These shipments may include EPA Hazardous Waste in 5-gallon cans and 55-gallon drums. Most DOT containers are approved. The list of approved containers for packing Groups II and III Class 9 Hazardous Substances are listed in §173.203 for liquids and §173.213 for solids. These lists include steel, aluminum, plastic and fiber drums (solids only). Quantity limitations are shown in 49 CFR 172.101, Column 9.

Table 3-1. Packaging by Common Carrier

Instructions	By Common Carrier	
	Non-hazardous Samples	Hazardous Samples
Secure sample container lids with strapping tape.	1*	1*
Mark the level of material in each sample container with a grease pencil.	2	2
Place each container in a clear plastic resealable food bag so that the sample container label can be read.		3
Place about ½ inch of inorganic cushioning material such as vermiculite in the bottom of a metal can.		4
Place each container in a separate can and fill the remaining volume of the can with an inorganic cushioning material such as vermiculite (do not use plastic foam cushioning material as it could dissolve if the sample container were to leak).		5
Close the can using three clips to secure the lid.		6
Write the sample number on the can lid. Indicate “This Side Up” by drawing an arrow on the can.		7
Put about 1 inch of cushioning material (e.g., vermiculite or plastic foam) in the bottom of a watertight metal or equivalent strength plastic shipping container. If the container is a cooler, seal the drain plug on the inside of the cooler with tape. Also line the inside of the container with a plastic bag.	3	8
Wrap glass bottles and jars in plastic bubble wrap.	4	
Place cans in the container and fill the remaining volume of the shipping container with packing material. Add ice bags if required.		9
Place the sample containers top-up in the shipping container. Arrange the sample containers so that glass containers are surrounded by plastic containers.	5	
Fill the void space around and on top of the sample containers with plastic bags filled with ice cubes or ice chips.	6	
Seal the COC record in a clear plastic resealable food bag and tape it securely to the inside of the shipping container lid.	7	10
Close and lock or latch the shipping container.	8	11

Instructions	By Common Carrier	
	Non-hazardous Samples	Hazardous Samples
If the shipping container used is a picnic cooler, use tape to seal the drain plug.	9	12
After acceptance by the shipper, tape the shipping container completely around with strapping tape at two locations. Secure the lid with tape. Do not cover any labels.		13
Place the laboratory address on the top of the shipping container.		14
For all hazardous shipments, complete shipper's hazardous material certification form.		15
Place a "This End Up" label on the lid and on all four sides of the shipping container.	10	16
Affix the signed and dated custody seals on the front right and back left of the shipping container. Cover the seals with wide, clear tape.	11	17

*Numbers indicate the instructions that must be followed.

Table 3-2. Packaging Not by Common Carrier

Instructions	Flammable Liquid	Poison DOT Class 6.1	DOT Class 9
<p><i>Quantity limitations shipped by cargo aircraft</i> Gross weight of package:</p> <p>Total quantity of flammable liquid:</p> <p>Maximum sample container size:</p>	<p>66 pounds</p> <p>49 CFR 172.101 Table, Column 6b</p> <p>49 CFR 172.101 Table, Column 5 <i>or</i> The flash point of the liquid</p>	<p>66 pounds</p> <p><i>Liquids – 4 liters (1 gallon)</i> <i>Solids – 5 kilograms (11 pounds)</i></p>	<p>66 pounds</p> <p><i>Liquids – 4 liters (1 gallon)</i> <i>Solids – 5 kilograms (11 pounds)</i></p>
Check the caps of all sample containers to assure that they are secure. Tape caps.	1*	1*	1*
Place each sample container in an individual 6-mL plastic bag and secure with a twist tie. The sample identification tag should be positioned to enable it to be read through the bag.	2	2	2
Place sample containers in paint cans in a manner that will prevent bottle breakage.	3	<i>Liquids: 3</i>	
Place vermiculite in the paint can around the samples. The amount of vermiculite used should be sufficient to absorb the sample if a sample container should break.	4		
Secure the lid to the paint can with can clips and label the outside of the can with the sample ID numbers and quantity.	5		
Wrap bubble wrap around each glass sample container and fix with tape.		<i>Solids: 4</i>	3
Package the paint cans in DOT boxes or cooler. Use additional packaging to secure cans.	6		
Seal the drain plug with tape on the inside and outside of the cooler and line		5	4

Instructions	Flammable Liquid	Poison DOT Class 6.1	DOT Class 9
the cooler with a plastic bag. Place the canned or bagged sample containers in the cooler. If plastic bottles are being used, alternate them with any glass container.			
Fill any voids in the cooler with additional packing material.	7	6	5
Place ice contained in bags on top of all sample containers within the cooler. Use as much ice as space will allow.	8	7	6
Place the COC record in a clear plastic resealable food bag and tape to the inside of the cooler lid. Label the outside of the cooler as containing the COC record.	9	8	7
Seal the cooler lid with clear tape or strapping tape. Affix security seals.	10	9	8

*Numbers indicate the instructions that must be followed.

3.6.2. Marking and Labeling

All samples *must be labeled* to prevent misidentification and should include the following information:

- Sample # or ID
- Date of collection
- Collector
- Analysis requested
- Preservative
- Sample location.

Sample labels must clearly link the sample to the field sheet or the COC record and must be written legibly and in permanent ink. In addition, all containers must be labeled and listed on the COC record.

Note: If a three-bottle set is used for VOAs, all three bottles must be labeled and listed on the COC record.

EPA TSCA regulations [40 CFR 761.40(e)] require that a PCB label be put on all containers whose surfaces are in direct contact with material that is over 50 parts per million (ppm) PCBs.

This requirement applies to sample containers as well as pails, drums, and other containers that are in direct contact with the PCB material. The labeling requirement does not apply to containers in which PCB sample containers are shipped.

Although the sample containers must be individually labeled, this requirement is not affected by the quantity of sample or whether the sample is classified as hazardous under RCRA or DOT regulations. For DOT Class 9 and EPA Hazardous Waste the following labeling requirements apply:

- If EPA Hazardous Waste Manifest is required:
 - Hazardous waste
 - liquid, NOS, NA3082
 - solid, NOS, NA3077
- If EPA Hazardous Waste Manifest *is not* required:
 - Environmentally hazardous substances
 - liquid, NOS, UN3082
 - solid, NOS, UN3077

OSHA's Hazard Communication Standard requires all containers of hazardous materials coming in or out of a workplace to be labeled with the contents, appropriate hazard warnings, and the name and address of the manufacturer. OSHA does not specify a standard labeling method, but some commonly used ones are provided by National Fire Protection Association (NFPA), Hazardous Materials Identification System (HMIS), ANSI, and DOT.

3.6.3. Shipping Papers

Ship high hazard samples via overnight courier following the courier's documentation requirements. A special airbill must be completed for each shipment. An EPA manifest must be prepared if the shipping container contains hazardous waste *unless* the samples are exempt. The Hazardous Waste Manifest must bear the handwritten signatures of the generator, transporter, and designated facility. A copy of the manifest must be kept for 3 years by the shipper. The shipping papers must contain the name, address, and handwritten signature of the shipper.

The shipping papers (and Hazardous Waste Manifest if used) must contain a 24-hour emergency response telephone number. This phone number must be monitored at all times while the hazardous material is in transportation, including storage incidental to transportation. The phone must be monitored by a person who is either knowledgeable of the hazards and characteristics of the hazardous material being shipped and has comprehensive emergency response and incident mitigation information for that material, or who has immediate access to a person who possesses such knowledge and information. The emergency response phone number must be entered on the shipping paper immediately following the description of the hazardous material or entered once on a shipping paper if the number applies to all of the hazardous materials and is indicated for emergency response information.

3.7. QA/QC Protocol

QC is a normal part of good field and laboratory practice. QC includes all of the procedures ap-

plied to data collection and generation activities to achieve and maintain the level of pre-established data quality. The desired level of data quality should be based on the intended use of the data. Therefore, the QC protocol should include all technical controls (e.g., sampling and analytical methods, use of field blanks, field duplicate samples, inclusion of performance testing or reference samples, statistical analysis, etc.). The controls start with the regulatory requirements of the data acquisition project and carry through to the ultimate data reporting and completion of all of the documentation of the use of these controls.

QA refers to the procedures used by management to assure that the QC is what is required and that it is being adhered to at any point in the project. QA constitutes the overview and monitoring processes designed to ensure that the quality of the data generated meets the desired levels as established by management. These controls include establishing DQOs based on the intended use of the data, the institution of procedures for formalizing planning documents prior to the initiation of data collection activities, and the use of audits to identify problems in both QC and QA.

The QA/QC protocol is specified in the SAP for each job that involves field sampling. QA/QC requirements are based on the level of data quality required for the project, and may address specific regulatory requirements. The purpose of a QA/QC protocol is to ensure the following:

- The laboratory receives samples that accurately represent the conditions existing at the sample site
- The results of the analysis are traceable to the specific sample location
- Compliance requirements are met.

The methods used to attain this protocol include training of personnel, providing detailed procedures for preparation, collection, marking and handling, packaging, packing, transfer of samples, and validation and verification of the administrative process and sampling techniques.

3.7.1. Decontamination of Sampling Equipment

The SAP should address the extent of decontamination and specify the procedures to prevent sample contamination. Sampling may be performed using separate laboratory cleaned equipment for each sample location. Procedure effectiveness should be checked for each matrix by submitting equipment decontamination blank samples to the laboratory for analysis.

Note: For specific information regarding the decontamination of field equipment, refer to *ASTM D5088-02, Standard Practices for Decontamination of Field Equipment Used at Waste Sites*. This standard describes the decontamination process for field equipment used in the sampling of soils, soil gas, sludges, surface water, and groundwater at waste sites. According to this standard, these practices are applicable only at sites where chemical (organic and inorganic) wastes are a concern, *not* for radiological, mixed (chemical and radiological), or biohazard sites.

3.7.2. Sample Container Cleanliness Requirements

Sample containers are a possible source of sample contamination. The SAP should specify the level of QC for sample containers. Pre-cleaned containers meeting EPA CERCLA cleanliness endurance criteria are available from several suppliers. If these containers are used, the serial number and QA batch number of each one should be recorded in the FLB/FN or on the field form. A review of the cleanliness should be made to ensure all parameters are checked to be below the detection limit of the contaminants to be tested for compliance. Some SDWA and CWA parameters may require laboratory cleaned containers proven to be below the limit of detection for the method.

Note: In no case should an effort be made in the field to decontaminate a sample container. If a container becomes contaminated, it should be replaced, with a note to that effect recorded in the FLB/FN.

3.7.3. Sample Container Type and Size Requirements

The types and sizes of sample containers to be filled for each sample will depend on method requirements and on QC requirements of the SAP. General sample container requirements are shown in Appendix B for different matrices and

analytical parameters. Compliance with specific instructions in the SAP is mandatory. If specified sample containers are not available, permission must be obtained from the Program Manager in writing for the use of other sizes and types of sample containers.

3.7.4. Sample Preservation and Storage Requirements

Special preservation and storage requirements should be specified in the SAP to ensure that samples do not undergo chemical changes from the time they were collected until their analysis by the laboratory. General requirements are specified in Appendix B. The specific requirements of the SAP will govern.

The quality of the reagents, water and materials used for preservation should be verified to ensure these items do not invalidate the reported results. Chemicals used as preservatives may be traced by lot number and quality by maintaining a reagent record keeping system. The water and acid preservatives used for trip and field blanks may be checked prior to use in the field and lot controlled to ensure no contamination is present prior to the material leaving the laboratory.

3.7.5. Sample Holding Time Limits

Even with preservation and special storage procedures, the composition of samples can change over time. The holding time for samples is the time from collection to laboratory preparation or analysis. Holding time limits summarized in Appendix B are method and program requirements. Site-specific holding times specified in the SAP take precedence.

3.7.6. Laboratory and Field Analytical Procedures

Laboratory analytical procedures for each parameter are specified based on the compliance limits, permit limits and data needs stated in the SAP. The SAP or COC record indicates to the laboratory which sample containers are to be analyzed for what parameters and specifies the analytical methods. Based on the DQOs, field testing may require the same level of QC as laboratory testing, and the procedures specified in

the field sampling or test plan must be followed exactly. Any deviations from established test procedures must be entered in the FLB/FN or on the field form and the Program Manager must be informed immediately of sample numbers affected.

3.7.7. QC Samples

Field QC samples are prepared and analyzed to determine whether test samples have become accidentally contaminated, check on the repeatability of the method, and ensure the samples are representative of the site or matrix sampled. A number of different QC samples may be specified. Each of the QC samples checks for a potential problem that can affect data reliability. The recommended frequency for each type of QC sample is summarized in Appendix C.

3.7.7.1. Test Sample

The test sample consists of one or more sample containers filled with material collected at one sampling point within a stated time. Several sampling containers may be required if material collected for analysis for different parameters must be preserved differently or sent to different laboratories. For a specific test sample, all containers are designated by the same sample location number, but may have different sample container numbers or designations to indicate variations made to the samples.

3.7.7.2. Field Duplicates and Split Samples

Field duplicate samples are two separate samples taken from the same source and are used to determine data repeatability based on field sampling and laboratory analysis procedures. Field duplicate samples are as follows:

- Assigned different container numbers
- Specified in the FLB/FN or on the field form
- Distinguished from the test samples on the COC record or field records
- Often submitted blind as to designation so the laboratory data assures objectivity.

Exception: Each test sample collected for a specific organic analysis may consist of two or more containers filled with the same material; these may be given different

container numbers but are designated as the same sample on the COC record. Only one sample container will be analyzed; the other being saved as a backup in case the laboratory must repeat the extraction and/or analysis. Duplicate samples for analysis consist of sets of two containers, with each pair of containers being designated on the COC record.

Field duplicate samples may be submitted to one laboratory for analysis for the same parameters. The comparability of the results provides information on the repeatability of the field sampling and laboratory analysis procedures.

The containers may be submitted to different laboratories for identical analyses to obtain information on inter-laboratory repeatability of field sampling and laboratory analysis procedures. This is a split sample.

Sample heterogeneity may cause major problems with the representativeness of field duplicate or split samples of soil/sediment matrices. Proper sample homogenization in the field will significantly improve the repeatability of the field sampling procedure. (Gy P. 1993, *Sampling for Analytical Purposes*, Wiley, West Sussex. Pitard F. F. 1993, *Pierre Gy's Sampling Theory and Sampling Practice: Heterogeneity, Sample Correctness and Statistical Process Control*, Books Britain, London.)

Typically, both field duplicates and split samples will be collected at a rate of 10% of field samples or at a minimum of one, per analyte, matrix, and sampling technique. More duplicates and split samples may be collected depending on the data quality needs.

3.7.7.3. Equipment Decontamination Blanks

Equipment decontamination blanks, or rinsate blanks, provide information on the levels of cross-contamination resulting from field or laboratory sample preparation actions. These blanks are specified in the SAP and on field sampling forms, and are prepared in the field. An equipment decontamination blank is usually reagent or deionized water that is free of the analyte of interest and is transported to the site, opened in the field, and poured over or through the sample collection device, collected in a sample container,

and returned to the laboratory and analyzed. This serves as a check on sampling device cleanliness. For example:

- **Field Groundwater Equipment Decontamination Blank for Metals Analysis.** Handled by the bailer, use ASTM Type II water, or better. Filter, place in a sample container, and preserve using the same procedures as for the test and duplicate samples.
- **Soil Sampling Equipment Decontamination Blank for Semivolatile Organics.** Rinse the field equipment prior to its use and collect the rinsate for analysis.
- **PCB Wipe Sample Equipment Decontamination Blank.** Use a wipe pad to wipe the sampling template in the same way the pad is handled during the actual wipe sampling of a surface.

One equipment decontamination blank is collected for each type of equipment used during the day or sampling event. Equipment decontamination blanks are assigned container numbers from the same sequence as the test samples, and may not be distinguished from the test samples on the COC record. More blanks may be collected depending on the data quality needs.

3.7.7.4. Field Blanks

Field blanks are prepared and analyzed to check cleanliness of sample containers, environmental contamination, and purity of reagents or solvents used in the field. A sample container is filled with laboratory ASTM Type I or II water, preserved, shipped to the field with clean sample containers, opened in the field to exposure to ambient field air for a time compatible to field sampling process, and is closed and submitted for analysis using the same parameters as the test sample. The reported results will indicate the presence of contamination. Field blanks are most often used when measuring for volatile analytes.

3.7.7.5. Trip Blanks

A trip blank is used with VOA analysis of water. A blank may consist of two 40-milliliter VOA vials filled at the laboratory with laboratory ASTM Type I or II water, transported to the

sampling site and returned to the laboratory without being opened. This serves as a check on sample contamination during sample transport and shipping.

Note: The caps used on VOA vials have Teflon®-lined septa. The Teflon® side of the silicone septum should face the sample. Prior to closing a vial, make sure there is no soil particle or dirt on the sealing surface of the VOA vial to prevent leaks. If a high concentration of volatile chemicals is present in the air in a shipping container, these chemicals can pass through the septum and contaminate the sample.

A trip blank is included in each shipping container used to ship VOA water samples. One VOA trip blank (two vials) is submitted to the laboratory in each cooler or per sampling event. The frequency of collection for trip blanks is specified in the SAP and is based on the data quality needs. Trip blanks are assigned container numbers from the same sequence used for the test samples, and are not designated as blanks on the COC record.

3.7.7.6. Matrix Spike (MS)/Matrix Spike Duplicate (MSD)

Project or compliance QC procedures require that the laboratory spike a portion of the matrix with a predetermined quantity of analyte(s) prior to sample extraction/digestion and analysis. The frequency of performing an MS is dependent on the data quality needs and method requirements.

A spiked sample is processed and analyzed in the same manner as the sample. The result of the analysis of the spike compared with the non-spike sample indicates the ability of the test procedures to recover the analyte from the matrix, and provides a measure of the performance of the analytical method executed by the laboratory.

For an MSD, a second portion of the matrix is spiked, and the recovery of the MSD can be compared with the recovery of the MS.

Depending on the matrix and analysis, additional sample containers may be specified to provide enough material for this laboratory procedure. These sample containers are assigned container numbers from the same sequence as the test samples and are designated MS/MSD materials on the COC record.

The MS/MSD samples are commonly used in CERCLA testing, but are not commonly used in CWA or SDWA testing. MSs are routinely performed by the laboratory as part of its internal QC on randomly chosen samples. If MS data is required for SDWA or CWA reporting requirements, a request must be made to the laboratory to ensure the MS is performed and reported on the appropriate sample. The sample selected for MSs should have the same or similar matrix as the field samples' but without high levels of target analytes.

3.7.8. Field Audits

The SAP will specify who will conduct field audits, along with their frequency and procedures. QA/QC procedures of the sample collection effort must identify and determine the magnitude of error associated with the contamination introduced through the sample collection effort. Audits are perhaps the most effective tool to ensure that the sampling is done correctly. The two factors most likely to influence the magnitude of the sample collection error are collection methods and frequency of sampling.

In general, a field sampling audit provides an independent outside check on the following:

- **Field Records**
 - COC records
 - Sample container labels
 - FLBs or field forms
 - Personnel training records
- **Sampling Procedures**
 - Equipment
 - Sample containers
 - Accuracy of sample location descriptions
 - Comparability of field sampling techniques
 - Collection and preparation of QC samples
 - Sample preservation
 - Equipment decontamination
 - Contaminated waste storage and disposal

- Sample packing, storage, security, and transportation
- Shipping containers, including use of custody seals (if applicable).

3.8. Generic Sampling Equipment List

Equipment specific to each type of media is found at the end of the related chapters. The following is a generic sampling equipment list:

- Map of sampling location(s)
- Sampling SOP
- FLB or field form
- Pens
- Containers
- Preservatives
- Labels
- Markers
- Coolers
- Ice
- Packing material
- Packaging tape
- COC form
- Custody seals (if required)
- Decontamination storage containers, equipment, and materials
- Personal safety equipment, safety test equipment
- Field screening or testing equipment, standards, reagents, and SOP
- Testing field forms or logbooks
- Laboratory instructions (if different from custody form).

Standard Operating Procedure (SOP) #6

SVE Treatment System Flow Meter Use

I. Equipment List

- Personal protective equipment including safety vest, sun/wind protection (if necessary), steel-toe boots and nitrile gloves
- Anemometer
- PTFE tape
- Tools
- Field logbook, indelible ink pens, field forms and camera

II. Procedure

- 1) The SVE System must be running prior to taking anemometer readings.
- 2) A monitoring port is located at the SVTU influent. The monitoring point will be located at least five pipe diameters away from any bend in the conveyance piping or any valve.
- 3) PTFE tape is wrapped around the anemometer to ensure a good seal is made when inserted in to the monitoring port.
- 4) The anemometer is powered on using the "On/Off" button and the probe is inserted into the port with the probe perpendicular to air flow.
- 5) Use the "Temp/Velocity" button to switch between measuring the temperature and velocity and use the "Range" button to switch ranges in the velocity reading if the reading is out of range. Use the "Units" button to switch between fpm (feet per minute) and mps (meters per second) and F (Fahrenheit) and C (Celsius).
- 6) Record the readings in the field logbook and field data sheet.
- 7) Power down the anemometer
- 8) Replace the cap on the monitoring port.

Eurofins Air Toxics, LLC
STANDARD OPERATING PROCEDURE

**ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN
SUMMA™ POLISHED CANISTERS**

**EPA METHOD TO-15 and MODIFIED EPA METHOD TO-14A
(5 & 20 ppbv)**

SOP #91

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1.0 SCOPE AND APPLICATION

The procedures in this SOP describe the use of EPA Methods TO-14A and TO-15 to determine the concentration of volatile organic compounds in ambient air, soil gas, or other vapor matrix using an evacuated specially treated stainless steel canister or a Tedlar bag. Note: Tedlar bags are not listed as suitable sample container in TO-14A or TO-15. This SOP details the analytical procedures and the required QC protocols. A list of target compounds can be found in *Appendix A*.

2.0 METHOD SUMMARY

2.1 Description

EPA Methods TO-14A and TO-15 describe techniques for the analysis of airborne VOCs collected as whole air samples in evacuated, specially treated stainless steel canisters. An aliquot of up to 50 mLs of air is withdrawn from the canister utilizing either a syringe or a mass flow controller and is loaded onto a hydrophobic multibed sorbent trap to remove water and carbon dioxide and to concentrate the vapor sample, see Work Instruction #WI91 for more information. The focused air sample is then flash heated onto a GC/MS for compound separation and detection. A summary of the method QC can be found in *Appendix A*.

2.2 Deviations

Eurofins Air Toxics takes no modifications of technical significance to Method TO-15. Since Eurofins Air Toxics applies TO-15 methodology to all Summa canisters regardless of whether TO-14A or TO-15 is specified by the project, Eurofins Air Toxics performs a modified version of method TO-14A as detailed in Table 1. Please note that Methods TO-14A and TO-15 were validated for specially treated canisters. As such, the use of Tedlar bags for sample collection is outside the scope of the method and not recommended for ambient or indoor air samples. It is the responsibility of the data user to determine the usability of TO-14A and TO-15 results generated from Tedlar bags.

Table 1. Summary of Method TO-14A Modifications

Requirement	TO-14A	EATL Modifications
Sample Drying System	Nafion Drier	Multibed hydrophobic sorbent
Blank acceptance criteria	< 0.2 ppbv	< RL
BFB ion abundance criteria	Ion abundance criteria listed in Table 4 of TO-14A	Follow abundance criteria listed in TO-15
BFB absolute abundance criteria	Within 10% when comparing to the previous daily BFB.	CCV internal standard area counts are compared to ICAL, corrective action for recovery less than 60%.
Initial Calibration	≤ 30% RSD for listed 39 VOCs	≤ 30% RSD with 2 of EATL's 62 standard compounds allowed out to ≤ 40%.

3.0 HEALTH AND SAFETY

- 3.1 Normal laboratory safety precautions must be used when handling samples, preparing standards from neat materials, and analyzing samples. Appropriate eye wear, gloves, and lab coat must be worn when handling any chemical used in this method. All manipulation of standards, solvents, and solutions should be done with the utmost care in the hood. MSDS for each chemical should be consulted for specific dangers and precautions.
- 3.2 Personnel must handle high pressure cylinders safely. This includes transport of cylinders fully secured on a cart. During storage, the cylinders must be secured at all times with a chain. When installing a pressure regulator, stand to the side of the cylinder.
- 3.3 Care must also be taken when handling syringes to ensure that a needle stick does not occur. *All personnel installing or performing maintenance on a capillary column must wear eye protection.*
- 3.4 For information regarding pollution prevention and waste disposal, see Eurofins Air Toxics SOP #24: Storage and Disposal of Hazardous Wastes.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 Sample Containers

An air sample is collected in an evacuated stainless steel Summa™ / canister or Tedlar bag. Tedlar bags are not included as acceptable media in either Compendium Method TO-14A or TO-15, but are accepted for analysis per client request. Upon receipt, the canisters will be approximately in the range of 2.5 - 10" Hg vacuum.

4.2 Sample Handling

Prior to analysis, the canister is pressurized in accordance with Eurofins Air Toxics SOP #60 (Canister Pressurization) and/or per project specific requirements.

By client request some samples are pressurized with Helium to allow for additional analyses by the laboratory (i.e. ASTM D-1946). However, the flow controllers used in the laboratory are calibrated for Nitrogen. Since Helium pressurized samples typically have a higher flow rate than Nitrogen pressurized samples, all samples that have been pressurized with Helium are loaded by syringe.

4.3 Sample Storage

Samples are stored in the sample cage in the main laboratory. Analysis must occur within 30 days for canisters. Some projects may require a 14 day hold for canisters. The project profile must be reviewed prior to analysis to verify holding time requirements.

Holding time in the case of Tedlar bags is limited to a maximum of 3 days as losses of VOCs are observed specifically for compounds with low vapor pressure. Holding time may be extended to a maximum of 30 days by transferring the sample from a Tedlar bag to a clean, evacuated Summa™ canister prior to the expiration of the 3 day hold. The transfer must be documented using the Tedlar bag transfer form and documented in the case narrative as per Eurofins Air Toxics SOP #69.

5.0 ***INTERFERENCES AND POTENTIAL PROBLEMS***

- 5.1 Interferences to this method generally include high concentrations of water. Very high levels of moisture in the samples may cause low internal standard responses which may interfere with accurate compound quantification. In these situations, dry lab blanks may be required between samples to maintain internal standard recoveries. In extreme cases, sample dilution may be required.
- 5.2 When a sample has high levels of acidic gases such as HCl and SO₂ and/or high levels of non-target compounds, the analyst may have to dilute the sample more than the target compound level requires. This ensures that internal standard recoveries meet QC requirements and/or that the system is not contaminated from carryover or damaged by high levels of acidic gases.
- 5.3 See Eurofins Air Toxics WI91 C2 interface (5&20) work instructions for examples when to red tag canisters due to high level concentrations.

- 5.4 Tedlar bags are not appropriate sample collection media for low vapor pressure compounds such as naphthalene due to severe adsorption and subsequent losses on the Tedlar surface. A sample discrepancy report (SDR) must be submitted when naphthalene measurements are requested from a Tedlar bag. All naphthalene results are flagged as estimated values.

6.0 ***EQUIPMENT/APPARATUS***

6.1 List of Equipment

- TO-15 Air Concentrator Systems (Documentation on system designs and manufacturer's information is maintained at each unit and is maintained on the secure QA drive.)
- Column: Restek RTX-624 20m X 0.18mm ID, 1.0µm Film. The alternative Restek columns, RTX-624 30m X 0.25mm ID or RTX-624 60m X 0.32mm ID, may also be used.
- Gas Chromatograph: Agilent 7890 or equivalent equipped with Electronic Pressure Control and a split/splitless injection port
- Mass Spectrometer: Agilent 5975, 5977 or equivalent
- Agilent Chemstation Software for Data Acquisition
- Thru-Put Target Software for Data Analysis
- NIST08.1 or NIST 11.1 Library Search Software
- Certified NIST Traceable VOC blends – Praxair, Linde, Absolute Standards, Accustandard, etc. A
- High Purity Neat Chemicals
- Zero-Air
- Tedlar Bags (1-20 L) SKC or equivalent
- Ultra High Purity Helium (Local Supplier)
- Liquid Nitrogen certified VOC free by TO-15 analysis
- Commercially purchased dilution system to blend working standards from stock cylinder standards, Entech 4700
- Laboratory Designed Canister Receiving Station equipped with high-resolution vacuum/pressure gauge and diluent gas (N₂, He, or Zero air) inlet.

(Documentation on system design is maintained at each unit and on the secure QA drive.)

6.2 Operating Parameters -GC/MS

Since analytical equipment varies in configuration and as a function of intended specific target analytes, operating parameters are optimized on an instrument-by-instrument basis. Instrument specific operating parameters are recorded in the respective Instrument Maintenance Logbook and in the QA-controlled Work Instructions at each work station.

Note: Only trained and qualified laboratory staff are authorized to vary the operating parameters under special circumstances. Any time a change is made, it is documented in the instrument maintenance logbook.

7.0 **STANDARD PREPARATION**

The formula used to calculate the volume required for the preparation of primary and second source standards for gas blends is the following:

$$C1*V1 = C2*V2,$$

Where, C1 is the concentration of the gas blend, V1 is the volume of standard to be used in the preparation, C2 is the desired concentration for the resulting standard and V2 is the final volume (at pressure*) of the newly blended standard.

*At 15 psi the final volume for a 6L canister is 12L and for a 1L canister it is 2L (volume reflects a twofold dilution as a function of pressurization).

7.1 Stock Standards

- 7.1.1 The TO-15 analytes are purchased from Praxair or other available vendors at a concentration of 1-5 ppmv in a high pressure cylinder blend. The standard expires according to the manufacturer's expiration date on the certificate of analysis.
- 7.1.2 Analytes not present in the commercially available blends are purchased in neat form from a commercial vendor. These compounds are then blended into the gas phase as described in SOP #33. Once the neat compound exceeds the manufacturer's expiration date, these standards must be purity-checked by the laboratory annually.

7.2 Working Standards

- 7.2.1 Starting with a clean evacuated canister, a volume of 100 microliters of purged deionized water is injected to humidify the standard prior to preparation.
- 7.2.2 Entech NT4700 Precision Diluter

The Entech NT4700 Precision Diluter is used to prepare primary and secondary standards for VOC analysis. The diluter prepares dilution of stock standards by monitoring small pressure changes digitally. There are a total of four positions on the diluter that can accommodate high pressure standard cylinders. There is an additional inlet to allow for secondary dilutions of working standards. See *WI-4700* for the complete work instructions.

- 7.2.3 Working standards prepared from source standards originating from neat or liquid mixes are prepared by injecting appropriate amounts of standard into an evacuated canister that contains 100 μ L of water. Details on how to prepare these working standards are provided in SOP #33. These working standards typically contain 'non-routine' compounds which are blended in canisters separate from the routine TO-15 VOC working calibration standard.
- 7.2.4 All working standards are pressurized to 15 psi with Zero Air. Two concentrations of working standard canisters are prepared: 50 ppbv and 200 ppbv. (Some compounds may be blended at concentrations 10x lower, e.g. Naphthalene.)
- 7.2.5 Working standards can be re-pressurized with zero air when pressure becomes near ambient. This re-pressurization step is recorded in the standard preparation logbook (See SOP #33 for details.) The concentration of the working standard is then adjusted to reflect the addition of zero air.
- 7.2.6 Working standards may be used for up to 3 months from the date of preparation. The TO-15 working standard may not be used past the expiration date of the NIST-traceable high pressure cylinder.

Note: Tedlar bags are used for the static dilution medium due to their inherent inertness to polar analytes vs. glass dilution jars. Standards made using a Tedlar bag should not, however, be stored in the Tedlar bags beyond one day. Fresh calibration standards are prepared and then transferred to SummaTM canisters for storage. The standards prepared from neat materials are stable in SummaTM canisters for 6 months.

7.3 Internal Standard/Surrogate (IS/S) gas blend

The IS/S gas blend is an internal reference material purchased from Praxair or another available vendor. The gas blend's nominal requested concentration is 10ppmv and the gas cylinder expires based on the manufacturer's date on the certificate of analysis. A 15L summa canister can be directly filled from this gas cylinder for use at the instrument as a working standard. Expiration date for this 15L summa canister is 3 months from the date of direct fill or the manufacturer's expiration date of the gas cylinder, whichever comes first.

7.4 Tune Check Standard

The IS/S gas blend contains 4-Bromofluorobenzene (BFB) at a nominal concentration of 10ppmv and is also utilized for tune checks.

8.0 ***CALIBRATION AND QUALITY CONTROL PROCEDURES***

The following sections outline the laboratory's routine calibration and quality control procedures. Specific programs such as DoD QSM 5.1 and projects may require additional QC samples and/or tighter QC criteria. These requirements are found in the Project Requirement Table (PRT) generated during the project set-up/QAPP review steps as outlined in SOP#1. PRTs are attached to the relevant profile in ATLAS and stored electronically on the network (O:\Project Requirement Tables).

8.1 Tuning Criteria

- 8.1.1 At the start of every 24 hours, a tune check with 4-Bromofluorobenzene (BFB) is performed by loading IS/S from the 10ppmv working standard onto the TO-15 air concentrator IS/S 2ml loop. A 50ml flow controller load from a laboratory blank is utilized to establish flow. See work instruction WI 91 for further details on flow controller loads. The final on column BFB concentration is 3.6ng.
- 8.1.2 The relative abundances of selected ions are tabulated and reported as outlined in *Appendix B*. Analysis cannot proceed unless all criteria of the tune check are met in accordance with EATL SOP # 53. The acceptance criteria are based on TO-15 which are wider than tuning criteria outlined in TO-14A.
- 8.1.3 The BFB standard must be re-analyzed when the EM voltage is changed or when the instrument has been re-tuned.

8.2 Initial Calibration Procedures

Initial Calibration of the GC/MS is achieved via the internal standard technique. The multi-point initial calibration is generated by loading volumes of the concentrated stock standards and the working standards. The on-column concentrations typically range from 5.0 to 5000 ppbv. Other levels may be added to the calibration per specific project requirements.

8.2.1 Standard Compound criteria

Standard (routine) TO-14A/TO-15 compounds are listed in Table A-1. Initial Calibration is performed using a minimum of five levels for

standard TO14A/TO-15 compounds. The low-level standard must be less than or equal to the reporting limit (Limit of Quantitation) and must be verified on a quarterly basis.

The percent relative standard deviations (%RSD) must be $\leq 30\%$ for all standard compounds, with two exceptions not to exceed $\leq 40\%$.

8.2.2 Non-Standard Compound criteria

Compounds that are not included in Table are defined as non-standard compounds. Examples of Non-Standard TO-14A/TO-15 compounds are listed in Table A-b. A one, three or five point calibration may be performed for non-standard compounds with documented client approval. The requirements for TPH are described in SOP #111. The lowest level standard must be less than or equal to the reporting limit. In general, LOQ (Limit of Quantitation) evaluations are not maintained for non-standard compounds which are not part of DoD accreditation.

8.2.2.1 Initial calibration criterion for non-standard compounds is $\leq 40\%$ RSD subject to verification of client requirements in the Project Requirement Table. It is the responsibility of all analytical personnel to verify agreed upon Initial Calibration requirements prior to sample analysis as more stringent criteria than $\leq 40\%$ RSD may be required.

8.2.2.2 Scientific experience and knowledge must be applied to the performance evaluation of specially requested compounds. It may be that a non-standard compound does not perform well due to its vapor pressure or instability. In these cases, the clients are notified of this non-conformance to TO-15 performance requirements, and data qualifiers may be required.

8.2.3 After an Initial Calibration has been evaluated and meets laboratory criteria, the midpoint is copied by adding an extension onto the file identifier, and then it is requantified as a Continuing Calibration Verification. All calibration points are then requantified and the Internal Standard area counts and Retention Time Windows for each file are compared to that of the midpoint. Internal Standards in each calibration point compared in this manner must meet laboratory criteria (area count $\pm 40\%$ and Retention Time ± 0.33 minutes compared to the CCV). If %D criterion for the CCV (see section 8.4) is met, analysis of the Initial Calibration Verification Standard may proceed. On days when an Initial Calibration is not performed, the Internal Standard area counts and Retention Time Windows in all subsequent samples and QC must meet

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laboratory criteria (area count \pm 40% and Retention Time \pm 0.33 minutes compared to the CCV analyzed at the beginning of the analytical sequence).

- 8.2.4 The average relative response factor (RRF) from the Initial Calibration Curve is used to quantitate results.
- 8.2.5 All initial calibrations needing re-analysis of a calibration level require explanation in the Initial Calibration Case Narrative Template. One (1) calibration level is allowed for re-analysis per Initial Calibration Curve due to anomalous unacceptable linearity for compound(s). A bad load or unopened can does not count towards a re-analysis. If more than one standard is used for curving an instrument (i.e. 2 or more), and it is obvious that the RF is not linear relative to the other points of the curve, then all the points ran from that standard must be re-analyzed. The reason for the reanalysis must be narrated and included with the ICAL raw data.
- 8.2.6 The reporting limit (Limit of Quantitation – LOQ) must be verified quarterly on each instrument that performs the methods the lab is accredited for by DoD-ELAP (Refer to DoD scope at O:\QA\Certifications). The LOQ is verified by evaluating the point of the initial calibration that corresponds to the LOQ. The concentration recovered is used to calculate the precision and bias of each compound. A minimum of three points is required to perform the calculations. If there are insufficient points, a primary source standard is analyzed at the LOQ. Precision and bias is determined by calculating the % relative standard deviation (% RSD) and the % bias of the mean concentration recovered. The acceptance criterion for the LOQ verification is \leq 30% RSD and \leq 50% bias for each compound. If more than 10% of the compound list exceeds the acceptance criteria, the instrument and standard used will be evaluated to determine the source of the error. Quarterly LOQ verifications are evaluated by the QA Department and the values are maintained on a secure network drive.
- 8.2.7 All current Initial Calibrations and Method Detection Limit (MDL) studies are kept in a folder near the instrument.
- 8.2.8 Initial calibration curves are generated in accordance to appropriate laboratory practices:
- 8.2.8.1 The lowest or highest calibration levels of an analyte may be dropped from the curve to achieve linearity. This will affect the analyte's reporting limit and/or calibration range. The minimum number of calibration levels must still be used for the curve.

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8.2.8.2 An additional mid-range calibration level may be added to achieve linearity.

8.2.8.3 Alternate calibration levels may be used to meet client requirements.

8.2.8.4 It is not acceptable to drop a calibration level that is found somewhere the middle of the curve to achieve linearity (i.e. dropping the 50ppbv or the 25ppbv calibration level). The calibration level that is in question may be re-analyzed and re-quantified into the curve.

8.2.9 The indicated flow on the instruments must be measured and the units must be calibrated with a NIST flow meter to provide a true indication of the actual flow through the unit before each time an Initial Calibration is performed and/or on a quarterly basis, whichever is more frequent.

8.3 Initial Calibration Verification. (ICV)

8.3.1 Standard Compound criteria

An independently prepared (i.e., same vendor, different lot number or second vendor) standard containing all target compounds is analyzed after each Initial Calibration Curve, to verify that the standards are correct and the calibration is accurate. The acceptance criterion for the ICV recoveries is as follows: For the compounds listed in Table A-1, recoveries for 85% of the compounds must meet the listed acceptance criteria with no recoveries <50%.

8.3.2 Recoveries of any compound that is found to exceed these criteria, but are within 50-150%, are narrated on the ICAL Narrative sheet that is attached to the ICAL packet. However, exceeding the acceptance criterion of 50-150% recovery permits for a reanalysis of an ICV.

8.3.3 Recovery of any compound in the ICV that is < 50% of the expected value will result in standard re-preparation, system maintenance if needed and re-calibration, or sample analysis on a different instrument. Recovery of any compound that is $\geq 150\%$ of expected value, analysis may continue with manager approval after evaluation of whether the data meets client project needs. However, the system and/or standard preparation should be evaluated. If the problem is determined to be systematic (i.e. occurs on more than three consecutive days), corrective action outlined above should be conducted to resolve the issue.

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8.3.4 Non-Standard Compound criteria

An Initial Calibration Verification will be performed for non-standard compounds only when prior arrangements with the client have been made and are documented in the Project Requirement Table. Initial calibration verification criterion for these analytes is 60 - 140% Recovery and is subject to verification of client requirements in the Project Requirement Table. It is the responsibility of all analytical personnel to verify the agreed upon ICV requirements prior to sample analysis as more stringent criteria than 60 - 140 % may be required. If the non-standard ICV does not comply with the client requirements in the Project Requirements table, then refer to section 8.2.2.2 and discuss with the manager.

8.3.5 See SOP #111 for requirements for TPH.

8.4 Continuing Calibration Verification (CCV)

A Continuing Calibration Verification (CCV) is performed at the start of each day after the BFB Tune Check. This is an analysis of the primary source at a concentration between the low point and the midpoint of the initial calibration.

8.4.1 Standard Compound criteria

The acceptance criteria for the percent Difference (%D) between the daily CCV response and average response from the Calibration Curve is as follows: All standard CCV compounds must be $\leq 30\%$ D. Any compounds exceeding this criterion will be flagged and associated data likewise flagged and narrated. If more than two compounds from the standard list recover outside of 70-130%, corrective action will be taken. Corrective action may include instrument maintenance, re-calibration, and/or re-preparation of the calibration standards (See 8.4.3). If any compound exceeds 60-140%, samples are not analyzed unless data meets project needs. The QA Manager or Lab Manager may approve exceedance of a compound under special circumstances after reviewing the impact to the data quality. Regardless, associated data will be flagged and narrated.

8.4.1.1 If a list of ≤ 20 target compounds is requested, no more than 10% of the compounds may be outside 70-130% criteria. For example, if a client is requesting 6 compounds then 0 compounds are allowed out; or if 15 compounds are requested then only 1 compound may be outside acceptance criteria.

8.4.2 Non-Standard Compound criteria

Compounds that are not included in Tables A-1 and A-1b are defined as non-standard compounds. Examples of Non-Standard TO-14A/TO-15 compounds are listed in Table A-1c. CCV criterion for these analytes is $\leq 40\%$ D subject to verification of client requirements in the Project Requirement Table. It is the responsibility of all analytical personnel to verify agreed upon CCV requirements prior to sample analysis as more stringent criteria than $\leq 40\%$ D may be required.

8.4.3 If the CCV fails to meet the performance criteria the CCV is reanalyzed and/or another standard is analyzed. If the CCV check fails, system maintenance should be performed and the test repeated. If the system still fails the calibration check, a new Calibration Curve is performed. If the CCV passes following maintenance to the instrument then the LCS must also pass before samples can be analyzed.

8.4.3.1 Certain projects have different CCV acceptance criteria (e.g., $\leq 25\%$ D for a set list of compounds). A specific list of target analytes is required when the CCV acceptance criteria required differ from the EATL standard criteria noted above.

8.4.3.2 Sensitivity Verification

8.4.3.2.1 A calculation is performed comparing the area counts of all internal standards from the midpoint of the Initial Calibration to the same internal standard in the continuing calibration verification to verify the instrument has not lost sensitivity from the ICAL to the daily batch. The area counts are compared with a maximum allowable percent drift of 40% D below the mid-point ICAL value. If the comparison passes then it should be documented in the instrument logbook. If the IS comparison fails this test another CCV will be analyzed. If this fails the comparison test as well, then the multiplier may be adjusted and the test repeated. If this also fails the $\%D$ test, the reason for the discrepancy is investigated, and if necessary a new analytical curve will be performed. A sensitivity check at the reporting limit or lower must be analyzed following the replacement of the concentrator trap. Once it has been verified as passing criteria, the analyst may proceed with

analysis of a CCV. If the sensitivity check fails, the problem must be resolved and a new curve analyzed.

8.4.3.2.2 Sensitivity must be evaluated for instances where the IS area counts in the CCV are greater than 40% drift when compared to the mid-point ICAL value. When this condition exists, all positive results greater than half the upper calibrated level, but less than the upper calibrated level, must be evaluated for saturation. If evidence of peak saturation is determined to be likely, as exhibited by more than two flat scans across the top of the peak in question, or a higher than normal abundance of secondary and tertiary ions in relation to the base peak, reanalysis of the sample in question must be performed at a dilution. The dilution should not exceed a factor of two greater than the original analysis in order to maintain reporting of all positive results detected in the initial analysis.

8.5 Laboratory Control Standard (LCS)

- 8.5.1 A Laboratory Control Spike is analyzed daily prior to sample analysis. Recovery limits are listed in Table A-1. Recoveries for 85% of standard compounds must be 70-130% with no recovery at <50%. If specified by the project in-house generated or DoD specified control limits may be used.
- 8.5.2 If the stated criteria are not met, the system is checked and the same standard or different standards are re-analyzed. In the event that the criteria cannot be met for the full TO-14A/TO-15 list of compounds, a review of the data will be done by a QA Manager or Lab Manager and may approve the exceedances of a compound under special circumstances after reviewing the impact to the data quality. If approved, the data will be flagged and narrated.
- 8.5.3 LCS criterion for Non-Standard analytes is 60 – 140 % recovery subject to verification of client requirements in the Project Requirement Table. It is the responsibility of all analytical personnel to verify agreed upon LCS requirements because in some cases, a commitment has been made to criteria more stringent than 60 – 140%.
- 8.5.4 Recovery of any compound in the LCS that is $\leq 50\%$ of the expected value will result in either re-calibration, or analysis on a different instrument. Recovery of any compound that is $\geq 150\%$ of expected value, analysis may

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continue; however the system and/or standard preparation should be evaluated. If the problem is determined to be systematic (i.e. occurs on more than three consecutive days and/or on multiple instruments), the instrument must be evaluated and recalibrated if needed.

- 8.5.5 Some projects require the LCS to be evaluated using control limits that are derived from historical data or specified in the DoD QSM. Refer to SOP #48 "Preparation and Review of Control Charts" for the generation of control limits procedure. Recovery of any compound in the LCS that is outside of the historically derived control limits but within the default limits of 70-130%, analysis may continue and the excursions flagged and narrated.
- 8.5.6 An LCS is analyzed in duplicate (LCSD) daily prior to sample analysis. Refer to section 8.9.1 for the RPD acceptance criteria between the LCS and LCSD.
- 8.5.7 LCSD % Recovery acceptance criterion must be met for common risk driver compounds including Benzene, Toluene, Ethyl Benzene, m,p-xylene, Vinyl Chloride, 1,1-Dichloroethene, cis-1,2-Dichloroethene, Trichloroethene, Tetrachloroethene or any other client specified risk driver compounds.

8.6 Internal Standards

- 8.6.1 Two mLs of the IS/S blend is injected into the canister interface as each standard, blank, and sample is being loaded. The final concentration is 400 ppbv for each noted compound:

Bromochloromethane
Chlorobenzene-d₅
1,4-Difluorobenzene

- 8.6.2 Internal Standards Retention Times for the blanks and samples must be within ± 0.33 minutes of the Retention Times in the Continuing Calibration Check. In addition, the IS area must be within $\pm 40\%$ of the CCV's IS area for the blanks and samples. The acceptance criteria are not subject to rounding. If the area count is below the lower limit as established by the CCV then the IS is deemed to have failed acceptance criteria and the steps in sections 8.6.2.1 and 8.6.2.2 are followed.

- 8.6.2.1 If the ISs for the blank do not pass the acceptance criteria, the system is inspected and the blank reanalyzed. Analysis is discontinued until the blank meets the IS criteria.
- 8.6.2.2 If the ISs in a sample do not pass the acceptance criteria, the sample must be reanalyzed. If the ISs are within limits in the reanalysis, the second analysis will be reported.
- 8.6.2.3 Oftentimes, the cause of low ISs in a sample are due to high levels of moisture in the sample. If this is suspected, a dry lab blank analysis is recommended prior to sample re-analysis. The affect of water is cumulative on the unit. If the ISs are out-of-limits a second time, then the sample will be diluted to get the ISs within limits. After an out-of-limit condition, a subsequent analysis must be done to demonstrate that the system is in control. If the ISs are within limits after the blank, the sample is analyzed again. If the sample causes the IS to be out-of-limits a second time, then it is determined that the sample contains matrix interference since the instrument was found to be in control when a system lab blank was analyzed. The data will be marked as acceptable and narrated accordingly.

8.7 Surrogates

- 8.7.1 Two mLs of the IS/S blend is injected into the canister interface as each standard, blank, and sample is being loaded. The acceptance limits for surrogate recoveries are 70 to 130% recovery. Concentrations of Surrogates are equivalent to internal standard concentrations:
- 1,2-Dichloroethane-d₄
 - Toluene-d₈
 - 4-Bromofluorobenzene.
- 8.7.2 If the surrogate recoveries for the QC samples (i.e. CCV, LCS, or blank) do not pass the acceptance criteria, the system is inspected and the QC sample reanalyzed. Analysis is discontinued until the QC sample meets the surrogate recovery criteria.
- 8.7.3 If the surrogate recoveries for a sample are outside of these limits, the sample is reanalyzed unless obvious matrix interference is documented. If the surrogate recoveries are within limits in the re-analysis, the second analysis will be reported.

- 8.7.4 If the surrogate recoveries are out-of-limits a second time, the data from the first analysis will be reported with a narrative indicating the acceptance criteria for surrogate recoveries were exceeded. Additionally, the system must be shown to be in control by the analysis of a blank or sample with acceptable surrogate recoveries. Upon request, the data from the matrix effect confirmation analysis is provided to the client.
- 8.7.5 Some projects require the Surrogates to be evaluated using control limits that are derived from historical data. Refer to SOP #48 "Preparation and Review of Control Charts" for the generation of control limits procedure.

8.8 Laboratory Blank

- 8.8.1 A humidified Zero-Air Blank is analyzed upon completion of all required QC including calibration standards at the beginning of each day and at least once in every batch or 24 hour shift. A blank is also analyzed in the event that saturation or when high concentrations over the initial calibration are measured in the sample. The acceptance criterion for Laboratory Blanks is a result less than the laboratory reporting limit (Limit of Quantitation) (*see Appendix A*).

8.8.1.1 In the event that the Laboratory Blank is contaminated by target compounds, Tentatively Identified Compounds (TICs), Non-Methane Organic Hydrocarbons (NMOC), or Petroleum Hydrocarbons (TPH) the following protocol should be observed: First, the Laboratory Blank should be reanalyzed to eliminate the possibility of an anomalous result. If re-analysis is acceptable, analysis of client samples may continue. Second, client samples that have analyte sublists that do not include the compound(s) in question should be substituted for full list samples. As these samples are analyzed, the operator should monitor the results to determine if the contamination has been removed from the system. In the event non-target contamination is present in the Laboratory Blank, analysis of samples not requiring TIC's, NMOC, or TPH calculations can proceed.

8.8.1.2 If there are no samples in-house that meet the criteria described in 8.8.1.1, the nature of the contamination should be evaluated. Samples that have been analyzed that cannot be re-analyzed will be flagged to note the non-conforming result.

8.8.1.3 Methylene Chloride, Acetone, Acetonitrile, Tetrahydrofuran, and 2-Butanone are acknowledged as common laboratory contaminants. Presence of these compounds at a concentration of <

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5X the client's reporting limit (Limit of Quantitation) is acceptable as long as the associated samples are not being analyzed for these analytes only. The associated hits for these compounds in samples will be flagged. If the contamination persists for more than two analytical batches, the issue should be escalated for evaluation in order to identify and remove the source of contamination.

8.8.1.4 If other target compounds are present in the Laboratory Blank, the appropriate Project Manager is notified and, as needed, will involve affected clients to determine whether flagged data for the compound(s) in question will meet their project's quality objectives.

8.8.1.5 If the analysis request is for the full list of compounds, laboratory blanks are not needed to continue running samples for Acetone, Ethanol, and 2-Propanol above the lab blank 'trigger'. These compounds can be present at high levels in air samples and are not critical risk drivers for client projects. If the sample or samples following the high level analysis contain these compounds below 5x the reporting limit then a narration must be used describing the potential for high bias of these results. If the result for these compounds in the subsequent samples is greater than 5x the reporting limit then no narration is necessary. If 10 or fewer compounds are requested and the list contains these compounds, blanks must be used to demonstrate cleanliness of the system.

8.8.2 Some projects including DoD projects require specific acceptance criteria for Method Blank as follow:

No analytes detected at $\geq \frac{1}{2}$ the RL. For common laboratory contaminants, no analytes detected \geq the RL.

If an analyte in the laboratory blank fails these criteria, a different Laboratory Blank canister is analyzed to rule out canister contamination. If the analyte in the Laboratory Blank still fails these criteria, the data is flagged with the appropriate data qualifying code (B) and the non-conformance is narrated unless, the analyte resulted in a non-detect in the samples.

8.8.3 In general, manual integration of target peaks in lab blanks is not permitted if the manual integration causes the analyte of interest to be below the reporting limit (Limit of Quantitation), except in the case of baseline contribution to the total ion current of a TIC or surrogate (usually Bromofluorobenzene) when calculating NMOC/TPH-G/TVH

concentrations. However, there may be times that integration of compounds in the Lab Blank is necessary to accurately represent the data. All manual integrations on Lab Blanks must be signed by either a qualified Lab Manager or the QA Manager. Refer to SOP #52 “Manual Peak Integration and Background Subtraction for GC/MS Analyses”.

8.9 Laboratory Duplicates

8.9.1 Every daily analytical batch must include an LCS and an LCSD to evaluate instrument precision. The acceptance criteria for the relative percent difference (RPD) between the LCS and LCSD analyses should meet $\leq 25\%$. Exceedances are narrated in the final report. Corrective action occurs when more than 5% of the compounds exceed this criterion or if any compound exceeds 40%RPD.

8.9.2 A duplicate sample analysis is performed only when specifically required by the Project Profile and/or Project Requirement Table associated with the workorder. The Relative Percent Difference (RPD) between the two analyses must be $\leq 25\%$ for all compounds detected at greater than 5 times the reporting limit (Limit of Quantitation). If this limit is exceeded, the sample is re-analyzed a third time, or analyzed on a different analytical system. If the limit is exceeded again, the cause is investigated and the system brought back to working order. If no problem is found on the system, the data is flagged to note the non-conforming event.

8.9.2.1 When three analyses do not result in acceptable precision ($\leq 25\%$ RPD for all compounds $> 5X$ LOQ), the instrument shall be eliminated as a potential source of the failure. This may be accomplished by choosing another sample and performing a duplicate analysis to determine if precision is possible in that instance. To whatever extent is possible, the original analytical conditions should be duplicated as well. This would imply use of the same syringe if relevant etc. Details to ensure duplication of original analytical conditions are left to the judgment of a scientist, Lab Manager, or the QA Manager. Do not dispose of the sample until the issue is resolved.

8.10 Field QC Samples

8.10.1 Neither TO-14A nor TO-15 describes field QC sample collection or acceptance criteria. However, clients may collect Field Blanks, Trip Blanks, and Field Duplicates. While there is no acceptance criteria established, analysts must monitor performance and note anomalies. For example, a positive result in the Field or Trip Blank requires closer

inspection to ensure the anomaly wasn't incurred during sample handling and loading. Inspection may include verification of the canister analyzed and verification that the analytical system was clean.

8.10.2 Likewise, a field duplicate sample which shows an inconsistent chromatographic pattern as compared to the paired sample or concentrations differing by more than 40%RPD for many of the detections requires further investigation at the time of sample loading. Verification of the canisters identification and load volume and comparison of the FID screens are all appropriate to verify results.

8.10.3 Notate on the data checklist the anomaly and the items verified. If an anomaly was uncovered, then the field QC should be reanalyzed. If there are no findings then an SDR is generated informing the appropriate project manager following a note on the Data Review Checklist indicating the non conformance. Narrate the presence of a detection in the Trip Blank and Field Blank.

9.0 CALCULATIONS

9.1 Response Factor

$$\text{Relative Response Factor (RRF)} = \frac{\text{Area of Compound}}{\text{Area of Int. Std}} \times \frac{\text{Conc. Int. Std (ppbv)}}{\text{Conc. of Compound (ppbv)}}$$

9.2 Sample Results

$$\text{Results Calculation} = \frac{\text{Area of Compound in Sample}}{\text{Area of Int. Std in Sample}} \times \frac{\text{Conc. Int. Std (ppbv)}}{\text{ICAL RRF}^*}$$

(ppbv on-column)

* *The average RRF from the Initial Calibration Curve is used to quantitate results.*

$$\text{ppbv in sample} = \text{ppbv on-column} \times \text{Dilution Factor}$$

$$\text{Dilution Factor} = \text{Pressurization Factor}^* \times \text{Analytical Dilution Factor}$$

$$\text{Analytical Dilution Factor} = \frac{\text{Max load volume of unit (ml)}}{\text{Sample volume loaded (ml)}} \times (\text{Off-line dilution factor})$$

* *The pressurization factor is determined by the lab measured canister receipt vacuum and final pressure. See Form 11.36 for calculations and a table of pressurization factors for a range of initial vacuums and final pressures of 5 and 15 psig.*

9.3 Total Petroleum Hydrocarbon (TPH) and Non-Methane Organic Compound (NMOC) calculations by GC/MS:

The calculations performed for TPH and NMOC as well as additional TPH characterization including the hydrocarbon fractionation is beyond the scope of this SOP and is detailed in SOP #111

9.4 Altitude Correction Factor for compound concentration

9.4.1 Per client's request, altitude correction factors may be applied to sample results using the following procedure: Atmospheric pollutant concentrations expressed as mass per unit volume of atmospheric air (e.g., mg/m³, µg/m³, etc.) at sea level will decrease with increasing altitude since the atmospheric pressure decreases with increasing altitude. The correction factor or change of atmospheric pressure with altitude can be estimated from the following equation:

$$\text{Correction Factor} = P_a = 0.9877^a$$

P_a = atmospheric pressure at altitude a, in atmospheres
a = altitude, in 100's of meters.

For example the altitude is 1800 meters. In 100 meters it would be 18.

$$P = 0.9877^{18} = 0.80$$

Apply correction factor to the final concentration. For example if the final concentration is 260µg/m³. Corrected concentration = 260 x correction factor = 260 x 0.8 = 208µg/m³ (See reference in section 15.0).

9.4.2 Correction factor must be provided to IT department in order for it to be applied to the formula for the final concentration. Results in the final report must be checked manually to validate the calculated result (see reference section for website information).

10.0 SAMPLE ANALYSIS

10.1 Analytical Batch

The analytical batch is defined as all samples* up to 20 analyzed within 24 hours on one instrument. All QC and samples must be processed with the method associated with the daily analytical batch.

*Samples per batch are reportable analytical runs excluding QC samples, dilutions, duplicate analysis and confirmation analysis.

10.2 Analytical Sequence

<u>Initial 24-hour period:</u>	<u>Subsequent 24-hour period:</u>
--------------------------------	-----------------------------------

BFB Tune Check	BFB Tune Check
Initial Calibration	CCV
LCS/LCSD	LCS/LCSD
Laboratory Blank	Laboratory Blank
Samples (up to 20)	Samples (up to 20)

The “Subsequent 24-hour” sequence is followed every 24 hour period during which samples are analyzed until the system fails quality control acceptance limits.

Additional QC samples may be required by the program or project to be analyzed within the analytical batch. These details are included in the project PRT.

In the event of a daylight savings transition, a time shift may be observed during analysis of samples. If this occurs, data is acceptable and may be used. The occurrence must be documented in the run log and on the Data Review Checklist of any affected work orders.

10.3 Validation of Reporting Limit (Limit of Quantitation)

10.3.1 Method Detection Limit (MDL) studies are analyzed for all standard TO-14A/TO-15 compounds following procedures as described in 40 CFR Pt. 136 App. B and Eurofins Air Toxics SOP #39.

10.3.2 The reporting limit (Limit of Quantitation) must be greater than the MDL before sample analysis can occur. If this is not achieved, corrective action, including raising the reporting limit is taken prior to continuing with sample analysis. See Appendix A for the TO-14A/TO-15 Reporting Limits.

10.3.3 In general, the reporting limit (Limit of Quantitation) is typically up to 10 times greater than the MDL but may be up to 50 times greater. If the reporting limit (Limit of Quantitation) exceeds this range, the Lab Manager and/or the QA Department will evaluate the reporting limit to determine the reason and its approval.

10.3.4 The MDL verification sample must be analyzed on a quarterly basis. Refer to SOP #39 and WI39 MDL Procedure for the acceptance criterion.

- 10.3.5 A Method Detection Limit study will be performed for non-standard compounds only when prior arrangements with the client have been made and are documented in the Project Requirement Table. The reporting limit must be greater than the MDL before sample analysis can occur.

10.4 Quantitative Analysis

Quantitation is based on the integrated abundance of the primary quantitation ion for each analyte. See *Table A-5*. If the response for any primary quantitation ion exceeds the Initial Calibration range of the GC/MS system, the sample is diluted and re-analyzed, excluding detections of the compounds noted in Section 10.5. If the response for any primary quantitation ion results in a value that rounds to the equivalent of the upper calibration range when expressing the result with 2 significant figures, re-analysis is not necessary.

- 10.4.1 When interference with the primary quantitation ion occurs, either the result is flagged with an “M” indicating matrix and a probable high bias or quantitation using the secondary ion is carried out after a new response factor (using the secondary ion) is generated from the initial calibration. Therefore, the same ion used to establish the Response Factor is used to quantify target analytes in the sample. This is noted in the laboratory narrative included in the report.

The criterion for using the secondary ion for quantitation is a difference in the reported result of 50% or more. Discussion with the project manager and/or QA Manager may determine that quantitation using the secondary ion is not necessary given the objectives of the project and flagging will suffice. In the case when the difference is less than 50% and/or there is interference with the primary and secondary ion, the possibility of high bias is notated on the Data Review Checklist and in the final report.

10.5 Detections Outside of the Calibrated Range

- 10.5.1 Per client request only, compounds detected between current instrument MDL and the Limit of Quantitation are reported and given “J” flags (Estimated Concentration).
- 10.5.2 Unless specifically directed otherwise by a client, detections of the following non-critical risk driver compounds: Acetone, Cyclohexane, Ethanol, Heptane, Hexane, 2-Butanone, 2-Propanol, Propylene, 2,2,4-Trimethylpentane, and Tetrahydrofuran above the high level of the curve are reported with an “E” flag and do not result in further dilution.

10.6 Tentatively Identified Compounds (TICs)

- 10.6.1 By client request, based on computer generated searches, the identification of the ten highest non-target unknown peaks is reported. The spectra of these peaks are searched against the NIST library of greater than 50,000 compounds.
- 10.6.2 The total ion current is used for quantitation and calculation of TIC results. A TIC is determined to be present when the quantitated concentration is found to be greater than one tenth the concentration of the spiked Internal Standard (i.e. for standard levels: 400 ppbv = 40ppbv).
- 10.6.3 The total ion current of the closest (by retention time, RT) non-interfered Internal Standard is used to calculate results (per SW-846 protocol). If all Internal Standards have interference, the Internal Standards in the Lab Blank are used to calculate results. A relative response factor of “1” is assumed. Match quality is useful in determining whether or not a tentative identification should be reported, but is not the only criteria.
- 10.6.4 There are cases in which the NIST library match contains masses for a compound that are not within the conventional scan range. In this instance match quality may appear to be poor when that is not the case. Eurofins Air Toxics protocol is to defer to the analyst’s judgment and experience with regard to identification.
- 10.6.5 If a tentative identification is made for a peak in one sample in a work order and a peak demonstrating similar spectra and elution time is present in another sample contained within the set, identification should remain consistent between the two samples. Peaks that exhibit obvious resolution but are integrated as a single compound should be split manually to evaluate concentration as accurately as possible.

10.7 Compound Identification

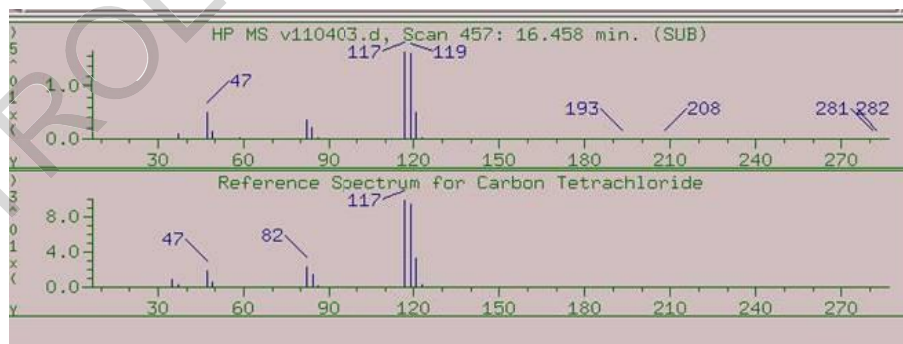
- 10.7.1 There are three criteria that must be satisfied to justify positive identification of a given compound.
1. The retention time must match the daily standard within a factor of less than 0.1 minutes for all characteristic ions (quantitation and qualifier ions) present in the spectrum. Shifts in target compound retention times that exceed this limit must be accompanied by a corresponding shift in retention time for the corresponding Internal Standard.

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2. The peak shape should be Gaussian for all monitored ions of interest with the exception of those at or near the reporting limit.
3. The mass ion fragmentation pattern should match the reference spectrum unless obvious interference is noted. There are two tools in the Target data processing software to determine a match – the compound’s spectral pattern (relative ion intensities) and the characteristic ion peak area ratios. In general, the visual assessment of the spectral pattern is sufficient to proceed with the identification; however, the ion area ratios can provide quantitative confirmation when interference is not present and qualifier ions are properly integrated.

Spectral Pattern: The spectral pattern displays the ion intensities at the peak scan. Evaluating the ion intensities at the peak scan is analogous to evaluating ion peak “height” at the retention time. As such, the spectral pattern is not affected by peak integration of the quantitation and qualifier ions. The relative intensity of the characteristic ions and the full spectral fragmentation pattern is compared to the reference spectra for a visual confirmation. See Figure 1 for an example of spectral pattern evaluation for a full scan data file.

Figure 1. Spectral pattern evaluation showing match of quantitation and qualifier ions (119 and 117) relative intensity as well as entire fragmentation pattern with the reference spectrum.



Ion area ratio: If needed, ion area ratios can also be evaluated using the integrated area of the quantitation ion and qualifier ions. As a guide, the ion area ratios of a compound should be within approximately 30% of the relative areas of these ions in the reference standard. The mid-point of the initial calibration curve is generally used as the reference to determine the ion area ratio target ranges. Figure 2 below shows the evaluation of the ion area ratio using the

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target ranges on the quantitation pages. (Please see Form I 1.80 for instruction to set up Target method to correctly show ranges in the Target report page.) Ion area ratios are less useful when the qualifier ions are not properly integrated due to low response or interference.

Figure 2. Ion area ratio evaluation showing compound ratio is within expected target range.

RT	EXP RT	(REL RT)	MASS	RESPONSE	CONCENTRATIONS		TARGET RANGE	RATIO
					ON-COL	FINAL		
59	Carbon Tetrachloride					CAS #:	56-23-5	
16.458	16.457	(1.033)	119	589303	7.20066	7.201	80.00- 120.00	100.00
16.458	16.457	(1.033)	117	601525			69.67- 129.67	102.07

10.7.2 To evaluate interferences and aid in comparison of the peak spectra to the reference spectral pattern, background subtraction can be used by the laboratory staff as a data evaluation tool to remove interfering ion masses from co-eluting compounds that may be masking the target compound. If background subtraction is needed in order to make a decision regarding the presence of a target compound then the subtraction should be included in the data package. The background subtraction of the spectra should be within ± 20 scans of the target peak. If the decision can be made without the use of background subtraction (i.e. retention time, ion abundance or area ratio match, and Gaussian peak shape) then it is not required to be included in the data package. Some ions in the reference spectra are not within the scan range (35-350 amu) and as such are not considered to be relevant to a positive identification.

10.7.3 If the above criteria are met then the compound has been positively identified. However, matrix interference may make it impossible to satisfy one or all of the above conditions. In these instances the chemist must evaluate the available data and determine whether the compound should be positively identified on the basis of the chemist's "best judgment". Advanced data analysis tools are available in the data processing software to aid in this evaluation as needed.

10.8 Manual Integrations

At times performing manual integrations on Initial Calibrations standards, QC, Laboratory Blanks and samples may be necessary. To accurately document ion ratios on the Target report page, if manual peak integration for quantification ion is needed in the ICAL or QC samples, then the qualifier ion peak shape needs to be evaluated as well and properly integrated if needed. Refer to SOP #52 for Manual Integrations by GC/MS for proper manual integration procedures and documentation.

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10.9 Analytical Procedures

The sample containers are connected to the inlet line of the TO-15 concentrator. Following the work instructions for the specific system, the sample connection is checked for leaks, and the sample is loaded onto the system. During the load step, a 1 mL gas sample loop filled with IS/S is swept onto the sorbent trap. If samples are pressurized with Helium, then they are loaded by using a syringe.

10.10 Sample Dilutions

- 10.10.1 To obtain analyte concentrations within the calibrated range of the detectors and to prevent contamination of the system, samples may be screened prior to analysis on a GC/FID. The total VOC concentrations are approximated and used to determine the volume of sample loaded for TO-14A/TO-15 analysis.
- 10.10.2 An undiluted analysis involves loading 50 mLs (or the standard load volume as identified by the ICAL) of the sample. The Dilution Factor is obtained by dividing the full load volume of 50 mL by the sample volume loaded.
- 10.10.3 Alternatively, samples that contain over approximately 50,000 ppbv of target analytes are diluted by adding a measured aliquot of the sample to a Tedlar bag that has a metered volume of Nitrogen as the diluent gas. For this alternative dilution technique, up to 50 mL of the diluted sample is loaded. As mentioned in Section 9.2, the final Dilution Factor includes canister pressurization dilution as well. Furthermore, a Summa canister may be used for preparation of sample dilutions per project specifics.
- 10.10.4 The appropriate dilution should result in one of the following:
- The most concentrated target compound detected from approximately 60% to 100% of the calibrated range. For the 5&20 calibration range this means targeting between 4000 and 10000 ppbv for compounds that are calibrated to 10,000 ppbv, 2000 to 5000 ppbv for compounds that are calibrated to 5000 ppbv, and 400 to 1000 ppbv for compounds that are calibrated to 1000 ppbv.
 - the total area count >100,000,000 area counts,
 - TICs >25,000 area counts,
 - or, if screening data indicates that they may saturate the detector.

Analyst judgment must be used to avoid damage to the instrumentation resulting from sample loads containing high levels of non-target analytes. Typically, peaks that are greater than ten times the height of the nearest

Internal Standard are evidence of non-target contamination that is concentrated enough to eliminate the need of a higher volume load. Screening data can be very helpful in determining optimal sample volume loads for in consideration of both target and non-target compounds. In addition, unless specifically directed to do so by the client, detections of the following compounds at levels above the high end of the curve do not result in a subsequent dilution: Acetone, Cyclohexane, Ethanol, Heptane, Hexane, 2-Butanone, 2-Propanol, Propylene, 2,2,4-Trimethylpentane and Tetrahydrofuran. Instead, these results are reported with an “E” flag to indicate the levels detected exceeded the upper limit of the range of calibration.

- 10.10.5 In the event the Project Profile requires that all analytical runs be reported, the analytical runs must be evaluated at the time of analysis to ensure that the RPD for compounds within linear range and greater than 5 times the RL do not exceed 25%. Re-analysis or system inspection/maintenance may be required to resolve any discrepancies.
- 10.10.6 A detailed procedure for performing sample dilutions can be found in WI91 (Work Instructions for C2 interface for 5&20).

11.0 CORRECTIVE ACTION PROCEDURES

A Request for Corrective Action (CAR) is initiated any time either the EATL SOPs or client-prescribed QC protocol are not followed, or in any other instance that sample results are adversely affected. The corrective action procedure is documented in EATL SOP #61.

12.0 DATA REVIEW

12.1 Analytical/Write-Up Data Review

As the analytical sequence is run / reviewed, the data is reviewed by both the bench chemist and the data reduction chemist using the following nine steps:

- 12.1.1 Check for any project-specific requirements.
- 12.1.2 Verify holding time.
- 12.1.3 Verify the BFB Tune check, CCV, LCS, (LCSD if required)
- 12.1.4 Verify that the Method Blank has no hits above the reporting limit (with the exceptions outlined in 8.8.1.1-8.8.1.5).
- 12.1.5 Verify sample results:
 - a) Verify the retention time.
 - b) Verify that correct amount of sample was analyzed.
 - c) Verify the automated peak integration.

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d) Verify that result concentrations are within linear range of Calibration Curve (upper 60% for dilutions.)

12.1.6 Initial and date raw data and/or logbook entry to indicate that the data is acceptable.

12.1.7 Apply appropriate data flags.

12.1.8 Describe unusual events on Data Review Checklist.

12.1.9 Verify results of the data validation report from Lumen, make corrections to the raw data as needed to remove errors identified by Lumen.

Notes:

- *A secondary review of the analytical runs is required when the bench chemist is not signed off on the analysis.*
- *Preparation and review of Laboratory Narratives are carried out as explained in EATL SOP #45.*

12.2 Technical Data Review

The Scientist or designated personnel performs a Technical Data Review on 100% reports if the write-up review analyst is not signed-off for the method. This review follows all the steps mentioned in the Analytical /Write-Up Data Review (Section 12.1).

12.3 Report Review

The Scientist or designated personnel performs a final data review on 100% reports prior to submitting to the client. The review consists of an overall verification of QC/data results and compliance to project specific requirements. Sample trends including Field and Trip Blanks are also verified.

* Unless the write-up review analyst is not signed-off in the method or if the project has specific data review requirements (i.e. DoD and client specific) the write-up review and report review are performed by the same individual.

12.4 QA Data Review

A thorough QA data review is performed by the QA Department on final data packages requesting 100% review. The QA Review entails verification that project and QC requirements are met. Failure to meet QC and/or project requirements results in a Corrective Action Request (CAR) and documentation. Dilution factors, analyte retention times, peak integration areas, concentration calculations, unit conversions, and Limit of Quantitation (reporting limits) are also checked. Field and Trip Blanks are checked and trends are observed.

13.0 INSTRUMENT MAINTENANCE

13.1 Instruments are monitored on a daily basis by the bench analyst for any potential failure. The analysis of blanks and control standards at the start of the day and as

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analysis continues helps to provide real-time feedback to the analyst on the condition of the instruments. Routine maintenance includes: mass spectrometers, sample introduction system and gas chromatograph.

13.1.1 The bench analyst will document any routine or major maintenance in the bound instrument maintenance logbook assigned to each instrument. The date of the maintenance, what work was performed, analyst initials and evidence of return to services are included.

13.2 Mass Spectrometers

- Periodic check of vacuum ion gauge (Increase in ion count indicates a potential leak)
- Daily (every 24 hours) tune check with BFB.
- Cleaning of ion source as needed.
- The oil level and quality is visually checked every month and at the time of source cleaning to ensure proper vacuum pump function, and oil is changed as needed.
- A sensitivity check must follow a routine maintenance to ensure that a standard representing the reporting limit or lower meets criteria.

13.3 Sample Introduction System

To ensure a clean sample introduction system the lines and trap are “steam-cleaned” by analyzing a humidified Zero Air system blank. This takes place every day following standard and laboratory control spike analysis. Humidified System blanks are also analyzed after saturation-level detections in samples.

13.4 Gas Chromatograph

Routine maintenance includes the following:

- 13.4.1 *As needed*, clip approx. 3 feet off the front end of the capillary column, and if necessary, the back end as well.
- 13.4.2 Replace the injection port liner as needed. The liner is replaced by removing the inlet cap using a wrench and releasing the liner from the inlet body using a pair of tweezers. Care should be taken not to get fingerprints on any inside surface.
- 13.4.3 Visually inspect the septum on the valve syringe injection port and replace as needed. Change the septum on the GC as needed. Always use Supelco Thermogreen septa and take care not to leave fingerprints on any inside heated surface. Wear a pair of white cotton gloves or use tweezers to

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handle the septa. Lower the oven temperature to 40°C. Remove the inlet cap with a wrench, remove the old septa with a pair of tweezers and insert the new septa.

13.4.4 The column is replaced when chromatography peak shape or resolution degrades. Similarly, if the column bleed profile rises with age then the column needs replacing. Use new black vespel/graphite ferrules each time and clip off approximately 1” of column after inserting it through the ferrule. This will remove any graphite particles that may have scrapped off into the column. Tighten the column nut and ferrule finger tight and one-quarter turn with a wrench. Tightening any more only crushes the ferrule and may damage the column.

13.4.4.1 If a new column of the same dimension and phase is being replaced a new MDL is not necessary.

14.0 DELIVERABLES

Data reporting packages are prepared as described in SOP #78 – Generation of Eurofins Air Toxics Data Deliverables, Electronic Conversion, and Archival.

15.0 REFERENCES

EPA Method TO-14A

Compendium of Methods for Determination of Toxic Organic Compounds in Air, EPA Methods, Second Edition, January 1997. *EPA/625/R-96/010b*

EPA Method TO-15

Compendium of Methods for Determination of Toxic Organic Compounds in Air, EPA Methods, Second Edition, January 1997. *EPA/625/R-96/010b*

SW-846 Method 8000B

Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Final Update III, Revision 1, December 1996.

Volatile Organic Analysis of Ambient Air in Canisters - Draft Method USEPA Contract Laboratory Program, Revision VCAA01.0, December 1991

Eurofins Air Toxics Laboratory Quality Assurance Manual
Definitions and Terms, Appendix A.

<http://www.air-dispersion.com/formulas.html>. Author Milton R. Beychok (accessed October 29, 2009).

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Appendix A
Reporting and QC Limits

Table A-1. Method TO-14A/TO-15 5&20 Compounds

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS* (%R)	Precision Limits (Max. RPD)
1,1,2,2-Tetrachloroethane	5.0	≤30%	70 - 130	70 - 130	± 25
1,1,2-Trichloroethane	5.0	≤30%	70 - 130	70 - 130	± 25
1,1-Dichloroethane	5.0	≤30%	70 - 130	70 - 130	± 25
1,1-Dichloroethene	5.0	≤30%	70 - 130	70 - 130	± 25
1,2,4-Trichlorobenzene	20	≤30%	70 - 130	70 - 130	± 25
1,2,4-Trimethylbenzene	5.0	≤30%	70 - 130	70 - 130	± 25
1,2-Dibromoethane (EDB)	5.0	≤30%	70 - 130	70 - 130	± 25
1,2-Dichlorobenzene	5.0	≤30%	70 - 130	70 - 130	± 25
1,2-Dichloroethane	5.0	≤30%	70 - 130	70 - 130	± 25
1,2-Dichloropropane	5.0	≤30%	70 - 130	70 - 130	± 25
1,3,5-Trimethylbenzene	5.0	≤30%	70 - 130	70 - 130	± 25
1,3-Dichlorobenzene	5.0	≤30%	70 - 130	70 - 130	± 25
1,4-Dichlorobenzene	5.0	≤30%	70 - 130	70 - 130	± 25
Benzene	5.0	≤30%	70 - 130	70 - 130	± 25
Bromomethane	20	≤30%	70 - 130	70 - 130	± 25
Carbon Tetrachloride	5.0	≤30%	70 - 130	70 - 130	± 25
Chlorobenzene	5.0	≤30%	70 - 130	70 - 130	± 25
Chloroethane	20	≤30%	70 - 130	70 - 130	± 25
Chloroform	5.0	≤30%	70 - 130	70 - 130	± 25
Chloromethane	20	≤30%	70 - 130	70 - 130	± 25
Chlorotoluene (Benzyl Chloride)	5.0	≤30%	70 - 130	70 - 130	± 25
cis-1,2-Dichloroethene	5.0	≤30%	70 - 130	70 - 130	± 25
cis-1,3-Dichloropropene	5.0	≤30%	70 - 130	70 - 130	± 25
Dichloromethane (Methylene Chloride)	20	≤30%	70 - 130	70 - 130	± 25
Ethylbenzene	5.0	≤30%	70 - 130	70 - 130	± 25
Freon 11 (Trichlorofluoromethane)	5.0	≤30%	70 - 130	70 - 130	± 25
Freon 113 (Trichlorotrifluoroethane)	5.0	≤30%	70 - 130	70 - 130	± 25
Freon 114	5.0	≤30%	70 - 130	70 - 130	± 25
Freon 12 (Dichlorodifluoromethane)	5.0	≤30%	70 - 130	70 - 130	± 25
Hexachlorobutadiene	20	≤30%	70 - 130	70 - 130	± 25
m,p-Xylene	5.0	≤30%	70 - 130	70 - 130	± 25
Methyl Chloroform (1,1,1-Trichloroethane)	5.0	≤30%	70 - 130	70 - 130	± 25
o-Xylene	5.0	≤30%	70 - 130	70 - 130	± 25
Styrene	5.0	≤30%	70 - 130	70 - 130	± 25
Tetrachloroethene	5.0	≤30%	70 - 130	70 - 130	± 25
Toluene	5.0	≤30%	70 - 130	70 - 130	± 25
trans-1,3-Dichloropropene	5.0	≤30%	70 - 130	70 - 130	± 25
Trichloroethene	5.0	≤30%	70 - 130	70 - 130	± 25
Vinyl Chloride	5.0	≤30%	70 - 130	70 - 130	± 25
1,3-Butadiene	5.0	≤30%	70 - 130	70 - 130	± 25
1,4-Dioxane	20	≤30%	70 - 130	70 - 130	± 25

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Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS* (%R)	Precision Limits (Max. RPD)
2-Butanone (Methyl Ethyl Ketone)	20	≤30%	70 - 130	70 - 130	± 25
2-Hexanone	20	≤30%	70 - 130	70 - 130	± 25
4-Ethyltoluene	5.0	≤30%	70 - 130	70 - 130	± 25
4-Methyl-2-Pentanone (MIBK)	5.0	≤30%	70 - 130	70 - 130	± 25
Acetone	20	≤30%	70 - 130	70 - 130	± 25
Bromodichloromethane	5.0	≤30%	70 - 130	70 - 130	± 25
Bromoform	5.0	≤30%	70 - 130	70 - 130	± 25
Carbon Disulfide	20	≤30%	70 - 130	70 - 130	± 25
Cyclohexane	5.0	≤30%	70 - 130	70 - 130	± 25
Dibromochloromethane	5.0	≤30%	70 - 130	70 - 130	± 25
Ethanol	20	≤30%	70 - 130	70 - 130	± 25
Heptane	5.0	≤30%	70 - 130	70 - 130	± 25
Hexane	5.0	≤30%	70 - 130	70 - 130	± 25
Isopropanol	20	≤30%	70 - 130	70 - 130	± 25
Methyl t-Butyl Ether (MTBE)	5.0	≤30%	70 - 130	70 - 130	± 25
Tetrahydrofuran	5.0	≤30%	70 - 130	70 - 130	± 25
trans-1,2-Dichloroethene	5.0	≤30%	70 - 130	70 - 130	± 25
2,2,4-Trimethylpentane	5.0	≤30%	70 - 130	70 - 130	± 25
Cumene	5.0	≤30%	70 - 130	70 - 130	± 25
Propylbenzene	5.0	≤30%	70 - 130	70 - 130	± 25
3-Chloroprene	20	≤30%	70 - 130	70 - 130	± 25

* In-house generated or DoD specified control limits may be used for LCS per project requirement.

Table A-1b. Method TO-14A/TO-15 5&20 Example Non-Standard Compounds

Analyte	RL/LOQ (ppbv)	QA Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
Butane	20	≤40	60-140	60 – 140	± 25
Ethyl tert-Butyl Ether	20	≤40	60-140	60 – 140	± 25
Isopentane	20	≤40	60-140	60 – 140	± 25
Isopropyl Ether	20	≤40	60-140	60 – 140	± 25
Methylcyclohexane	20	≤40	60-140	60 – 140	± 25
Propylene	20	≤40	60-140	60 – 140	± 25
tert-Amyl Methyl Ether	20	≤40	60-140	60 – 140	± 25
Vinyl Acetate	20	≤40	60-140	60 – 140	± 25
tert-Butyl Alcohol	20	≤40	60-140	60 – 140	± 25
Naphthalene	20	≤40	60-140	60 – 140	± 25
TPH (Gasoline)	200	1- Point Calibration	NA	ICV only: 60 – 140	± 25
NMOC (Hexane/Heptane)	100	1- Point Calibration	NA	NA	± 25

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Table A-2. Internal Standards

Analyte	Recovery Limits (%R)
Bromochloromethane	60 – 140
1,4-Difluorobenzene	60 – 140
Chlorobenzene-d ₅	60 – 140

Table A-3. Surrogates

Analyte	Recovery Limits (%R)*
1,2-Dichloroethane-d ₄	70 – 130
Toluene-d ₈	70 – 130
4-Bromofluorobenzene	70 – 130

* In-house generated control limits may be used per project requirement.

Table A-4. Summary of Calibration and QC Procedures for Methods TO-14A/TO-15

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours.	TO-15 ion abundance criteria	Correct problem then repeat tune.
Minimum 5-Point Calibration (ICAL)	Prior to sample analysis.	% RSD ≤ 30 with two compounds allowed out to ≤ 40% RSD.	Correct problem then repeat Initial Calibration Curve.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS)	After each initial calibration curve, and daily, prior to sample analysis.	Recoveries for 85% of "Standard" compounds must be 70-130%. No recovery may be <50%. ICV evaluated on a full list basis at time of calibration. * If specified by the project, in-house generated or DoD specified control limits may be used for the LCS.	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS) for Non-standard Compounds	Per client request or specific project requirements only.	Recoveries of compounds must be 60-140%. No recovery may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	At the start of each analytical clock after the tune check.	70-130%.	Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than two compounds from the standard list recover outside of 70-130% or > 10% of VOCs if short list is used (20 compounds or less), corrective action will be taken. If any compound exceeds 60-140%, samples are not analyzed unless data meets project needs. Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present.	Results less than the laboratory reporting limit (Tables A-1).	Inspect the system and re-analyze the blank. B-flag data for common contaminants
Surrogates	As each standard, blank, and sample is being loaded.	70 - 130%. * If specified by the project in-house generated control limits may be used.	For blanks: inspect the system and reanalyze the blank. For samples: re-analyze the sample unless obvious matrix interference is documented. If the %Rs are within limits in the re-analysis, report the second analysis. If %Rs are out-of-limits a second time, report data from first analysis and narrate.
Laboratory Control Spike Duplicate (LCSD)	One per analytical batch.	RPD \leq 25%.	Narrate exceedances. If more than 5% of compound list outside criteria or if compound is >40%RPD, investigate the cause and perform maintenance as required. If instrument maintenance is required, calibrate as needed
Internal Standard (IS)	As each standard, blank, and sample is being loaded.	Retention time (RT) for blanks and samples must be within ± 0.33 min of the RT in the CCV and within $\pm 40\%$ of the area counts of the daily CCV internal standards.	For blanks: inspect the system and reanalyze the blank. For samples: re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.

Table A-5. Method TO14A/TO-15 Suggested Primary Quantitation Ions

Analyte	Suggested Primary Quantitation Ion
1,1,2,2-Tetrachloroethane	83
1,1,2-Trichloroethane	97
1,1-Dichloroethane	63
1,1-Dichloroethene	61
1,2,4-Trichlorobenzene	180
1,2,4-Trimethylbenzene	105
1,2-Dibromoethane (EDB)	107
1,2-Dichlorobenzene	146
1,2-Dichloroethane	62
1,2-Dichloropropane	63
1,3,5-Trimethylbenzene	105
1,3-Dichlorobenzene	146
1,4-Dichlorobenzene	146
Benzene	78
Bromomethane	94
Carbon Tetrachloride	119
Chlorobenzene	112
Chloroethane	64
Chloroform	83
Chloromethane	50
Alpha-Chlorotoluene (Benzyl Chloride)	91
cis-1,2-Dichloroethene	61
cis-1,3-Dichloropropene	75
Dichloromethane (Methylene Chloride)	49
Ethylbenzene	106
Freon 11 (Trichlorofluoromethane)	101
Freon 113 (Trichlorotrifluoroethane)	151
Freon 114	135
Freon 12 (Dichlorodifluoromethane)	85
Hexachlorobutadiene	225
m,p-Xylene	106
Methyl Chloroform (1,1,1-Trichloroethane)	97
o-Xylene	106
Styrene	104
Tetrachloroethene	166
Toluene	91
trans-1,3-Dichloropropene	75
Trichloroethene	95
Vinyl Chloride	62
1,3-Butadiene	54
1,4-Dioxane	88
2-Butanone (Methyl Ethyl Ketone)	72
2-Hexanone	58
4-Ethyltoluene	105

Analyte	Suggested Primary Quantitation Ion
Acrolein	56
Bromodichloromethane	83
Bromoform	173
Carbon Disulfide	76
Cyclohexane	84
Dibromochloromethane	129
Ethanol	45
Heptane	100
Hexane	57
Isopropanol (2-Propanol)	45
Methyl t-Butyl Ether (MTBE)	73
Propylene	41
Tert-Butyl-Alcohol	59
Tetrahydrofuran	42
trans-1,2-Dichloroethene	96
Cumene	105
Propylbenzene	91
3-Chloropropene	76
2,2,4-Trimethylpentane	57
Naphthalene	128
4-Methyl-2-Pentanone (MIBK)	58
Acetone	58
Internal Standards	
Bromochloromethane	130
1,4-Difluorobenzene	114
Chlorobenzene-d ₅	117
Surrogates	
1,2-Dichloroethane-d ₄	65
Toluene-d ₈	98
4-Bromofluorobenzene	174

Appendix B

BFB Tune Criteria

Mass	ION ABUNDANCE CRITERIA
50	8.0 to 40.0% of mass 95
75	30.0 to 66.0% of mass 95
95	Base Peak, 100% Relative Abundance
96	5.0 to 9.0% of mass 95
173	< 2.0% of mass 174
174	50.0 to 120.0% of mass 95
175	4.0 to 9.0% of mass 174
176	93.0% to 101.0% of mass 174
177	5.0 to 9.0% of mass 176

Appendix C

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Title: TO-14A/TO-15 QUAD
 IS and Associated Target compnds.and Surr.
 Instructcn #: 11.20

Release Date: 02/11/2010
 Revision Date: 4/9/2007
 Revision#: 1
 Page#: 1 of 1

Modified EPA Methods TO-14A/TO-15
 Internal Standard and Associated Target Compounds and Surrogates

Bromochloromethane	1,4-Difluorobenzene	Chlorobenzene-d5
Target Compounds:	Target Compounds:	Target Compounds:
Freon 12	Benzene	trans-1,3-Dichloropropene
Freon 114	1,2-Dichloroethane	1,1,2-Trichloroethane
Chloromethane	Heptane	Tetrachloroethene
Vinyl Chloride	Trichloroethene	2-Hexanone
1,3-Butadiene	1,2-Dichloropropane	Dibromochloromethane
Bromomethane	1,4-Dioxane	1,2-Dibromoethane (EDB)
Chloroethane	Bromodichloromethane	Chlorobenzene
Freon 11	cis-1,3-Dichloropropene	Ethyl Benzene
Ethanol	4-Methyl-2-pentanone	m,p-Xylene
Freon 113	Toluene	o-Xylene
1,1-Dichloroethene	Surrogates:	Styrene
Acetone	Toluene-d8	Bromoform
2-Propanol		Cumene
Carbon Disulfide		1,1,2,2-Tetrachloroethane
3-Chloropropene		Propylbenzene
Methylene Chloride		4-Ethyltoluene
Methyl tert-butyl ether		1,3,5-Trimethylbenzene
trans-1,2-Dichloroethene		1,2,4-Trimethylbenzene
Hexane		1,3-Dichlorobenzene
1,1-Dichloroethane		1,4-Dichlorobenzene
2-Butanone (Methyl Ethyl Ketone)		alpha-Chlorotoluene
cis-1,2-Dichloroethene		1,2-Dichlorobenzene
Tetrahydrofuran		1,2,4-Trichlorobenzene
Chloroform		Hexachlorobutadiene
1,1,1-Trichloroethane		Surrogates:
Cyclohexane		Bromofluorobenzene
Carbon Tetrachloride		
2,2,4-Trimethylpentane		
Surrogates:		
1,2-Dichloroethane-d4		

SOP Revision History

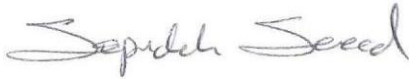
Revision Date	Revision #	Changes	Reviewer(s)
06/16/15	10	Added reference to work instructions WI91 in section 2.1 Added section 10.10.6 which references new work instructions for C2 Interface 5&20 analysis.	Mike Skidmore
2/23/16	11	Updated Table A-5 now Table A-4 ICV criteria, Section 8.5.5 LCSD criteria updated, Section 8.7.1 defined surrogates are same concentration as internal standards, Table A-1b updated to include all TO-15 specials. Table A-4 (ICAL levels summary) removed- not needed Added dilution factor calculation in section 9.0 Removed section 8.3.5 referencing the use of control limits for ICV. Clarified text in QC table A-4 to specify statistical control limits and/or DoD specified apply only to LCS. Added "DoD specified" limits as an option along with in-house statistically generated limits throughout SOP.	Samantha Black Heidi Hayes
6/21/16	12	Updated Appendix A reporting limits for 1,4-Dioxane, 2-Hexanone, 3-Chloroprene, Acetone, Bromomethane, Carbon Disulfide, Ethanol, Methylene Chloride and THF (RTC #079).	Samantha Black
11/30/16	13	Added section 8.4.1.1: 10% exceedance criteria defined for CCV. Table A-4: added 10% exceedance criteria. Updated Section 8.2.3 to clarify setting Retention Time Windows using the ICAL and when to use the CCV.	Ed Jakab Melanie Levesque
02/17/17	14	Section 5.3: Red-tagging canisters moved to WI91 C2 interface (5&20).	Ed Jakab

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3/23/18	15	Section 7.2.2: Entech NT7400 Precision replaced Entech NT4600 Dynamic Diluter.	Gretchen Hehir
4/17/18		Updated Section 8.8.1 for current procedure regarding carryover during sample analysis. Section 10.3.3 updated to reference WI39 MDL Procedure. Added section 8.5.6: LCSD % R acceptance criterion for risk drivers Section 8.2.1 – removed no longer relevant exceptions to QC criterion; Section 8.0 – Added introduction statement referencing DoD/project specific QC parameters in PRTs; Section 8.2.8 – Removed verbiage and replaced with verbiage relevant to subsequent sections; Section 10.2 – Added verbiage to indicate additional QC samples may be required in sequence; Sections 10.3.1 and 10.3.2 – Updated MDL verbiage; Table A-1: Removed ** and *** asterisks and notes.	Ed Jakab Melanie Levesque
05/31/19	16	Section 10.10.4 – Updated calibration ranges. Section 6.1 – Removed NIST library NBS54.1 and added NIST11.1	Mike Skidmore Heidi Hayes
06/29/20	16.1	No updates needed.	Gracianne Dela Cruz
10/06/20	17	Section 6.1 and 7.1. –Updated list of vendors. Section 7.3 – Removed references of methanolic BFB use and procedure of BFB direct injection into the GC injection port per RTC-119. Section 7.4 Internal Standard/Surrogate (IS/S) Mix – Updated section numbering to 7.3. Updated list of vendors. Updated IS/S concentration to 10ppmv. Added procedure of using directly filled summa canisters for IS/S as working standards and the associated	Steven Nguyen

10/21/20		expiration dates. Removed use of neat materials to make IS/S – no longer relevant. Section 7.4 – Updated to BFB tune check standard and added applicable information per RTC-119. Section 8.1.1.-Updated BFB tuning procedures per RTC-119. Sections 7.2.1 and 7.2.3 – Update water addition to a 100 uL aliquot to humidify working standards. (RTC-122)	
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Document Owner/Laboratory Director:



Date: 10/21/2020

Technical Director:



Date: 10/21/2020

Quality Assurance:



Date: 10/21/20

**Eurofins Air Toxics, LLC
STANDARD OPERATING PROCEDURE**

**ANALYSIS OF VOLATILE ORGANIC COMPOUNDS IN SUMMATM
POLISHED CANISTERS BY GC/MS LOW LEVEL**

MODIFIED EPA METHODS TO-14A/TO-15

SOP # 83

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1.0 SCOPE AND APPLICATION

The procedures in this SOP describe the use of Modified EPA Methods TO-14A and TO-15 to determine the concentration of volatile organic compounds in ambient air using an evacuated specially treated stainless steel canister. Details of the analytical procedures and the required QC protocols are provided. A list of target compounds can be found in *Appendix A*.

2.0 METHOD SUMMARY

2.1 Description

EPA Methods TO-14A and TO-15 describe techniques for the analysis of airborne VOCs collected as whole air samples in evacuated, specially treated stainless steel canisters. An aliquot of up to 400 mL of air is withdrawn from the canister using a mass flow controller. This volume is loaded onto a hydrophobic multibed sorbent trap to remove water and carbon dioxide and to concentrate the vapor sample. The focused sample is then flash heated to sweep adsorbed VOCs onto a GC/MS for separation and detection. Compounds are detected using a mass spectrometer operating in the full scan mode.

2.2 Deviations

EPA Methods TO-14A and TO-15 were written for ambient air applications targeting VOC concentrations at and above 0.5 ppbv. To provide VOC measurements below 0.5 ppbv, several modifications were implemented to accommodate a 5-fold reduction in reporting limits.

Modifications to EPA Methods TO-14A and TO-15 used to carry out the analyses of air samples are summarized in Tables 1 and 2.

Table 1. Summary of Method TO-14A Modifications

Requirement	TO-14A	EATL Modifications
Sample Drying System	Nafion Dryer	Multibed hydrophobic sorbent
Blank acceptance criteria	< 0.2 ppbv	< RL
BFB ion abundance criteria	Ion abundance criteria listed in Table 4 of TO-14A	Follow abundance criteria listed in TO-15.
BFB absolute abundance criteria	Within 10% when comparing to the previous daily	CCV internal standard area counts are compared to ICAL, Corrective action when recovery is less than 60%.

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Requirement	TO-14A	EATL Modifications
	BFB.	
Blanks and standards	Zero Air	UHP Nitrogen provides a higher purity gas matrix than zero air for trace level measurements.
Initial Calibration	≤ 30% RSD for listed 39 VOCs	≤ 30% RSD with 4 compounds allowed out to ≤ 40%.

Table 2. Summary of Method TO-15 Modifications

Requirement	TO-15	EATL Modifications
Initial Calibration	≤ 30% RSD with 2 compounds allowed out to < 40% RSD.	≤ 30% RSD with 4 compounds allowed out to ≤ 40%.
Blanks and standards	Zero Air	UHP Nitrogen provides a higher purity gas matrix than zero air.

3.0 HEALTH AND SAFETY

- 3.1 Normal laboratory safety precautions must be used when handling samples, preparing standards from neat materials, and analyzing samples. Appropriate eye wear, gloves, and lab coat must be worn when handling any chemical used in this method. All manipulation of standards, solvents, and solutions should be done with the utmost care in the hood. SDS for each chemical should be consulted for specific dangers and precautions.
- 3.2 Personnel must handle high pressure cylinders safely. This includes transport of cylinders fully secured on a cart. During storage, the cylinders must be secured at all times with a chain. When installing a pressure regulator, stand to the side of the cylinder.
- 3.3 Care must also be taken when handling syringes to ensure that a needle stick does not occur. *All personnel installing or performing maintenance on a capillary column must wear eye protection.*
- 3.4 For information regarding pollution prevention and waste disposal, see Eurofins Air Toxics SOP #24: Storage and Disposal of Hazardous Wastes.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

4.1 Sample Containers

An air sample is collected in an evacuated stainless steel SummaTM / canister or Tedlar bag. Tedlar bags are not included as acceptable media in either Compendium Method TO-14A or TO-15, but are accepted for analysis per client request. Upon receipt, the canisters will be approximately in the range of 2.5 – 10” Hg vacuum.

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4.2 Sample Handling

Prior to analysis, the canister is pressurized in accordance with Eurofins Air Toxics SOP #60 (Canister Pressurization) and/or per project specific requirements. Pressurization may not be required depending on project requirements.

Samples pressurized with Helium cannot be analyzed by low level analysis. The flow controllers used in the laboratory are calibrated for Nitrogen only. Helium pressurized samples typically have a higher flow rate than Nitrogen pressurized samples. Please see SOP 6 or 91 for analysis of Helium pressurized samples.

4.3 Sample Storage

Samples are stored in the sample cage in the main laboratory. Analysis must occur within 30 days for canisters. Some projects may require a 14 day hold for canisters. The project profile must be reviewed prior to analysis to verify holding time requirements.

- 4.4 Holding time in the case of Tedlar bags is limited to a maximum of 3 days as losses of VOCs are observed specifically for compounds with low vapor pressure. Holding time may be extended to a maximum of 30 days by transferring the sample from a Tedlar bag to a clean, evacuated Summa™ canister prior to the expiration of the 3 day hold. The transfer must be documented using the Tedlar bag transfer form and documented in the case narrative as per Eurofins Air Toxics SOP #69.

5.0 ***INTERFERENCES AND POTENTIAL PROBLEMS***

- 5.1 Interferences to this method generally include high concentrations of water. Very high levels of moisture in the samples may cause low internal standard and surrogate responses which will interfere with accurate compound quantification. In these situations, dry lab blanks may be required between samples to maintain internal standard recoveries. In extreme cases, sample dilution may be required.
- 5.2 When a sample has high levels of acidic gases such as HCl and SO₂ and/or high levels of non-target compounds, the analyst may have to dilute the sample more than the target compound level requires. This ensures that internal standard recoveries meet QC requirements and/or that the system is not contaminated from carryover or damaged by high levels of acidic gases.
- 5.3 See Eurofins Air Toxics WI38_83 C2 interface and WI38_83 PE interface (MSD-E) work instructions for examples when to red tag canisters due to high level concentrations.

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- 5.4 Tedlar bags are not appropriate sample collection media for low vapor pressure compounds such as naphthalene due to severe adsorption and subsequent losses on the Tedlar surface. A sample discrepancy report (SDR) must be submitted when naphthalene measurements are requested from a Tedlar bag. All naphthalene results are flagged as estimated values.

6.0 ***EQUIPMENT/APPARATUS***

6.1 List of Equipment

- TO-15 Air Concentrator Systems (Documentation on system designs and manufacturer's information is maintained at each unit and is maintained on the secure QA drive).
- Column: RTX-624 60m X 0.32mm I.D. or 30m X 0.25mm I.D. Capillary Column (Restek) or equivalent column
- Gas Chromatograph: Agilent 7890 or equivalent equipped with Electronic Pressure Control and a split/splitless injection port, and a cryo valve
- Mass Spectrometer: Agilent 5975, 5977 or equivalent
- Agilent Chemstation Software for Data Acquisition
- Thru-Put Target Software (UNIX Operating System) for Data Analysis
- NIST11.1 or NIST08.1 Library Search Software
- Certified NIST Traceable VOC cylinder blends – Praxair, Linde Spectra Environmental Gases, etc.
- High Purity Neat Chemicals
- Tedlar Bags (various sizes) SKC
- Heating Tapes- Various Lengths (Cole Palmer)
- Power Controllers (Cole Palmer)
- Liquid Nitrogen certified VOC free by TO-15 analysis
- Ultra High Purity Helium (Local Supplier)
- Commercially purchased dilution system to blend working standards from stock cylinder standards, Entech NT4700.
- Laboratory Designed Canister Receiving Station equipped with high-resolution vacuum/pressure gauge and diluent gas (N₂, He, or Zero air) inlet

6.2 Operating Parameters for GC/MS

Since analytical equipment varies in configuration and as a function of intended specific target analytes, operating parameters are optimized on an instrument-by-instrument basis. Instrument specific operating parameters are recorded in the respective Instrument Maintenance Logbook and in the QA-controlled Work Instructions at each work station.

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Note: Only trained laboratory staff are authorized to vary the operating parameters under special circumstances. Any time a change is made, it is documented in the instrument maintenance logbook.

7.0 STANDARD PREPARATION

The formula used to calculate the volume required for the preparation of primary and second source standards for gas blends is the following:

$$C1*V1 = C2*V2,$$

Where, C1 is the concentration of the gas blend, V1 is the volume of standard to be used in the preparation, C2 is the desired concentration for the resulting standard and V2 is the final volume (at pressure*) of the newly blended standard.

*At 15 psi the final volume for a 6L canister is 12L and for a 1L canister it is 2L (volume reflects a twofold dilution as a function of pressurization).

7.1 Stock Standards

7.1.1 The TO-15 analytes are purchased from Praxair, Linde Spectra Environmental Gases, or other available vendors at a concentration of 1-5 ppmv in a high pressure cylinder blend. The standard expires according to the manufacturer's expiration date on the certificate of analysis.

7.1.2 Analytes not present in the commercially available blends are purchased in neat form from a commercial vendor. These compounds are then blended into the gas phase as described in SOP #33. Once the neat compound exceeds the manufacturer's expiration date, these standards must be purity-checked by the laboratory annually.

7.2 Working Standards

7.2.1 Starting with a clean evacuated canister, a volume of 100 microliters of purged deionized water is injected to humidify the standard prior to preparation.

7.2.2 Entech NT4700 Precision Diluter

The Entech NT4700 Precision Diluter is used to prepare primary and secondary standards for VOC analysis. The diluter prepares dilution of stock standards by monitoring small pressure changes digitally. There are a total of four positions on the diluter that can accommodate high pressure standard cylinders. There is an additional inlet to allow for secondary

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dilutions of working standards. See WI-4700 for the complete work instructions.

- 7.2.3 Working standards prepared from source standards originating from neat or liquid mixes are prepared by injecting appropriate amounts of standard into an evacuated canister that contains 100 μ L of water. Details on how to prepare these working standards are provided in SOP #33. These working standards typically contain 'non-routine' compounds which are blended in canisters separate from the routine TO-15 VOC working calibration standard.
- 7.2.4 All working standards are pressurized to 15 psi with Nitrogen. Concentrations of working standard canisters are typically prepared at 0.05 ppbv (if needed), 2.0 ppbv, and 50 ppbv. (Some compounds may be blended at concentrations 10x lower, e.g. Naphthalene).
- 7.2.5 Working standards can be re-pressurized with Nitrogen when pressure becomes near ambient. This re-pressurization step is recorded in the standard preparation logbook (See SOP #33 for details.) The concentration of the working standard is then adjusted to reflect the addition of Nitrogen.

Working standards may be used for up to 3 months from the date of preparation. The TO-15 working standard may not be used past the expiration date of the NIST-traceable high pressure cylinder. *Note: Tedlar bags are used for the static dilution medium due to their inherent inertness to polar analytes vs. glass dilution jars. Standards made using a Tedlar bag should not, however, be stored in the Tedlar bags beyond one day. Fresh calibration standards are prepared and then transferred to SummaTM canisters for storage. The standards prepared from neat materials are stable in SummaTM canisters for 6 months.*

7.3 BFB Check Mix

A 10ppmv NIST-traceable cylinder containing BFB is used to make a diluted working standard at 1.25ppmv.

7.4 Internal Standard/Surrogate (IS/S) Mix

The IS/S blend is an internal reference material purchased from Praxair, Linde Spectra Environmental Gases, or another available vendor. The nominal requested concentration is 10 ppmv and expires based on the manufacturer's date on the certificate of analysis. A working standard is prepared by taking a known aliquot of the standard and diluting it to achieve a final concentration of 1.25ppmv.

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8.0 ***CALIBRATION AND QUALITY CONTROL PROCEDURES***

The following sections outline the laboratory's routine calibration and quality control procedures. Specific programs such as DoD QSM 5.1 and projects may require additional QC samples and/or tighter QC criteria. These requirements are found in the Project Requirement Table (PRT) generated during the project set-up/QAPP review steps as outlined in SOP#1. PRTs are attached to the relevant profile in ATLAS and stored electronically on the network (O:\Project Requirement Tables\).

8.1 Tuning Criteria

8.1.1 At the start of every 24 hours, a tune check with 4-Bromofluorobenzene (BFB) is performed by loading IS/S from the 1.25ppmv working standard onto the TO-15 air preconcentrator 1ml IS/S loop. A 50ml flow controller load from a laboratory blank is utilized to establish flow. See work instruction WI38_83 C2 interface for further details on flow controller loads. The final on column BFB concentration is 8.9ng.

8.1.2 The relative abundances of selected ions are tabulated and reported as outlined in *Appendix B*. Analysis cannot proceed unless all criteria of the tune check are met in accordance with EATL SOP #53. The acceptance criteria are based on TO-15 which are wider than tuning criteria outlined in TO-14A. The BFB standard must be re-analyzed when the EM voltage is changed or when the instrument has been re-tuned.

8.2 Initial Calibration Procedures

Initial Calibration of the GC/MS is achieved via the internal standard technique. The concentrations used for standard analysis typically range from 0.1 to 40 ppbv. Other levels may be added to the calibration per specific client/project request.

8.2.1 Standard Compound criteria

Standard (routine) TO-14A/TO-15 compounds are listed in Tables A-1 and A-1b. Initial Calibration is performed using a minimum of five levels for standard TO14A/TO-15 compounds. The low-level standard must be less than or equal to the reporting limit (Limit of Quantitation) and must be verified on a quarterly basis.

The percent relative standard deviations (%RSD) for all standard compounds must be $\leq 30\%$, with four exceptions not to exceed $\leq 40\%$.

Non-Standard Compound criteria

Compounds that are not included in Table A-1 are defined as non-standard compounds. Examples of Non-Standard TO-14A/TO-15 compounds are listed in Table A-1b. A one, three or five point calibration may be performed for non-standard compounds with documented client

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approval. The requirements for TPH are described in SOP #111. The lowest level standard must be less than or equal to the reporting limit (Limit of Quantitation). In general, LOQ (Limit of Quantitation) evaluations are not maintained for non-standard compounds which are not part of DoD accreditation.

8.2.2.1 Initial calibration criterion for non-standard compounds is $\leq 40\%$ RSD subject to verification of client requirements in the Project Requirement Table. It is the responsibility of all analytical personnel to verify agreed upon Initial Calibration requirements prior to sample analysis as more stringent criteria than $\leq 40\%$ RSD may be required. If linearity requirements are not met a new Initial Calibration Curve is performed.

8.2.2.2 Scientific experience and knowledge must be applied to the performance evaluation of specially requested compounds. It may be that a non-standard compound does not perform well due to its vapor pressure or instability. In these cases, the clients are notified of this non-conformance to TO-15 performance requirements, and data qualifiers may be required.

8.2.2 After an Initial Calibration has been evaluated and meets laboratory criteria, the midpoint for standard compounds is copied by adding an extension onto the file identifier, and then it is re-quantified as a Continuing Calibration Verification. All calibration points are then re-quantified and the Internal Standard area counts and Retention Time Windows for each file are compared to that of the midpoint. Internal Standards in each calibration point compared in this manner must meet laboratory criteria (area count $\pm 40\%$ and Retention Time ± 0.33 minutes compared to the CCV). If %D criterion for the CCV (see Section 8.4) is met, analysis of the Initial Calibration Verification Standard may proceed. On days when an Initial Calibration is not performed, the Internal Standard area counts and Retention Time Windows in all subsequent samples and QC must meet laboratory criteria (area count $\pm 40\%$ and Retention Time ± 0.33 minutes compared to the CCV analyzed at the beginning of the analytical sequence).

8.2.3 The multipoint calibration is constructed by loading varying amounts of the combined calibration blend (Section 7.2) onto the canister interface. The maximum load volume for the air interface system (e.g. 250 mL) defines the dilution factor. Lesser volumes of a standard calibration loaded onto the concentrator results in a proportionally lower on-column concentration. In this manner, several working standard concentrations are used to generate a multi-level calibration curve.

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- 8.2.4 The average relative response factor (RRF) from the Initial Calibration Curve is used to quantitate results.
- 8.2.5 Initial calibration curves are generated in accordance to appropriate laboratory practices:
- 8.2.5.1 The lowest or highest calibration levels of an analyte may be dropped from the curve to achieve linearity. This will affect the analyte's reporting limit and/or calibration range. The minimum number of calibration levels must still be used for the curve.
 - 8.2.5.2 An additional mid-range calibration level may be added to achieve linearity.
 - 8.2.5.3 Alternate calibration levels may be used to meet client requirements.
 - 8.2.5.4 It is not acceptable to drop a calibration level that is found somewhere in the middle of the curve to achieve linearity (i.e. dropping the 50ppbv or the 25ppbv calibration level). The calibration level that is in question may be re-analyzed and re-quantated into the curve.
- 8.2.6 All Initial Calibrations needing re-analysis of a calibration level require explanation in the Initial Calibration Case Narrative Template. Only one calibration level is allowed for re-analysis per Initial Calibration Curve due to anomalous unacceptable linearity for compound(s). A bad load or unopened can does not count towards a re-analysis. If more than one standard was used for curving an instrument (i.e. 2 or more), and it is obvious that the RF is not linear relative to the other points of the curve, then all points ran from that standards must be re-analyzed. The reason for the reanalysis must be narrated and included with the ICAL raw data.
- 8.2.7 The reporting limit (Limit of Quantitation – LOQ) must be verified quarterly on each instrument that performs the methods the lab is accredited for by DoD-ELAP (Refer to DoD scope at O:\QA\Certifications). The LOQ is verified by evaluating the point of the initial calibration that corresponds to the LOQ. The concentration recovered is used to calculate the precision and bias of each compound. A minimum of three points is required to perform the calculations. If there are insufficient points, a primary source standard is analyzed at the LOQ. Precision and bias is determined by calculating the % relative standard

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deviation (% RSD) and the % bias of the mean concentration recovered. The acceptance criterion for the LOQ verification is $\leq 30\%$ RSD and $\leq 50\%$ bias for each compound. If more than 10% of the compound list exceeds the acceptance criteria, the instrument and standard used will be evaluated to determine the source of the error. Quarterly LOQ verifications are evaluated by the QA Department and the values are maintained on a secure network drive.

8.2.8 All current Initial Calibrations and Method Detection Limit (MDL) studies are kept in a folder near the instrument.

8.2.9 The indicated flow on the instrument must be measured and its units must be calibrated with a NIST flow meter to provide a true indication of the actual flow through the unit before each time an Initial Calibration is performed and/or on a quarterly basis, whichever is more frequent.

8.3 Initial Calibration Verification (ICV)

8.3.1 Standard Compound criteria

An independently prepared (i.e., same vendor, different lot number, or second vendor) standard containing all target compounds is analyzed after each Initial Calibration Curve, to verify that the standards are correct and the calibration is accurate. The acceptance criterion for the ICV recoveries are as follows: For the compounds listed in Table A-1, recoveries for 85% of the compounds must meet the listed acceptance criteria with no recoveries $< 50\%$. For the compounds listed in Table A-1, recoveries for 90% of the compounds must be $\pm 30\%$. For the compounds listed in Table A-1b recoveries for 80% of the compounds must be $\pm 40\%$.

8.3.2 Recoveries of any compound that is found to exceed these criteria, but are within 50-150%, are narrated on the ICAL Narrative sheet that is attached to the ICAL packet. However, exceeding the acceptance criterion of 150% recovery permits for a reanalysis of an ICV.

8.3.3 Recovery of any compound in the ICV that is $< 50\%$ of the expected value will result in standard re-preparation, system maintenance if needed and re-calibration, or sample analysis on a different instrument. Recovery of any compound that is $\geq 150\%$ of expected value, analysis may continue with manager approval after evaluation of whether the data meets client project needs. However, the system and/or standard preparation should be evaluated. If the problem is determined to be systematic (i.e. occurs on more than three consecutive days), corrective action outlined above should be conducted to resolve the issue.

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8.3.4 Non-Standard Compound criteria

An Initial Calibration Verification will be performed for non-standard compounds only when prior arrangements with the client have been made and are documented in the Project Requirement Table. Initial calibration verification criterion for these analytes is 60 – 140 % Recovery and is subject to verification of client requirements in the Project Requirement Table. It is the responsibility of all analytical personnel to verify the agreed upon ICV requirements prior to sample analysis as more stringent criteria than 60 – 140 % may be required.

8.3.5 See SOP #111 for requirements for TPH.

8.4 Continuing Calibration Verification (CCV)

8.4.1 A Continuing Calibration Verification (CCV) is performed at the start of each day after analysis of the BFB Tune Check. This is an analysis of the primary source at a concentration between the low point and the midpoint of the initial calibration.

8.4.2 The acceptance criteria for the percent Difference (%D) between the daily CCV response and average response from the Calibration Curve is as follows:

8.4.2.1 The acceptance criteria for the percent Difference (%D) between the daily CCV response and average response from the Calibration Curve is as follows: All standard CCV compounds must be $\leq 30\%$ D. Any compounds exceeding this criterion will be flagged and associated data likewise flagged and narrated. If more than four compounds from the standard list recover outside of 70-130%, corrective action will be taken. Corrective action may include instrument maintenance, re-calibration, and/or re-preparation of the calibration standards (See 8.4.4). If any compound recovery exceeds 60-140%, samples are not analyzed unless data meets project needs. The QA Manager or Lab Manager may approve exceedance of a compound under special circumstances after reviewing the impact to the data quality. Regardless, associated data will be flagged and narrated.

8.4.2.2 If a list of ≤ 40 target compounds is requested, no more than 10% of the compounds may be outside 70-130% criteria. For example, if a client is requesting 6 compounds then 0 compounds are

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allowed out; or if 15 compounds are requested then only 1 compound may be outside acceptance criteria.

8.4.3 Non-Standard Compound criteria

Compounds that are not included in Tables A-1 are defined as non-standard compounds. Examples of Non-Standard TO-14A/TO-15 compounds are listed in Table A-1b. CCV criterion for these analytes is $\leq 40\%$ D subject to verification of client requirements in the Project Requirement Table. It is the responsibility of all analytical personnel to verify agreed upon CCV requirements prior to sample analysis as more stringent criteria than $\leq 40\%$ D may be required.

8.4.4 If the CCV fails to meet the performance criteria, the CCV is re-analyzed and/or another standard is analyzed. If the CCV fails again, system maintenance should be performed and the test repeated. If the system still fails the calibration check, a new Calibration Curve is performed. If the CCV passes following maintenance to the instrument then the LCS must also pass before samples can be analyzed.

8.4.5 Certain projects have different CCV acceptance criteria (e.g., $\leq 25\%$ D for a set of compounds). A specific list of target analytes is requested when the CCV acceptance criteria required are different from the EATL standard criteria noted above.

8.4.6 Sensitivity Verification:

8.4.6.1 A calculation is performed comparing the area counts of all internal standards from the midpoint of the Initial Calibration to the same internal standard in the continuing calibration verification to verify the instrument has not lost sensitivity from the ICAL to the daily batch. The area counts are compared with a maximum allowable percent drift of 40% D below the mid-point ICAL value. If the comparison passes then it should be documented in the instrument runlog. If the IS comparison fails this test another CCV will be analyzed. If this fails the comparison test as well, then the multiplier may be adjusted and the test repeated. If this also fails the %D test, the reason for the discrepancy is investigated, and if necessary a new analytical curve will be performed. A sensitivity check at the Reporting Limit or lower must be analyzed following the replacement of the concentrator trap. Once it has been verified as passing criteria, the analyst may proceed with analysis of a CCV. If the sensitivity check fails, the problem must be resolved and a new curve analyzed.

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8.4.6.2 Sensitivity must be evaluated when the trap is replaced or the changing to a different filament. Other instances include when the IS area counts in the CCV are greater than 40% drift when compared to the mid-point ICAL value. When this condition exists, all positive results greater than half the upper calibrated level, but less than the upper calibrated level, must be evaluated for saturation. If evidence of peak saturation is determined to be likely, as exhibited by more than two flat scans across the top of the peak in question, or a higher than normal abundance of secondary and tertiary ions in relation to the base peak, reanalysis of the sample in question must be performed at a dilution. The dilution should not exceed a factor of two greater than the original analysis in order to maintain reporting of all positive results detected in the initial analysis.

8.5 Laboratory Control Spike (LCS)

8.5.1 A Laboratory Control Spike is analyzed daily prior to sample analysis. Recovery limits are listed in Table A-1. Recoveries for 85% of standard compounds must be 70-130% with no recovery at <50. Alternatively, projects may require in-house generated or DoD QSM specified control limits.

8.5.2 If the stated criteria are not met, the system is checked and the same standard or different standards are re-analyzed. In the event that the criteria cannot be met for the full TO-14A/TO-15 list of compounds, a review of the data will be done by a QA Manager or Lab Manager and may approve the exceedances of a compound under special circumstances after reviewing the impact to the data quality. If approved, the data will be flagged and narrated.

8.5.3 LCS criterion for Non-Standard analytes is 60 – 140 % recovery subject to verification of client requirements in the Project Requirement Table. It is the responsibility of all analytical personnel to verify agreed upon LCS requirements because in some cases, a commitment has been made to criteria more stringent than 60 – 140%.

8.5.4 Recovery of any compound in the LCS that is $\leq 50\%$ of the expected value will result in either re-calibration, or analysis on a different instrument. Recovery of any compound that is $\geq 150\%$ of expected value, analysis may continue; however the system and/or standard preparation should be evaluated. If the problem is determined to be systematic (i.e. occurs on more than three consecutive days and/or on multiple instruments), the instrument must be evaluated and recalibrated if needed.

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- 8.5.5 Some projects require the LCS to be evaluated using control limits that are derived from historical data. Refer to SOP #48 "Preparation and Review of Control Charts" for the generation of control limits procedure. Recovery of any compound in the LCS that is outside of the historically derived control limits but within the default limits of 70-130%, analysis may continue and the excursions flagged and narrated.
- 8.5.6 An LCS is analyzed in duplicate (LCSD) daily prior to sample analysis. Refer to section 8.9.1 for the RPD acceptance criteria between the LCS and LCSD.
- 8.5.7 LCSD % Recovery acceptance criterion must be met for common risk driver compounds including Benzene, Toluene, Ethyl Benzene, m,p-Xylene, Vinyl Chloride, 1,1-Dichloroethene, cis-1,2-Dichloroethene, Trichloroethene, Tetrachloroethene or any other client specified risk driver compound.

8.6 Internal Standards

- 8.6.1 One mL of the IS blend is injected into the canister interface as each standard, blank, and sample is being loaded.
- Bromochloromethane
 - Chlorobenzene-d₅
 - 1,4-Difluorobenzene
- 8.6.2 Internal Standards' retention times for the blanks, QC samples and samples must be within ± 0.33 minutes of the retention times in the Continuing Calibration Check. In addition, the IS area must be within $\pm 40\%$ of the CCV's IS area for the blanks, QC samples and samples. The acceptance criteria are not subject to rounding. If the area count is below the lower limit as established by the CCV then the IS is deemed to have failed acceptance criteria and the steps in sections 8.6.2.1 and 8.6.2.2 are followed.
- 8.6.2.1 If the IS's for the blank do not pass the acceptance criteria, the system is inspected and the blank re-analyzed. Analysis is discontinued until the blank meets the IS criteria.
- 8.6.2.2 If the IS's in a sample do not pass the acceptance criteria, the sample must be re-analyzed. If the IS's are within limits in the re-analysis, the second analysis will be reported. If the IS's are out-of-limits a second time, then the sample will be diluted to get the IS's within limits.

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8.6.2.3 Oftentimes, the cause of low ISs in a sample are due to high levels of moisture in the sample. If this is suspected, a dry lab blank analysis is recommended prior to sample re-analysis. The affect of water is cumulative on the unit. If the ISs are out-of-limits a second time, then the sample will be diluted to get the ISs within limits. After an out-of-limit condition, a subsequent analysis must be done to demonstrate that the system is in control. If the ISs are within limits after the blank, the sample is analyzed again. If the sample causes the IS to be out-of-limits a second time, then it is determined that the sample contains matrix interference since the instrument was found to be in control when a system lab blank was analyzed. The data will be marked as acceptable and narrated accordingly.

8.7 Surrogates

One mL of the Surrogate blend is injected into the canister interface as each standard, blank, and sample is being loaded. The acceptance limits for Surrogate recoveries are 70 to 130%. Concentrations of Surrogates are equivalent to internal standard concentrations:

- 1,2-Dichloroethane-d4
- Toluene-d8
- 4-Bromofluorobenzene

8.7.1 If the Surrogate recoveries for the QC samples (i.e. CCV, LCS, or blank) do not pass the acceptance criteria, the system is inspected and the QC sample is re-analyzed. Analysis is discontinued until the QC sample meets the Surrogate recovery criteria.

8.7.2 If the surrogate recoveries for a sample are outside of these limits, the sample is re-analyzed unless obvious matrix interference is documented. If the Surrogate recoveries are within limits in the re-analysis, the second analysis will be reported. If the Surrogate recoveries are out-of-limits a second time, the data from the first analysis will be reported with a narrative indicating the acceptance criteria for Surrogate recoveries were exceeded. Additionally, the system must be shown to be in control by the analysis of a blank or sample with acceptable surrogate recoveries.

8.7.3 Some projects require the Surrogates to be evaluated using control limits that are derived from historical data. Refer to SOP #48 "Preparation and Review of Control Charts" for the generation of control limits procedure.

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8.8 Laboratory Blank

8.8.1 A humidified Nitrogen Laboratory Blank is analyzed upon completion of all required QC including calibration standards at the beginning of each day and at least once in every 24 hour shift. A Lab Blank is also analyzed in the event saturation-level concentrations are incurred to demonstrate that contamination does not exist in the chromatographic system. Each system has been evaluated for carryover, and a specific sample load volume or on-column concentration has been determined as the trigger for a Lab Blank. These ‘triggers’ are detailed in the QA-controlled Work Instructions next to each TO-15 unit. The acceptance criterion for Laboratory Blanks is a result less than the laboratory reporting limit (Limit of Quantitation) (see *Appendix A*).

8.8.1.1 In the event that the Laboratory Blank is contaminated by target compounds, tentatively identified compounds (TICs), non-Methane organic hydrocarbons (NMOC), or petroleum hydrocarbons (TPH), the following protocol should be observed. Initially, the Laboratory Blank should be re-analyzed to eliminate the possibility of an anomalous indication. If re-analysis is acceptable, analysis of client samples may continue.

8.8.1.2 Client samples that have analyte sublists that do not include the compound(s) in question should be substituted for full list samples. As these samples are analyzed, the operator should monitor the results to determine if the contamination has been removed from the system. In the event non-target contamination is present in the Laboratory Blank, analysis of samples not requiring TIC’s, NMOC, or TPH calculations can continue.

8.8.1.3 If there are no samples in-house that meet the criteria described in 8.8.1.2, the nature of the contamination should be evaluated. Samples that have been analyzed that cannot be re-analyzed will be flagged to note the non-conforming result.

8.8.1.4 Methylene Chloride, Acetone, Acetonitrile, Tetrahydrofuran, Ethanol, Bromomethane and 2-Butanone are acknowledged as common laboratory contaminants. Presence of these compounds at a concentration of < 5X the reporting limit is acceptable as long as the associated samples are not being analyzed for these analytes only. The associated samples will be flagged. If the contamination persists for more than two analytical batches, the issue should be escalated for evaluation in order to identify and remove the source of contamination.

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8.8.1.5 If other target compounds are present in the Laboratory, operations management will determine the impact on the usability of the data based on project requirements. If analysis proceeds, the associated data will be flagged to indicate detection above the RL in the Laboratory Blank.

8.8.1.6 If any Lab Blank contains either target or non-target hits at or above the reporting limit that are questionable, the blank should be re-analyzed to confirm the contamination. Manual integration of blanks is not permitted if the manual integration causes the analyte of interest to be below the reporting limit, except in the case of baseline contribution to the total ion current of a Surrogate (usually Bromofluorobenzene) when calculating NMOC/TPH-G concentrations and baseline contribution such as described in SOP #52 for Manual Integrations by GC/MS analyses. The Laboratory Manager, QA Manager or designated personnel must sign all manual integrations on Lab Blanks.

8.8.1.7 If the analysis request is for the full list of compounds laboratory blanks are not needed to continue running samples for the following compounds at the given concentration; Acetone at or above 60 ppbv, 2-Propanol at or above 80 ppbv, and Ethanol at or above 60 ppbv. If a sample contains any or all of these compounds above these concentrations, analysis of samples may continue. If the sample(s) following the high level analysis contain these compounds below 5x the reporting limit then a narration must be used describing the potential for high bias of these results. If the result for these compounds in the subsequent sample(s) is greater than 5X the reporting limit then no narration is necessary. If 10 or fewer analytes are requested and the target list contains these compounds, blanks must be used to demonstrate cleanliness of the system if concentrations are above the values listed previously.

8.8.2 Some projects require specific acceptance criteria for Method Blank as follow:

No analytes detected at $\geq \frac{1}{2}$ the RL. For common laboratory contaminants, no analytes detected \geq the RL.

If an analyte in the laboratory blank fails these criteria, a different Laboratory Blank canister is analyzed to rule out canister contamination. If

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the analyte in the Laboratory Blank still fails these criteria, the data is flagged with the appropriate data qualifying code (B) and the non-conformance is narrated unless, the analyte resulted in a non-detect in the samples.

- 8.8.3 In general, manual integration of target peaks in lab blanks is not permitted if the manual integration causes the analyte of interest to be below the reporting limit (Limit of Quantitation), except in the case of baseline contribution to the total ion current of a TIC or surrogate (usually Bromofluorobenzene) when calculating NMOC/TPH-G concentrations. However, there may be times that integration of compounds in the Lab Blank is necessary to accurately represent the data. All manual integrations on Lab Blanks must be signed by either a qualified Lab Manager or the QA Manager. Refer to SOP #52 “Manual Peak Integration and Background Subtraction for GC/MS Analyses”.

8.9 Laboratory Duplicates

- 8.9.1 Every daily analytical batch must include an LCS and an LCSD to evaluate instrument precision. The acceptance criteria for the relative percent difference (RPD) between the LCS and LCSD analyses should meet $\leq 25\%$. Sample analysis can continue as long as no more than 5% of the compound list (3 VOCs for a 62 compound list) exceeds the 25% RPD criterion. No compound should exceed 40%RPD. Any compound exceeding 25% RPD is narrated in the lab report. If a compound exceeds 40%RPD, the LCS is analyzed a 3rd time. If the limit is exceeded again, the system is evaluated. This evaluation includes verification of LCS canister pressure and instrument flow rates.

- 8.9.2 A duplicate sample analysis is performed only when specifically required by the Project Profile and/or Project Requirement Table associated with the workorder. The Relative Percent Difference (RPD) between the two analyses must be $\leq 25\%$ for all compounds detected at greater than 5 times the reporting limit (Limit of Quantitation). If this limit is exceeded, the sample is re-analyzed a third time, or analyzed on a different analytical system. If the limit is exceeded again, the cause is investigated and the system brought back to working order. If no problem is found on the system, the data is flagged to note the non-conforming event.

- 8.9.2.1 When three analyses do not result in acceptable precision ($\leq 25\%$ RPD for all compounds $> 5X$ LOQ), the instrument shall be eliminated as a potential source of the failure. This may be accomplished by choosing another sample and performing a duplicate analysis to determine if precision is possible in that

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instance. To whatever extent is possible, the original analytical conditions should be duplicated as well. This would imply use of the same syringe if relevant etc. Details to ensure duplication of original analytical conditions are left to the judgment of a scientist, Lab Manager, or the QA Manager. Do not dispose of the sample until the issue is resolved.

8.10 Field QC Samples

8.10.1 Neither TO-14A nor TO-15 describes field QC sample collection or acceptance criteria. However, clients may collect Field Blanks, Trip Blanks, and Field Duplicates. While there is no acceptance criteria established, analysts must monitor performance and note anomalies. For example, a positive result in the Field or Trip Blank requires closer inspection to ensure the anomaly wasn't incurred during sample handling and loading. Inspection may include verification of the canister analyzed and verification that the analytical system was clean.

8.10.2 Likewise, a field duplicate sample which shows an inconsistent chromatographic pattern as compared to the paired sample or concentrations differing by more than 40%RPD for many of the detections requires further investigation at the time of sample loading. Verification of the canisters identification and load volume and comparison of the FID screens are all appropriate to verify results.

8.10.3 Notate on the data checklist the anomaly and the items verified. If an anomaly was uncovered, then the field QC should be reanalyzed. If there are no findings then an SDR is generated informing the appropriate project manager following a note on the Data Review Checklist indicating the non conformance. Narrate the presence of a detection in the Trip Blank and Field Blank.

9.0 ***CALCULATIONS***

9.1 Response Factor

$$\text{Relative Response Factor (RRF)} = \frac{\text{Area of Compound}}{\text{Area of Int. Standard}} \times \frac{\text{Conc. Int. Standard (ppbv)}}{\text{Conc. of Compound (ppbv)}}$$

9.2 Sample Results

$$\text{Results Calculation} = \frac{\text{Area of Compound in Sample}}{\text{Area of Int. Standard in Sample}} \times \frac{\text{Conc. Int. Standard (ppbv)}}{\text{ICAL RRF}^*}$$

(ppbv on-column)

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* *The average RRF from the Initial Calibration Curve is used to quantitate results.*

ppbv in sample = ppbv on-column X Dilution Factor

Dilution Factor = Pressurization Factor* x Analytical Dilution Factor

Analytical Dilution Factor = $\frac{\text{Max load volume of unit (ml)}}{\text{Sample volume loaded (ml)}} \times (\text{Off-line dilution factor})$

* *The pressurization factor is determined by the lab measured canister receipt vacuum and final pressure. See Form 11.36 for calculations and a table of pressurization factors for a range of initial vacuums and final pressures of 5 and 15 psig.*

9.3 Total Petroleum Hydrocarbon (TPH) and Non-Methane Organic Compound (NMOC) calculations by GC/MS

The calculations performed for TPH and NMOC as well as additional TPH characterization including the hydrocarbon fractionation is beyond the scope of this SOP and is detailed in SOP #111.

9.4 Altitude Correction Factor for compound concentration

9.4.1 Per client's request, altitude correction factors may be applied to sample results using the following procedure: Atmospheric pollutant concentrations expressed as mass per unit volume of atmospheric air (e.g., mg/m³, µg/m³, etc.) at sea level will decrease with increasing altitude since the atmospheric pressure decreases with increasing altitude. The correction factor or change of atmospheric pressure with altitude can be estimated from the following equation:

$$\text{Correction Factor} = P_a = 0.9877^a$$

Pa = atmospheric pressure at altitude a, in atmospheres

a = altitude, in 100's of meters.

For example the altitude is 1800 meters. In 100 meters it would be 18.

$$P = 0.9877^{18} = 0.80$$

Apply correction factor to the final concentration. For example if the final concentration is 260µg/m³. Corrected concentration = 260 x correction factor = 260 x 0.8 = 208µg/m³ (See reference in section 15.0).

9.4.2 Correction factor must be provided to IT department in order for it to be applied to the formula for the final concentration. Results in the final report must be checked manually to validate the calculated result (see reference section for website information).

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10.0 *SAMPLE ANALYSIS*

10.1 Analytical Batch

The analytical batch is defined as all samples* up to 20 analyzed within 24 hours on one instrument. All QC and samples must be processed with the method associated with the daily analytical batch.

* Samples per batch are reportable analytical runs excluding QC samples, duplicate analysis and confirmation analysis.

10.2 Analytical Sequence

Initial 24-hour period:

BFB Tune Check
Initial Calibration
LCS/LCSD
Laboratory Blank
Samples (up to 20)

Subsequent 24-hour period:

BFB Tune Check
CCV
LCS/LCSD
Laboratory Blank
Samples (up to 20)

The "Subsequent 24-hour" sequence is followed every 24 hour period during which samples are analyzed until the system fails quality control acceptance limits.

Additional QC samples may be required by the program or project to be analyzed within the analytical batch. These details are included in the project PRT.

10.3 In the event of a daylight transition, a time shift may be observed during analysis of samples. If this occurs, data is acceptable and maybe used. The occurrence must be documented in the run log and on the Data Review Checklist of any affected work orders.

10.4 Some MSD's can be set to acquire both SIM and full scan data simultaneously. This generates two separate data files in the analytical software. One file contains SIM data following the operating procedures outlined in SOP#38 and the other contains full scan data following the procedures outlined in this SOP. This allows a lower reporting limit for the selected SIM compounds. The results for each sample in a report will be from two separate data files originating from the same analytical run. The two data files have the same base file name and are differentiated with a "sim" extension on the SIM data file.

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10.5 Validation of Reporting Limit (Limit of Quantitation)

- 10.5.1 Method Detection Limit (MDL) studies are analyzed for all standard TO-14A/TO-15 compounds following procedures as described in 40 CFR Pt. 136 App. B and Eurofins Air Toxics SOP #39.
- 10.5.2 The reporting limit (Limit of Quantitation) must be greater than the MDL before sample analysis can occur. If this is not achieved, corrective action, including raising the reporting limit is taken prior to continuing with sample analysis. See Appendix A for the TO-14A/TO-15 Reporting Limits.
- 10.5.3 In general, the reporting limit (Limit of Quantitation) is typically up to 10 times greater than the MDL but may be up to 50 times greater. If the reporting limit (Limit of Quantitation) exceeds this range, the Laboratory Manager and/or the QA Department will evaluate the RL to determine the reason and its approval.
- 10.5.4 The MDL verification sample must be analyzed on a quarterly basis. Refer to SOP #39 and WI39 MDL Procedure for the acceptance criterion.
- 10.5.5 A Method Detection Limit study will be performed for non-standard compounds only when prior arrangements with the client have been made and are documented in the Project Requirement Table. The reporting limit must be greater than the MDL before sample analysis can occur.

10.6 Quantitative Analysis

- 10.6.1 Quantitation is based on the integrated abundance of the primary ion for each analyte. If the response for any primary quantitation ion exceeds the Initial Calibration range of the GC/MS system, the sample is diluted and re-analyzed, excluding detections of the compounds noted in Section 10.7.2. If the response for any primary quantitation ion results in a value that rounds to the equivalent of the upper calibration range when expressing the result with 2 significant figures, re-analysis is not necessary.
- 10.6.2 When interference with the primary quantitation ion occurs, either the result is flagged with an "M" indicating matrix and a probable high bias or quantitation on the secondary ion is carried out after a new response factor (using the secondary ion) is generated from the initial calibration. Therefore, the same ion used to establish the response factor is used to quantify target analytes in the sample. This is noted in the laboratory narrative included in the report.

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10.6.3 The criterion for using the secondary ion for quantitation is a difference in the reported result of 50% or more. Discussion with the project manager and/or QA Manager may determine that quantitation using the secondary ion is not necessary given the objectives of the project and flagging will suffice. In the case when the difference is less than 50% and/or there is interference with the primary and secondary ion, the possibility of high bias is notated on the Data Review Checklist and in the final report.

10.7 Detections Outside of the Calibrated Range

10.7.1 Compounds detected in between current instrument MDL values and the low point of the calibration curve are reported only by client request and are flagged to indicate that the concentration is estimated.

10.7.2 Unless specifically directed otherwise by a client, detections of the following non-critical risk driver compounds: Acetone, Cyclohexane, Ethanol, Heptane, Hexane, Methyl Ethyl Ketone (2-Butanone), 2-Propanol, Propylene, 2,2,4-Trimethylpentane and Tetrahydrofuran above the high level of the curve are reported with an "E" flag and do not result in further dilution.

10.8 Tentatively Identified Compounds (TICs)

10.8.1 By project request, based on the computer generated searches, the identification of the ten highest non-target unknown peaks is reported. The spectra of these peaks are searched against the NIST library of greater than 50,000 compounds.

10.8.2 The total ion current is used for quantitation and calculation of TIC results. A TIC is determined to be present when the quantitated concentration is found to be greater than one tenth the concentration of the spiked Internal Standard.

10.8.3 The total ion current of the closest (by retention time, RT) non-interfered Internal Standard is used to calculate results (per SW-846 protocol). If all Internal Standards have interference, the Internal Standards in the Lab Blank are used to calculate results. A relative response factor of "1" is assumed. Match quality is useful in determining whether or not tentative identification should be reported, but is not the only criteria.

10.8.4 There are cases in which the NIST library match contains masses for a compound that are not within the conventional scanning range. In this instance match quality may appear to be poor when that is not the case.

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Eurofins Air Toxics protocol is to defer to the analyst's judgment and experience with regard to identification.

10.9 Compound Identification

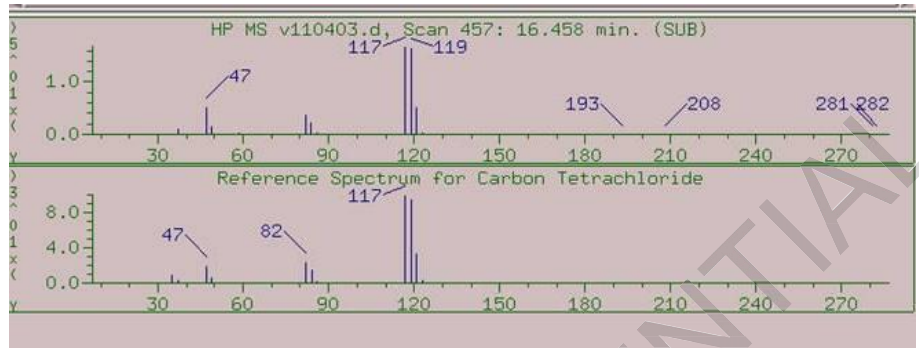
10.9.1 There are three criteria that must be satisfied to justify positive identification of a given compound.

1. The retention time must match the daily standard within a factor of less than 0.1 minutes for all characteristic ions (quantitation and qualifier ions) present in the spectrum. Shifts in target compound retention times that exceed this limit must be accompanied by a corresponding shift in retention time for the corresponding Internal Standard.
2. The peak shape should be Gaussian for the compound's characteristic ions with the exception of those at or near the reporting limit.
3. The mass ion fragmentation pattern should match the reference spectrum unless obvious interference is noted. There are two tools in the Target data processing software to determine a match – the compound's spectral pattern (relative ion intensities) and the characteristic ion peak area ratios. In general, the visual assessment of the spectral pattern is sufficient to proceed with the identification; however, the ion area ratios can provide quantitative confirmation when interference is not present and qualifier ions are properly integrated.

Spectral Pattern: The spectral pattern displays the ion intensities at the peak scan. Evaluating the ion intensities at the peak scan is analogous to evaluating ion peak "height" at the retention time. As such, the spectral pattern is not affected by peak integration of the quantitation and qualifier ions. The relative intensity of the characteristic ions and the full spectral fragmentation pattern is compared to the reference spectra for a visual confirmation. See Figure 1 for an example of spectral pattern evaluation for a full scan data file.

Figure 1. Spectral pattern evaluation showing match of quantitation and qualifier ions (119 and 117) relative intensity as well as entire fragmentation pattern with the reference spectrum.

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Ion area ratio: If needed, ion area ratios can also be evaluated using the integrated peak area of the quantitation ion and qualifier ions. As a guide, the ion area ratios of a compound should be within approximately 30% of the relative areas of these ions in the reference standard. The mid-point of the initial calibration curve is generally used as the reference to determine the ion area ratio target ranges. Figure 2 below shows the evaluation of the ion area ratio using the target ranges on the quantitation pages. (Please see Form I 1.80 for instruction to set up Target method to correctly show ranges in the Target report page.) Ion area ratios are less useful when the qualifier ions are not properly integrated due to low response or interference.

Figure 2. Ion area ratio evaluation showing compound ratio is within expected target range.

RT	EXP RT	(REL RT)	MASS	RESPONSE	CONCENTRATIONS		TARGET RANGE	RATIO
					ON-COL	FINAL		
59	Carbon Tetrachloride					CAS #: 56-23-5		
16.458	16.457	(1.033)	119	589303	7.20066	7.201	80.00- 120.00	100.00
16.458	16.457	(1.033)	117	601525			69.67- 129.67	102.07

10.9.2 To evaluate interferences and aid in comparison of the peak spectra to the reference spectral pattern, background subtraction can be used by the laboratory staff as a data evaluation tool to remove interfering ion masses from co-eluting compounds that may be masking the target compound. If background subtraction is needed in order to make a decision regarding the presence of a target compound then the subtraction should be included in the data package. The background subtraction of the spectra should be within ± 20 scans of the target peak. If the decision can be made without the use of background subtraction (i.e. retention time, characteristic ion pattern or area ratio match, and Gaussian peak shape) then it is not required to be included in the data package. Some ions in the reference

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spectra are not within the scan range (35-350 amu) and as such are not considered to be relevant to a positive identification.

10.9.3 If the above criteria are met then the compound has been positively identified. However, matrix interference may make it impossible to satisfy one or all of the above conditions. In these instances the chemist must evaluate the available data and determine whether the compound should be positively identified on the basis of the chemist's "best judgment". Advanced data analysis tools are available in the data processing software to aid in this evaluation as needed.

10.10 Manual Integrations

At times performing manual integrations on Initial Calibrations standards, QC, Laboratory Blanks and samples may be necessary. To accurately document ion ratios on the Target report page, if manual peak integration for the quantitation ion is needed in the ICAL or QC samples, then the qualifier ion peak shape is evaluated as well and properly integrated if needed. Refer to SOP #52 for Manual Integrations by GC/MS for proper manual integration procedures and documentation.

10.11 Analytical Procedures

The sample containers are connected to the inlet line of the TO-15 concentrator. Following the work instructions for the specific system, the sample connection is checked for leaks, and the sample is loaded onto the system. During the load step, a 1 mL gas sample loop filled with IS/S is swept onto the sorbent trap. If samples are pressurized with Helium, see work instructions for adjusting and verifying accurate mass flow controller operation.

10.12 Sample Dilutions

10.12.1 To obtain analyte concentrations within the calibrated range of the detectors and prevent contamination of the system, samples are typically screened prior to analysis on a GC/MS. (See SOP#114). Occasionally, a GC/FID unit may be used specifically when high petroleum concentrations are expected. Dilutions are determined based on estimated concentrations of the highest target and/or non-target compounds.

10.12.2 Following the screening process, if the sample or samples is deemed to require a dilution factor of more than 10, the sample(s) can be analyzed via TO-14A/TO-15 (5&20 ppbv) following SOP #91 with the following stipulations:

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- 5&20 instrumentation must be validated via Initial Calibration, MDL and daily CCV/LCS/Lab Blank for all of the target compounds associated with the Project Profile and workorder in question.
- 10.12.3 Other necessary changes may be made on a project by project basis in order to make the transition from SOP #83 to SOP #91 analytically consistent.
- 10.12.4 An undiluted analysis involves loading the standard load volume as identified by the ICAL. In general this standard load volume is 250 mL for the LL analysis, or 400ml on the autosampler systems. The dilution factor is obtained by dividing the full load volume by the sample volume loaded. All samples submitted for TO-15 LL are screened prior to analysis. If samples contain high concentrations of target and/or non-target VOCs, samples may be analyzed by an alternative TO-15 method (i.e. standard or 5&20) with a higher dynamic calibration range.
- 10.12.5 When determining the load volume for the sample, both target and non-target compounds are evaluated by reviewing the screening data. First, the sample should be diluted for the highest target compound with the goal of hitting between 24 ppbv and 40 ppbv for LL analysis in an effort to provide the client with the lowest reporting limits. It is not required to re-analyze a sample at a lesser dilution if the actual on-column concentration is above 16ppbv. Additionally, unless specified by the project, detections of the following compounds at levels above the high end of the curve do not result in a subsequent dilution: Acetone, Cyclohexane, Ethanol, Heptane, Hexane, 2-Butanone, 2-Propanol, Propylene, 2,2,4-Trimethylpentane, and Tetrahydrofuran. These compounds are often present at high concentrations but are not considered to be risk-drivers. Instead, these results are reported with an “E” flag to indicate the levels exceeded the upper limit of the calibration.

Second, the screening results should also be evaluated for non-target compounds since high concentrations can contaminate the loading interface and interfere with the analysis. Non-target compounds eluting early in the screen do not require as aggressive of a dilution since carryover and interference is expected to be minimal. Non-target compounds with a lower vapor pressure (eluting mid and late on the GC screening run) require dilution to minimize interference and carryover. In general, mid- and late-eluting non-target compounds

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require dilution when concentrations are above approximately 100 ppbv.

10.12.6 Dilution for the target and non-target compounds is required if screening data indicates that they may saturate the detector.

10.12.7 In the event the Project Profile requires that all analytical runs be reported, the analytical runs must be evaluated at the time of analysis to ensure that the RPD for compounds within linear range and greater than 5 times the RL do not exceed 25%. Re-analysis or system inspection/maintenance may be required to resolve any discrepancies.

11.0 CORRECTIVE ACTION PROCEDURES

A Request for Corrective Action (CAR) is initiated any time either the EATL SOPs or client-prescribed QC protocol are not followed, or in any other instance that sample results are adversely affected. The corrective action procedure is documented in EATL SOP #61.

12.0 DATA REVIEW

12.1 Analytical Data Review

As the analytical sequence is analyzed throughout the day, the data is reviewed by the analyst or scientist using the following steps:

- Check for any project-specific requirements.
- Verify holding time.
- Verify the BFB Tune check, CCV, LCS, LCSD.
- Verify that Method Blank has no hits above reporting limit (with the exceptions outlined in 8.7).
- Verify sample results:
 - a. Verify the retention time.
 - b. Verify that correct amount of sample was analyzed.
 - c. Verify the automated peak integration.
 - d. Verify that result concentrations are within linear range of Calibration Curve (generally in the upper 60% for dilutions).
- Initial and date raw data and /or logbook entry to indicate that the data is acceptable.
- Apply appropriate data flags.
- Describe unusual events on Data Review Checklist.
- Verify results of the data validation report from Lumen, make corrections to the raw data as needed to remove errors identified by Lumen.

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Notes:

- *A secondary review of the analytical runs is required when the analyst or scientist is not signed off on the analysis.*
- *Preparation and review of Laboratory Narrative are carried out as explained in EATL SOP #45.*

12.2 Write-up and Final Report Review

The analyst or scientist performing the data write-up and final client report reduces the data by reviewing the Target data files. The peaks in each sample are reviewed for the correct integration and identification. The integration is modified only when necessary following guidance in EATL SOP #52. The criteria for compound identification are described in section 10.9. The data is evaluated for the project required sublist only.

When the sample Target review is complete, the sample files are transferred to the Atlas database. The client report is compiled in the Atlas workorder editor following EATL SOP #78. Check the following when compiling the report:

- Prepare and review narrative (EATL SOP #45), detailing QC non-compliance as needed.
- Evaluate data package from a data user perspective. Does the data make sense?

After the Atlas report is complete, LUMEN, a rules-based data validation tool, is used to verify QC criteria, hold time, data qualifier flags, manual integration documentation, tune clock time, retention time, and appropriate ICAL. Review all errors, and correct or insure discrepancies are addressed on the Data Review sheet or explained in the Lab Narrative if data quality is impacted.

When complete sign and date the 'Write-up' field on the Data Review Checklist and email the report to the client. Update the Atlas database to reflect the 'date reported'.

12.3 Technical Data Review

The Scientist or designated personnel performs a technical data review on 100% reports if the write-up review analyst or scientist is not signed-off for the method. This review follows all the steps mentioned in the Analytical data review (see Section 12.1).

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12.4 Report Review

The Report Review represents a 3rd level of review which is required for DoD and client specific projects only. Refer to the Data Review Checklist for the specific items requiring review.

12.5 QA Data Review

A thorough QA data review is performed by the QA Department on final data packages requesting 100% review. The QA review entails verification that project and QC requirements are met. Failure to meet QC and/or project requirements results in a Corrective Action Request (CAR) and documentation. Dilution factors, analyte retention times, peak integration areas, concentration calculations, unit conversions, and reporting limits are also checked. Field and Trip Blanks are checked and trends are observed.

13.0 INSTRUMENT MAINTENANCE

13.1 Instruments are monitored on a daily basis by the bench analyst for any potential failure. The analysis of blanks and control standards at the start of the day and as analysis continues helps to provide real-time feedback to the analyst on the condition of the instruments. Routine maintenance includes: mass spectrometers, sample introduction system and gas chromatograph.

13.1.1 The bench analyst will document any routine or major non-routine maintenance in the bound instrument logbook assigned to each instrument. The date of the maintenance, what work was performed and analyst initials are included.

13.2 Mass Spectrometers

- Periodic check of vacuum ion gauge (Increase in ion count indicates a potential leak)
- Daily (every 24 hours) tune check with BFB
- Cleaning of ion source on quarterly basis or as needed
- The pump oil level and quality is visually checked every month and at the time of source cleaning to ensure proper vacuum pump function, and oil is changed as needed.
- A sensitivity check must follow a routine maintenance to ensure that a standard representing the low point concentration of the curve meets criteria.

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13.3 Sample Introduction System

13.3.1 To ensure a clean sample introduction system, if necessary, the lines and trap are “steam-cleaned” by analyzing a humidified system blank. This takes place every day or as needed based on the compound list following standard’s (i.e., CCV, LCS) analysis. Humidified system blanks are also analyzed after saturation-level detections in samples.

13.4 Gas Chromatograph

Routine maintenance includes the following:

13.4.1 As needed, clip approximately 3 feet off the front end of the capillary column, and if necessary, the back end as well.

13.4.2 Replace the injection port liner as needed.

- The liner is replaced by removing the inlet cap using a wrench and releasing the liner from the inlet body using a pair of tweezers. Care should be taken not to get fingerprints on any inside surface.

13.4.3 Visually inspect the septum on the valve syringe injection port and replace as needed.

- Change the septum on the GC as needed. Always use a high temperature/low bleed septa and take care not to leave fingerprints on any inside heated surface. Wear a pair of white cotton gloves or use tweezers to handle the septa. Lower the oven temperature to approximately 40°C. Remove the inlet cap with a wrench, remove the old septa with a pair of tweezers and insert the new septa.

13.4.4 The column is replaced when chromatography peak shape or resolution degrades. Similarly, if the column bleed profile rises with age then the column needs replacing.

- Use new black graphite ferrules each time and clip off approximately 1” of column after inserting it through the ferrule. This will remove any graphite particles that may have scrapped off into the column. Tighten the column nut and ferrule finger tight and one quarter turn with a wrench. Tightening any more only crushes the ferrule and may damage the column.

13.4.4.1 If a new column of the same dimension and phase is being replaced a new MDL is not necessary.

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14.0 DELIVERABLES

Data reporting packages are prepared as described in SOP #78 – Generation of Eurofins Air Toxics Data Deliverables, Electronic Conversion, and Archival.

15.0 REFERENCES

EPA Method TO-14A

Compendium of Methods for Determination of Toxic Organic Compounds in Air, EPA Methods, Second Edition, January 1999. *EPA/625/R-96/010b*

EPA Method TO-15

Compendium of Methods for Determination of Toxic Organic Compounds in Air, EPA Methods, Second Edition, January 1999. *EPA/625/R-96/010b*

SW-846 Method 8000B

Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Final Update III, Revision 1, December 1996

Volatile Organic Analysis of Ambient Air in Canisters - Draft Method USEPA Contract Laboratory Program, Revision VCAA01.0, December 1991

Eurofins Air Toxics NELAP Quality Manual
Definitions and Terms, Appendix A.

<http://www.air-dispersion.com/formulas.html>. Author Milton R. Beychok (accessed October 29, 2009).

List of Appendices

Appendix A. Reporting and QC Limits

Table A-1. Method TO-14A/TO-15 Standard Compounds

Table A-1b. Method TO-14A/TO-15 Example Non-Standard Compounds

Table A-2. Internal Standards

Table A-3. Surrogates

Table A-4. Summary of Calibration and QC Procedures for Methods TO-14A/TO-15 for Volatile Organic Compounds

Appendix B. BFB Tune Criteria

Appendix C. Dilution Decision Tree

Appendix D. Internal Standard and Associated Target Compounds and Surrogates

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Appendix A

Reporting and QC Limits

Table A-1. Method TO-14A/TO-15(Standard Compounds)

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS* (%R)	Precision Limits (%RPD)
1,1,2,2-Tetrachloroethane	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,1,2-Trichloroethane	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,1-Dichloroethane	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,1-Dichloroethene	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,2,4-Trichlorobenzene	0.5	≤30%	70 - 130	70 - 130	≤ 25
1,2,4-Trimethylbenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,2-Dibromoethane (EDB)	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,2-Dichlorobenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,2-Dichloroethane	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,2-Dichloropropane	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,3,5-Trimethylbenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,3-Dichlorobenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,4-Dichlorobenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Benzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Bromomethane	0.5	≤30%	70 - 130	70 - 130	≤ 25
Carbon Tetrachloride	0.1	≤30%	70 - 130	70 - 130	≤ 25
Chlorobenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Chloroethane	0.5	≤30%	70 - 130	70 - 130	≤ 25
Chloroform	0.1	≤30%	70 - 130	70 - 130	≤ 25
Chloromethane	0.5	≤30%	70 - 130	70 - 130	≤ 25
Chlorotoluene (Benzyl Chloride)	0.1	≤30%	70 - 130	70 - 130	≤ 25
cis-1,2-Dichloroethene	0.1	≤30%	70 - 130	70 - 130	≤ 25
cis-1,3-Dichloropropene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Dichloromethane (Methylene Chloride)	0.2	≤30%	70 - 130	70 - 130	≤ 25
Ethylbenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Freon 11 (Trichlorofluoromethane)	0.1	≤30%	70 - 130	70 - 130	≤ 25
Freon 113 (Trichlorotrifluoroethane)	0.1	≤30%	70 - 130	70 - 130	≤ 25
Freon 114	0.1	≤30%	70 - 130	70 - 130	≤ 25
Freon 12 (Dichlorodifluoromethane)	0.5	≤30%	70 - 130	70 - 130	≤ 25
Hexachlorobutadiene	0.5	≤30%	70 - 130	70 - 130	≤ 25
m,p-Xylene	0.1	≤30%	70 - 130	70 - 130	≤ 25

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Methyl Chloroform (1,1,1-Trichloroethane)	0.1	≤30%	70 - 130	70 - 130	≤ 25
o-Xylene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Styrene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Tetrachloroethene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Toluene	0.1	≤30%	70 - 130	70 - 130	≤ 25
trans-1,3-Dichloropropene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Trichloroethene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Vinyl Chloride	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,3-Butadiene	0.1	≤30%	70 - 130	70 - 130	≤ 25
1,4-Dioxane	0.1	≤30%	70 - 130	70 - 130	≤ 25
2-Butanone (Methyl Ethyl Ketone)	0.5	≤30%	70 - 130	70 - 130	≤ 25
2-Hexanone	0.5	≤30%	70 - 130	70 - 130	≤ 25
4-Ethyltoluene	0.1	≤30%	70 - 130	70 - 130	≤ 25
4-Methyl-2-Pentanone (MIBK)	0.1	≤30%	70 - 130	70 - 130	≤ 25
Acetone	1.0	≤30%	70 - 130	70 - 130	≤ 25
Bromodichloromethane	0.1	≤30%	70 - 130	70 - 130	≤ 25
Bromoform	0.1	≤30%	70 - 130	70 - 130	≤ 25
Carbon Disulfide	0.5	≤30%	70 - 130	70 - 130	≤ 25
Cumene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Cyclohexane	0.5	≤30%	70 - 130	70 - 130	≤ 25
Dibromochloromethane	0.1	≤30%	70 - 130	70 - 130	≤ 25
Ethanol	0.5	≤30%	70 - 130	70 - 130	≤ 25
Heptane	0.5	≤30%	70 - 130	70 - 130	≤ 25
Hexane	0.5	≤30%	70 - 130	70 - 130	≤ 25
Isopropanol	0.5	≤30%	70 - 130	70 - 130	≤ 25
Methyl t-Butyl Ether (MTBE)	0.1	≤30%	70 - 130	70 - 130	≤ 25
Propylbenzene	0.1	≤30%	70 - 130	70 - 130	≤ 25
Tetrahydrofuran	0.5	≤30%	70 - 130	70 - 130	≤ 25
trans-1,2-Dichloroethene	0.1	≤30%	70 - 130	70 - 130	≤ 25
2,2,4-Trimethylpentane	0.5	≤30%	70 - 130	70 - 130	≤ 25
3-Chloroprene	0.5	≤30%	70 - 130	70 - 130	≤ 25

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Appendix A (continued)

Table A-1b. Method TO-14A/TO-15 Example Non-Standard Compounds

Analyte	RL/LOQ (ppbv)	QA Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
Acrolein	0.5	≤40	60 – 140	60 – 140	± 25
Butane	0.5	≤40	60 – 140	60 – 140	± 25
Ethyl tert-Butyl Ether	0.5	≤40	60 – 140	60 – 140	± 25
Isopentane	0.5	≤40	60 – 140	60 – 140	± 25
Isopropyl Ether	0.5	≤40	60 – 140	60 – 140	± 25
Methylcyclohexane	0.5	≤40	60 – 140	60 – 140	± 25
Naphthalene	0.5	≤40	60 – 140	60 – 140	± 25
Propylene	1.0	≤40	60 – 140	60 – 140	± 25
tert-Amyl Methyl Ether	0.5	≤40	60 – 140	60 – 140	± 25
Vinyl Acetate	0.5	≤40	60 – 140	60 – 140	± 25
tert-Butyl Alcohol	0.5	≤40	60 – 140	60 – 140	± 25
TPH (Gasoline)	10	1- Point Calibration	NA	ICV only: 60 – 140	± 25
NMOC (Hexane/Heptane)	2.0	1- Point Calibration	NA	NA	± 25

Table A-2. Internal Standards

Analyte	Recovery Limits (%R)
Bromochloromethane	60 – 140
1,4-Difluorobenzene	60 – 140
Chlorobenzene-d ₅	60 – 140

Table A-3. Surrogates*

Analyte	Recovery Limits (%R)
1,2-Dichloroethane-d ₄	70 – 130
Toluene-d ₈	70 – 130
4-Bromofluorobenzene	70 – 130

* In-house generated control limits may be used per client's request.

Appendix A (continued)

Table A-4. Summary of Calibration and QC Procedures for Methods TO-14A/TO-15 (VOC)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours,	TO-15 ion abundance criteria	Correct problem then repeat tune.
Minimum 5-Point Initial Calibration (ICAL)	Prior to sample Analysis.	% RSD \leq 30 with four compounds allowed out to \leq 40% RSD.	Correct problem then repeat initial calibration curve.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS)	After each initial calibration curve, and daily prior to sample analysis.	Recoveries for 85% of Standard compounds must be 70-130%. No recovery may be $<$ 50%. ICV evaluated on a full list basis at time of calibration. * If specified by the project, in-house generated or DoD specified control limits may be used for the LCS.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Initial Calibration Verification and Laboratory Control Spike (ICV and LCS) for Non-Standard Compounds	Per client request or specific project requirements only.	Recoveries of compounds must be 60-140%. No recovery may be $<$ 50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.
Continuing Calibration Verification (CCV) for Standard compounds	At the start of each day and 24-hour clock	70-130%.	Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than four compounds from the standard list recover outside of 70-130% or $>$ 10% of VOCs if short list is used (40 compounds or less), corrective action will be taken. If any compound exceeds 60-140%, samples are not analyzed unless data meets project needs. Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV for Non-Standard Compounds	Per client request or specific project requirements only.	Recoveries of compounds must be 60-140%. No recovery may be $<$ 50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary to determine the source of error. Re-calibrate the instrument if the primary standard is found to be in error.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present.	Results less than the laboratory reporting limit (Tables A-1).	Inspect the system and Re-analyze the blank. B-flag data for common contaminants.
Internal Standard (IS)	As each standard, blank, and sample is being loaded.	Retention time (RT) for blanks and samples must be within ± 0.33 min of the RT in the CCV and within $\pm 40\%$ of the area counts of the daily CCV internal standards.	For blanks: inspect the system and reanalyze the blank. For samples: re-analyze the sample unless obvious matrix interference is documented. If the Iss are within limits in the reanalysis, report the second analysis. If Iss are out-of-limits a second time, report data from first analysis and narrate.
Surrogates	As each standard, blank, and sample is being loaded.	70 – 130% R. * If specified by the project, in-house generated control limits may be used.	For blanks: inspect the system and reanalyze the blank For samples: reanalyze the sample unless obvious matrix interference is documented. If the %Rs are within limits in the re-analysis, report the second analysis. If %Rs are out-of-limits a second time, report data from first analysis and narrate.
Laboratory Duplicates – Laboratory Control Spike Duplicate (LCSD)	One per analytical batch.	RPD $\leq 25\%$.	Narrate exceedances. If more than 5% of compound list outside criteria or if compound is $>40\%$ RPD, investigate the cause and perform maintenance as required. If instrument maintenance is required, calibrate as needed.

Appendix B

BFB Tune Criteria

4-BROMOFLUOROBENZENE KEY IONS AND ION ABUNDANCE CRITERIA

m/e	Ion Abundance Criteria
50	8.0 to 40.0% of mass 95
75	30.0 to 66.0% of mass 95
95	Base Peak, 100% Relative Abundance
96	5.0 to 9.0% of mass 95
173	<2.0% of mass 174
174	50.0 to 120.0% of mass 95
175	4.0 to 9.0% of mass 174
176	93.0% to 101.0% of mass 174
177	5.0 to 9.0% of mass 176

Appendix C

Eurofins Air Toxics, Inc.

Title: IS Low Level
 Instruction#: 1.1.23

Release Date: 10/10/2014
 Revision Date: 10/10/2014
 Revision#: 2

Modified EPA Methods TO-14A/TO-15 Low Level
 Internal Standard and Associated Target Compounds and Surrogates

Bromochloromethane	1,4-Difluorobenzene	Chlorobenzene-d5
Target Compounds:	Target Compounds:	Target Compounds:
Freon 12	Benzene	trans-1,3-Dichloropropene
Freon 114	1,2-Dichloroethane	1,1,2-Trichloroethane
Chloromethane	Heptane	Tetrachloroethene
Vinyl Chloride	Trichloroethene	2-Hexanone
1,3-Butadiene	1,2-Dichloropropane	Dibromochloromethane
Bromomethane	1,4-Dioxane	1,2-Dibromoethane (EDB)
Chloroethane	Bromodichloromethane	Chlorobenzene
Freon 11	cis-1,3-Dichloropropene	Ethyl Benzene
Ethanol	4-Methyl-2-pentanone	m,p-Xylene
Freon 113	Toluene	o-Xylene
1,1-Dichloroethene	Surrogates:	Styrene
Acetone	Toluene-d8	Bromoform
2-Propanol		Cumene
Carbon Disulfide		1,1,2,2-Tetrachloroethane
3-Chloropropene		Propylbenzene
Methylene Chloride		4-Ethyltoluene
Methyl tert-butyl ether		1,3,5-Trimethylbenzene
trans-1,2-Dichloroethene		1,2,4-Trimethylbenzene
Hexane		1,3-Dichlorobenzene
1,1-Dichloroethane		1,4-Dichlorobenzene
2-Butanone (Methyl Ethyl Ketone)		alpha-Chlorotoluene
cis-1,2-Dichloroethene		1,2-Dichlorobenzene
Tetrahydrofuran		1,2,4-Trichlorobenzene
Chloroform		Hexachlorobutadiene
1,1,1-Trichloroethane		Surrogates:
Cyclohexane		Bromofluorobenzene
Carbon Tetrachloride		
2,2,4-Trimethylpentane		
Surrogates:		
1,2-Dichloroethane-d4		

SOP Revision History

Revision Date	Revision #	Changes	Reviewer(s)
11/27/12	10	Amend Appendix A Table 1-A RL for Bromomethane changed from 0.1ppbv to 0.5ppbv	Excelsa Alcantara
1/9/13	10	10.12.5 sample dilution	Sepideh Saeed
07/19/13	11	Update RL for Chloromethane to 0.5ppbv	Sepideh Saeed
01/16/14 2-13-14	12	Added initial calibration section 8.2.11 Add NIST08.1 library search software Section 13.2 updated for oil maintenance	Samantha Black Heidi Hayes Bahar Amiri
8/7/14	13	Added comment to see WI #6 in section 10.9.3 Updated section 10.10 to cover ion ratios pertaining to manual integrations Updated 13.2 to reflect current practice of pump maintenance timed with source cleaning.	Ed Jakab Heidi Hayes
10/14/14 10/30/14 11/4/14	14	Updated select sections to include information pertaining to CIAA Advanced Auto sampler systems: 7.3, 8.1.1, & 10.12.5 Updated document to reflect the standard provider "Spectra" changed to Linde Spectra Environmental Gases. Updated Appendix D. Updated section 10.9 to include additional detail regarding qualitative identification, distinguishing between spectral pattern and ion area ratios. The comparison of ion area ratios to the mid-level of the ICAL was also specified as the reference.	Samantha Black Bahar Amiri Heidi Hayes
2/24/16	15	Section 8.5.6 LCSD criteria updated, Section 8.7 defined surrogates are same concentration as internal standards, Appendix C removed due to non-relevance, Amended table A-1b; RL for Propylene raised 0.5ppbv → 1.0ppbv Section 4.2 updated regarding low level analysis of Helium pressurized samples, Table A-4 updated to add ICV is evaluated based on full list.	Samantha Black

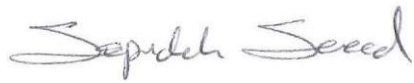
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		<p>Added dilution factor calculations to section 9.0</p> <p>Removed sections 8.3 referencing the use of control limits for ICV. Clarified text in QC table A-4 to specify statistical control limits and/or DoD specified apply only to LCS. Added “DoD specified” limits as an option along with in-house statistically generated limits throughout SOP.</p>	Heidi Hayes
11/30/16	16	<p>Added section 8.4.2.2: 10% exceedance criteria defined for CCV.</p> <p>Table A-4: added 10% exceedance criteria.</p> <p>Updated Section 8.2.3 to clarify setting Retention Time Windows using the ICAL and when to use the CCV</p> <p>Updated 4.2 to reference WI-MGP_TO-15 procedures</p>	<p>Ed Jakab</p> <p>Melanie Levesque</p> <p>Heidi Hayes</p>
02/17/17	17	<p>Section 5.3: Red-tagging canisters moved to WI38_83 C2 interface and WI38_83 PE interface (MSD-E).</p>	Ed Jakab
04/19/18	18	<p>Section 7.2.2: Entech NT7400 Precision replaced Entech NT4600 Dynamic Diluter.</p> <p>Section 10.5.3 updated to reference WI39 MDL Procedure</p> <p>Added section 8.5.7: LCSD %R acceptance criterion for risk drivers;</p> <p>Update Freon 12 RL to 0.5 ppbv per RTC-086; Section 8.2.1 and Table A-4 –</p> <p>Removed no longer relevant exceptions to QC criterion; Section 8.0 – Added introduction statement referencing DoD/project specific QC parameters in PRTs; Sections 8.2.6 and 8.2.10 –</p> <p>Removed verbiage and reference to calibration level table A-1 which is not applicable and replaced with verbiage relevant to subsequent sections; Section 10.2 – Added verbiage to indicate additional QC samples may be required in sequence; Sections 10.5.1 and 10.5.2 – Updated MDL verbiage.</p>	<p>Andrew Toyama</p> <p>Melanie Levesque</p>
4/29/19	19	<p>Update RL for Acetone and Hexane in</p>	Heidi Hayes

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		Table A-1. (Acetone from 0.5 to 1.0 ppbv and Hexane from 0.1 to 0.5 ppbv) RTC-106	
09/13/19	20	Update Heptane RL to 0.5ppbv per RTC-108 in Table A-1	Ed Jakob
02/28/20	21	Update Cyclohexane RL to 0.5ppbv per RTC-112 in Table A-1	Melanie Levesque
10/07/20	22	Sections 6.1 & 7.1.1 – Updated list of vendors. Section 7.3 – Removed references of methanolic BFB use and direct injection of BFB into the GC injection port per RTC-119. Updated BFB gas cylinder & working standard concentrations. Section 7.4 –Updated list of vendors. Updated concentration for IS/S gas cylinder blend & associated working standard. Removed reference of neat materials use to prepare IS/S-no longer relevant. Section 8.1.1.-Updated tune procedures per RTC-119.	Steven Nguyen
10/21/20		Sections 7.2.1 and 7.2.3 – Update water addition to a 100 uL aliquot to humidify working standards. (RTC-122)	Heidi Hayes

Document Owner/Laboratory Director:



Date: 10/21/2020



Technical Director:

Date: 10/21/20

Quality Assurance:



Date: 10/21/20

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ATTACHMENT B

Field Documentation Forms

1. Chain of Custody Form
2. SUMMA® Sample Train Shut-In Test Log
3. Soil Gas Probe Integrity Testing Log
4. Soil Gas Sample Collection Log



CHAIN OF CUSTODY

296 12th St
Marina, CA 93933
(831) 384-3735

Chain of Custody #: _____
Carbon Copies: White - Laboratory Yellow - Ahtna

Project Information:				Analysis Requested								Lab Sample Receipt	
Project Location: _____		Sampler/s: _____										Laboratory Sample Delivery Group #: _____	
Project Name: _____		Report To: _____										Custody Seal: _____	
Project Number: _____		E-Mail: _____										Temp (°C): _____	
Sampling Event: _____		Laboratory: _____											

Lab Number	Sample Number/Description	Sample Collection					SUMMA Sample Tracking			Notes
		Date	Time	Matrix	Preserv.	# of Containers	Canister ID	Regulator ID	Final Vacuum ("Hg)	

Turnaround Time: _____ : Standard _____ : 3-5 Day Rush _____ : 48 Hour Rush _____ : 24 Hour Rush	Comments: _____		
Matrix Types: A: Air; W: Water; S: Soil; SG: Soil Gas; O: Other: _____			
Preservative Types: H ₂ SO ₄ : sulfuric acid; HNO ₃ : nitric acid; HCl: hydrochloric acid; N: none; O: Other: _____			
Shipment: Shipment Method: _____ Shipment Tracking ID: _____			
Chain of Custody Tracking:			
Relinquished By Sampler: _____	Date/Time: _____	Received By: _____	Date/Time: _____
Relinquished By: _____	Date/Time: _____	Received By: _____	Date/Time: _____
Relinquished By: _____	Date/Time: _____	Received By Laboratory: _____	Date/Time: _____

SUMMA Sample Train Shut-In Test Log

Soil Gas Monitoring

Site 12, Former Fort Ord, California

Canister ID	Sample ID	Date	Start:	End:	Pass/Fail*
			Time/Inch, Hg	Time/Inch, Hg	

Notes:

Hg = mercury

The vacuum at the start and the end of the shut-in test shall be read by the same field person from the same perspective to assure comparability.

*Passing of the shut-in test is a maximum drop in vacuum of 1 inch Hg after 15 minutes of observation with Summa canister connected to the sample train (with inlet closed).

Page _____ of _____

Field Personnel Signature _____

Date _____



Soil Gas Probe Integrity Testing Log

Soil Gas Monitoring

Site 12, Former Fort Ord, California

		Time (min):	0*		5		10		15		End Time	Pass/Fail ²
			Detector:	Shroud	Inline	Shroud	Inline	Shroud	Inline	Shroud		
Probe ID ¹	Date	Start Time	% He	% He	% He	% He	% He	% He	% He	% He		

Notes:

% = percent (by volume) min = minutes
 He = helium ID = identification

¹ All probe depths in a nested soil gas probe cluster shall be integrity (leak) tested.

² DTSC's *Advisory, Active Soil Gas Investigations* provides the opinion that a 5% ambient air dilution is inconsequential to sample integrity. When sampling under a 20% helium in air atmosphere, 1% helium detected in the purge gas represents a 5% ambient air sample dilution. If the concentration of helium in the purge sample is greater than or equal to 5% of the helium concentration in the shroud, corrective action is necessary to remedy the leak.

* Inline detector reading at time =0 is baseline reading with the detector disconnected from sampling assembly.

Field Personnel Signature: _____ Date _____





Soil Gas Sample Collection Log

Soil Gas and Soil Vapor Extraction Treatment System Monitoring

Site 12, Former Fort Ord, California

Date: _____ Sampler: _____ Weather: _____

Probe ID: _____ Location Description: _____

Probe Leak Test (Pass/Fail): _____

ORIGINAL SAMPLE INFORMATION:

Sample Container: _____ 1.0-L SUMMA Canister _____ 100% Certified _____ Other: _____

Canister ID: _____ Sample Manifold ID: _____

Equipment (Shut-In) Test (Pass/Fail): _____ Sample ID: _____

Collection start time: _____ Collection end time: _____

Initial Canister Pressure/Vacuum (inches Hg): _____ (vacuum of at least – 25" Hg)

Final Canister Pressure/Vacuum (inches Hg): _____ (vacuum between – 4" to – 8" Hg)

DUPLICATE SAMPLE INFORMATION: _____ Not applicable

Sample Container: _____ 1.0-L SUMMA Canister _____ 100% Certified _____ Other: _____ Canister ID: _____

Duplicate Manifold ID: _____ Equipment (Shut-In) Test (Pass/Fail): _____

Sample ID: _____ Collection start time: _____ Collection end time: _____

Initial Canister Pressure/Vacuum (inches Hg): _____ (vacuum of at least –25" Hg)

Final Canister Pressure/Vacuum (inches Hg): _____ (vacuum between – 4" to – 8" Hg)

ANALYSES AND REVIEW:

Analytes Requested: _____ TCE and PCE _____ Other (list): _____

By method: _____ EPA TO-15 (5&20) _____ EPA TO-15 (Low-Level) Laboratory: Eurofins Air Toxics, Inc.

Comments: _____

Sampler Signature: _____ Date: _____

Reviewer Signature: _____ Date: _____

Notes:

"Hg = inches mercury

L = liter

ID = identification

ATTACHMENT C

Electronic Data Deliverable File Specifications

1. ADR Electronic Data Deliverable (EDD) File Specifications
2. Instructions for EDF File Specifications

ADR Electronic Data Deliverable (EDD) File Specifications

The ADR EDD consists of three separate, comma-delimited ASCII text files or Excel CSV files (two, if instrument calibration information is not required by the project). Each file corresponds to a table in the ADR application. These tables are identified as the Analytical Results Table (A1), Laboratory Instrument Table (A2), and Sample Analysis Table (A3). Each file follows the naming convention of using the Laboratory Reporting Batch ID (SDG Number or some other identifier for the EDD) followed by the table identifier (A1, A2, or A3), and then a ".txt" or ".csv" extension. For example, the EDD file names for a laboratory reporting batch identified as SDG001 that includes instrument calibration data would be as follows.

SDG001A1.txt or SDG001A1.csv
SDG001A2.txt or SDG001A2.csv (A2 file is optional)
SDG001A3.txt or SDG001A3.csv

Analytical Results Table (A1 File)

The Analytical Results table contains analytical results and related information on an analyte level for field samples and associated laboratory quality control samples (excluding calibrations and tunes). Field QC blanks and laboratory method blanks must report a result record for each analyte reported within a method. The method target analyte list is matrix dependent and specified in the project library. Laboratory control samples (LCS and LCSD) and matrix spike samples (MS and MSD) must report a result record for every analyte specified as a spiked analyte in the project library. The project library is a reference table ADR uses for both EDD error checking and automated data review. The project library is populated with information from the project QAPP. Refer to the User Manual for detailed information on project libraries. Table 1 in this document lists all field names and their descriptions for the Analytical Results Table (A1).

Laboratory Instrument Table (A2 File)

The Laboratory Instrument table contains results and related information on an analyte level for instrument initial calibration standards, initial calibration verification standards, continuing calibration standards, and GC/MS tunes. A record must exist for each target analyte reported in a method (specified in the project library), for every calibration type (the field named QCType) associated to samples reported in the EDD. Initial calibrations, initial calibration verifications, and associated samples are linked to each other using a unique Run Batch ID for every distinct initial calibration within a method. Continuing calibrations and associated samples are linked to each other using a unique Analysis Batch ID for every distinct continuing calibration within a method. GC/MS tunes are linked to initial and continuing calibrations (and hence samples) using the Run Batch and Analysis Batch IDs respectively. The Laboratory Instrument Table (A2) is optional. Depending on the level of validation required by the data user, the Laboratory Instrument table may not be requested in the deliverable. Table 2 in this document lists field names and descriptions for the Laboratory Instrument Table (A2).

Sample Analysis Table (A3 File)

The Sample Analysis table contains information on a sample level for field samples and laboratory quality control analyses (excluding calibrations and tunes). A sample record exists for each sample/method/matrix/analysis type combination. Table 3 in this document lists field names and descriptions for the Sample Analysis Table (A3).

EDD Field Properties

Tables 1, 2, and 3 in this document specify the EDD field properties for each file. These include the field name and sequence, field name description, data type and length for each field, and whether or not a particular field requires a standard field. Field elements in the EDD must be sequenced according to the order they appear in Tables 1, 2, and 3. For example, in the Analytical Result table (the A1 file), the field “ClientSampleID” will always be the first piece of information to start a new line of data (or database record), followed by the fields “LabAnalysisRefMethodID”, “AnalysisType”, and so on.

Table 4 in this document lists standard values for those fields that hold standard values. Required field constraints depend on the combination of sample, matrix, method, analyte type, and calibration or QC type information reported in a record. Tables 5 through 9 in this document indicate required fields for each EDD file (table) according to the method category, matrix, analyte type, sample, and QC or calibration type reported in a record.

When creating an EDD as a text file, use the ASCII character set in a file of lines terminated by a carriage return and line feed. No characters are allowed after the carriage return and line feed. Enclose each data set in double quotes (") and separate each field by a comma (comma delimited). Data fields with no information (null) may be represented by two consecutive commas. For example, in the Sample Analysis table, since the “Collected”, “ShippingBatchID”, and “Temperature” fields do not apply to laboratory generated QA/QC samples, the record for a Laboratory Control Sample by Method 8270C would be entered as follows. Note that the first two fields (“ProjectNumber” and “ProjectName”) are omitted in this example.

...“LCSW100598”,,”AQ”,,”LCSW100598”,,”LCS”,,”8270C”,... (and so on)

Do not pad fields with leading or trailing spaces if a field is populated with less than the maximum allowed number of characters. In the above example, although the “MatrixID” field can accommodate up to 10 characters, only 2 characters were entered in this field.

The EDD can be constructed within Excel and saved as .csv file for import into the application. Be sure to format all cells as text beforehand, otherwise Excel will reformat entered values in some cases.

Table 1

Field Descriptions for the Analytical Results Table (A1 file)

Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Standard Value List
ClientSampleID	<p>Client or contractor's identifier for a field sample as reported on the chain-of-custody</p> <p>If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field</p> <p>Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the AnalysisType field is used for this distinction). For example, MW01DL and MW01RE are not allowed</p> <p>Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Analytical Results table for both EDDs.</p>	Text	25	NO
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or a Lab Identifier for a method such as a SOP Number, however; method ID is specified by the project. The method ID must be entered into the standard list.	Text	25	YES (specified in project plan)
AnalysisType	Defines the analysis type (i.e., Dilution, Reanalysis, etc.). This field provides distinction for sample result records when multiple analyses are submitted for the same sample, method, and matrix; for example dilutions, re-analyses, and re-extracts.	Text	10	YES (See Table 4)
LabSampleID	<p>Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD. There are no restrictions for the LabSampleID except for field length and that the LabSampleID must be distinct for a given field sample or lab QC sample and method.</p> <p>Suffixes may be applied to the LabSampleID to designate dilutions, reanalysis, etc.</p>	Text	25	NO
LabID	Identification of the laboratory performing the analyses.	Text	7	NO
ClientAnalyteID	<p>CAS Number or unique client identifier for an analyte or isotope.</p> <p>If a CAS Number is not available, use a unique identifier provided by the client or contractor. The ClientAnalyteID for a particular target analyte or isotope should be specified by the project and must exist in the standard value tables for Analytes.</p> <p>For the LCS, LCSD, MS, and MSD, it is only necessary to report the compounds designated as spikes in the library (and surrogates for organic methods.)</p> <p>For TICs from GC/MS analyses, enter the retention time in decimal minutes as the Client Analyte ID.</p>	Text	12	YES (specified by project)

Table 1

Field Descriptions for the Analytical Results Table (A1 file)

Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Standard Value List
AnalyteName	Chemical name for the analyte or isotope. The project specifies how an analyte or isotope is named. The analyte name must be associated to a ClientAnalyteID in the standard values table for Analytes (excluding compounds designated as TIC's).	Numeric	60	YES (specified by project)
Result	Result value for the analyte or isotope. Entries must be numeric. For non-detects of target analytes or isotopes and spikes, do not enter "ND" or leave this field blank. If an analyte or spike was not detected, enter the reporting limit value corrected for dilution and percent moisture as applicable. Do not enter "0"	Text	10	NO
ResultUnits	The units defining how the values in the Result, DetectionLimit, and ReportingLimit fields are expressed. For radiochemistry this also includes how the value in the Error field is expressed.	Text	10	YES (specified by project in the library)
LabQualifiers	A string of single letter result qualifiers assigned by the lab based on client-defined rules and values. <u>The "U" Lab Qualifier must be entered for all non-detects.</u> Other pertinent lab qualifiers may be entered with the "U" qualifier. Order is insignificant. Lab qualifiers other than those listed in the standard values table may be used. If so, these must be added to the standard value table in the application.	Text	7	YES (See Table 4)
DetectionLimit	For radiochemistry methods, the minimum detectable activity for the isotope being measured. For all other methods: The minimum detection limit value for the analyte being measured.	Numeric	10	NO
DetectionLimitType	Specifies the type of detection limit (i.e., MDA, MDL, IDL, etc.).	Text	10	YES (See Table 4)
RetentionTime or Error	<u>For radiochemistry methods only</u> , enter the 2 Sigma Counting Error. The units for error are entered in the ResultUnits field. <u>For GC/MS methods only</u> , enter the time expressed in decimal minutes between injection and detection for <u>GC/MS TICs only</u> <u>For target analytes in all other methods</u> , leave this field blank. Note: GC retention times are not evaluated at this time.	Text	5	NO
AnalyteType	Defines the type of result, such as tracer, surrogate, spike, or target compound.	Text	7	YES (See Table 4)
PercentRecovery	For radiochemistry methods: The tracer yield, if applicable. For all other analytical methods: The percent recovery value of a spiked compound or surrogate. If the spike or surrogate was not recovered because of dilution, enter "DIL". If a spike or surrogate was not recovered because of matrix interference, enter "INT". If a spike or surrogate was not recovered because it was not added to the sample, enter "NS".	Numeric	5	NO

Table 1

Field Descriptions for the Analytical Results Table (A1 file)

Contains laboratory test results and related information for field and QC samples (excluding instrument calibrations) on an analyte level for environmental chemistry including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Standard Value List
RelativePercentDifference	The relative percent difference (RPD) of two QC results, such as MS/MSD, LCS/LCSD, and Laboratory Duplicates. Report RPD in Laboratory Duplicate, LCSD, and MSD records only.	Numeric	5	NO
ReportingLimit	Reporting limit value for the measured analyte or isotope Factor in the dilution factor and percent moisture correction, if applicable. The Reporting Limit for each analyte and matrix in a given method is specified in the project library or QAPP.	Numeric	10	NO
ReportingLimitType	Specifies the type of reporting limit (i.e., CRQL, PQL, SQL, RDL, etc). The Reporting Limit Type for each method and matrix is specified in the project library or QAPP.	Text	10	YES (specified by the project)
ReportableResult	<p>This field indicates whether or not the laboratory chooses an individual analyte or isotope result as reportable. Enter "YES" if the result is reportable. Enter "NO" if the result is not reportable. This field applies to target analytes only.</p> <p>If only one analysis is submitted for a particular sample and method, enter "YES" for all target compounds (where Analyte Type = TRG). For GC/MS methods enter yes for tentatively identified compounds (where Analyte Type = TIC).</p> <p>If two or more analyses are submitted for a particular sample and method (i.e. initial analysis, reanalysis and/or dilutions), enter "YES" from only <u>one</u> of the analyses for each target compound. For example: a sample was run a second time at dilution because benzene exceeded the calibration range in the initial, undiluted analysis. All target analytes are reported in each analysis. For the initial analysis, (Analysis Type = RES), enter "NO" for benzene and enter "YES" for all other compounds. For the diluted analysis (Analysis Type = DL), enter "YES" for benzene and enter "NO" for all other compounds.</p> <p>For TICs (Analyte Type = TIC), if more than one analysis is submitted for a particular sample and method, choose only one of the analyses where Reportable Result = YES for <u>all</u> TICs. For example, a sample was run a second time because one or more target compounds exceeded the calibration range in the undiluted analysis. Choose a particular analysis and enter "YES" for all TICs. In the other analysis enter "NO" for all TICs.</p> <p>Note that it is not necessary to report the full target analyte list for the initial result, dilution, re-analysis, or re-extraction. However, each target analyte must be reported YES once and once only in the case of multiple analyses for a given sample, method, and matrix. In the case of organics, all surrogates must be reported for all analyses submitted for a given sample, method, and, matrix.</p>	Text	3	YES (See Table 4)

Table 2

Field Descriptions for the Laboratory Instrument Table (A2 file)

Contains related to laboratory instrument calibration on an analyte level and GC/MS Tune information. This table is optional depending on project requirements. Do not report Table A2 for radiochemistry methods.

Field Name	Field Name Description	Field Type	Field Length	Standard Value List
InstrumentID	Laboratory instrument identification.	Text	15	NO
QCType	Type of instrument QC (i.e., Instrument_Performance_Check or type of calibration standard).	Text	10	YES (See Table 4)
Analyzed	Analysis date/time for BFB, DFTPP, initial calibration verification standards, calibration verification standards, and continuing calibration standards. For the <u>initial calibration</u> , enter date and time of the <u>last</u> standard analyzed. Also, see comments about initial calibrations in the Alternate_Lab_Analysis_ID field name description.	Date/Time	*	NO
AlternateLab_AnalysisID	Common laboratory identification used for standards (i.e., VOA STD50, CCAL100, BFB50, etc). For initial calibration, enter ICAL. Information from the initial calibration is entered as one record for each analyte that summarizes the results of the initial calibration (i.e. %RSD, correlation coefficient, and avg RF). Records are <u>not</u> entered for each individual standard within the initial calibration.	Text	12	NO
LabAnalysisID	Unique identification of the raw data electronic file associated with the calibration standard or tune (i.e., 9812101MS.DV). Leave this field blank for the initial calibration. See comments about initial calibrations in the Alternate_Lab_Analysis_ID field description. This field is only applicable where an electronic instrument file is created as part of the analysis.	Text	15	NO
LabAnalysisRefMethodID	Laboratory reference method ID (i.e., 8260B, 8270C, 6010B, etc.). The method ID is specified by the project. The LabAnalysisRefMethodID must be in the standard value list for Method IDs.	Text	25	YES (specified by the project)
ClientAnalyteID	CAS number or unique client identifier for an analyte. If a CAS number is not available, use a unique identifier provided by the client. The unique identifier for a particular analyte should be specified by the project and must exist in the standard value list for ClientAnalyteID. Records for each calibration must report the full target analyte list including surrogates as applicable. The target analyte list is specified for each method and matrix in the project	Text	12	YES (specified by the project)
AnalyteName	The chemical name for the analyte. The project specifies how an analyte is named. The AnalyteName must be associated to a ClientAnalyteID in the standard values.	Text	60	YES (specified by the project)

Table 2

Field Descriptions for the Laboratory Instrument Table (A2 file)

Contains related to laboratory instrument calibration on an analyte level and GC/MS Tune information. This table is optional depending on project requirements. Do not report Table A2 for radiochemistry methods.

Field Name	Field Name Description	Field Type	Field Length	Standard Value List
RunBatch	Unique identifier for a batch of analyses performed on one instrument under the control of one initial calibration and initial calibration verification. The Run Batch ID links both the initial calibration and initial calibration verification to subsequently analyzed and associated continuing calibrations, field samples, and QC analyses. For GC/MS methods, the Run_Batch ID also links a BFB or DFTPP tune and the initial calibration and initial calibration verification standards to associated samples and method QC analyses. A new and unique Run Batch ID must be used with every new initial calibration.	Text	12	NO
AnalysisBatch	<p>Unique laboratory identifier for a batch of analyses performed on one instrument and under the control of a continuing calibration or continuing calibration verification. The Analysis Batch ID links the continuing calibration or calibration verification to subsequently analyzed and associated field sample and QC analyses. For GC/MS methods, the Analysis Batch ID also links the BFB or DFTPP tune. A new and unique Analysis Batch ID must be used with every new continuing calibration or continuing calibration verification.</p> <p>For GC methods, only report opening standards, do not include closing standards (unless the closing standard functions as the opening standard for a subsequent set of analyses, in which case a new and unique Analysis Batch ID is assigned).</p> <p>When dual or confirmation columns/detectors are used, enter results from the primary column/detector only (this is similar to CLP Pesticide reporting).</p>	Text	12	NO
LabReportingBatch	Unique laboratory identifier for a batch of samples including associated calibrations and method QC, reported as a group by the lab (i.e., lab work order #, log-in #, or SDG). Links all instrument calibrations, samples, and method QC reported as a group or SDG.	Text	12	NO
PercentRelativeStandard Deviation	<p>The standard deviation relative to the mean used to evaluate initial calibration linearity. Organic methods may use either %RSD or Correlation Coefficient.</p> <p>If applicable, enter the %RSD. Leave this field blank if the Correlation Coefficient is used.</p>	Numeric	5	NO
CorrelationCoefficient	<p>The correlation coefficient resulting from linear regression of the initial calibration. For metals by ICAP, enter '1.0' if a two-point initial calibration was analyzed. Organic methods may use either %RSD or Correlation Coefficient.</p> <p>If applicable, enter the Correlation Coefficient. Leave this field blank if the %RSD is used</p>	Numeric	5	NO
RelativeResponseFactor	<p>This field applies to GC/MS only.</p> <p>For continuing calibration enter the relative response factor.</p> <p>For initial calibration enter the <u>average</u> relative response factor. Refer to comments about initial calibration records in the field description for Alternate Lab Analysis ID.</p>	Numeric	5	NO

Table 2

Field Descriptions for the Laboratory Instrument Table (A2 file)

Contains related to laboratory instrument calibration on an analyte level and GC/MS Tune information. This table is optional depending on project requirements. Do not report Table A2 for radiochemistry methods.

Field Name	Field Name Description	Field Type	Field Length	Standard Value List
Percent_Difference (or Percent Recovery)	<p>For <u>organic methods</u>, this field is the difference between 2 measured values expressed as a percentage.</p> <p>If %RSD is reported, enter the % difference between the average response factor of the initial calibration (IC) and the response factor of the initial calibration verification (ICV) or continuing calibration (CCV).</p> <p>If correlation coefficient is used, enter the % difference between the true value and the measured value.</p> <p>The Percent_Difference is expressed as a negative or positive value. Do not express Percent_Difference as an absolute value. Use a negative value if the CCV or ICV response factor is less than the IC average response factor or, in the case of correlation coefficient, the CCV or ICV measured value is less than the true value. Use a positive value if the CCV or ICV response factor is greater than the IC average response factor, or in the case of correlation coefficient, the CCV or ICV measured value is greater than the true value.</p> <p>For <u>inorganic methods</u>, this field is the recovery of an analyte expressed relative to the true amount (i.e., %R for a metal in the continuing calibration or initial calibration verification by Method 6010B).</p>	Numeric	5	NO
PeakID01	Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 50, for DFTPP enter 51.	Numeric	10	NO
PercentRatio01	<p>For BFB enter the relative percent abundance of m/z 50 measured relative to the raw abundance of m/z 95.</p> <p>For DFTPP enter the relative percent abundance of m/z 51 measured relative to the raw abundance of m/z 198.</p>	Numeric	10	NO
PeakID02	Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 75, for DFTPP enter 68.	Numeric	10	NO
PercentRatio02	<p>For BFB enter the relative percent abundance of m/z 75 measured relative to the raw abundance of m/z 95.</p> <p>For DFTPP enter the relative percent abundance of m/z 68 measured relative to the raw abundance of m/z 69.</p>	Numeric	10	NO
PeakID03	Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 95, for DFTPP enter 69.	Numeric	10	NO
PercentRatio03	<p>For BFB enter the ion abundance of m/z 95 as 100 percent.</p> <p>For DFTPP enter the relative percent abundance of m/z 69 measured relative to the raw abundance of m/z 198.</p>	Numeric	10	NO
PeakID04	Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 96, for DFTPP enter 70.	Numeric	10	NO

Table 2**Field Descriptions for the Laboratory Instrument Table (A2 file)**

Contains related to laboratory instrument calibration on an analyte level and GC/MS Tune information. This table is optional depending on project requirements. Do not report Table A2 for radiochemistry methods.

Field Name	Field Name Description	Field Type	Field Length	Standard Value List
PercentRatio04	For BFB enter the relative percent abundance of m/z 96 measured relative to the raw abundance of m/z 95. For DFTPP enter the relative percent abundance of m/z 70 measured relative to the raw abundance of m/z 69	Numeric	10	NO
PeakID05	Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 173, for DFTPP enter 127.	Numeric	10	NO
PercentRatio05	For BFB enter the relative percent abundance of m/z 173 measured relative to the raw abundance of m/z 174. For DFTPP enter the relative percent abundance of m/z 127 measured relative to the raw abundance of m/z 198	Numeric	10	NO
PeakID06	Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 174, for DFTPP enter 197.	Numeric	10	NO
PercentRatio06	For BFB enter the relative percent abundance of m/z 174 measured relative to the raw abundance of m/z 95. For DFTPP enter the relative percent abundance of m/z 197 measured relative to the raw abundance of m/z 198.	Numeric	10	NO
PeakID07	Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 175, for DFTPP enter 198.	Numeric	10	NO
PercentRatio07	For BFB enter the relative percent abundance of m/z 175 measured relative to the raw abundance of m/z 174. For DFTPP enter the ion abundance of m/z 198 as 100 percent.	Numeric	10	NO
PeakID08	Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 176, for DFTPP enter 199.	Numeric	10	NO
PercentRatio08	For BFB enter the relative percent abundance of m/z 176 measured relative to the raw abundance of m/z 174. For DFTPP enter the relative percent abundance of m/z 199 measured relative to the raw abundance of m/z 198.	Numeric	10	NO
PeakID09	Identifies individual m/z ions for GC/MS tuning compounds. For BFB enter 177, for DFTPP enter 275.	Numeric	10	NO
PercentRatio09	For BFB enter the relative percent abundance of m/z 177 measured relative to the raw abundance of m/z 176. For DFTPP enter the relative percent abundance of m/z 275 measured relative to the raw abundance of m/z 198.	Numeric	10	NO
PeakID10	Identifies individual m/z ions for GC/MS tuning compounds. For BFB leave blank, for DFTPP enter 365.	Numeric	10	NO

Table 2

Field Descriptions for the Laboratory Instrument Table (A2 file)

Contains related to laboratory instrument calibration on an analyte level and GC/MS Tune information. This table is optional depending on project requirements. Do not report Table A2 for radiochemistry methods.

Field Name	Field Name Description	Field Type	Field Length	Standard Value List
PercentRatio10	For BFB leave blank. For DFTPP enter the relative percent abundance of m/z 365 measured relative to the raw abundance of m/z 198.	Numeric	10	NO
PeakID11	Identifies individual m/z ions for GC/MS tuning compounds. For BFB leave blank, for DFTPP enter 441.	Numeric	10	NO
PercentRatio11	For BFB leave blank. For DFTPP the percent abundance of m/z 441 measured relative to the raw abundance of m/z 443	Numeric	10	NO
PeakID12	Identifies individual m/z ions for GC/MS tuning compounds. For BFB leave blank, for DFTPP enter 442.	Numeric	10	NO
PercentRatio12	For BFB leave blank. For DFTPP enter the relative percent abundance of m/z 442 measured relative to the raw abundance of m/z 198.	Numeric	10	NO
PeakID13	Identifies individual m/z ions for GC/MS tuning compounds. For BFB leave blank, for DFTPP enter 443.	Numeric	10	NO
PercentRatio13	For BFB leave blank. For DFTPP enter the relative percent abundance of m/z 443 measured relative to the raw abundance of m/z 442.	Numeric	10	NO

* Date/time format is: MM/DD/YYYY hh:mm where MM = month, DD = day, YYYY = four digits of the year, hh = hour in 24 hour format, and mm = minutes.

Table 3
Field Description for the Sample Analysis (A3 file)

This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Standard Value List
ProjectNumber	Project number assigned by the client.	Text	30	YES (specified by project)
ProjectName	Project name assigned by the client.	Text	90	YES (specified by project)
ClientSampleID	<p>Client or contractor's identifier for a field sample</p> <p>If a sample is analyzed as a laboratory duplicate, matrix spike, or matrix spike duplicate, append suffixes DUP, MS and MSD respectively to the Client Sample ID with no intervening spaces or hyphens (i.e. MW01DUP, MW01MS, and MW01MSD). For Method Blanks, LCS, and LCSD enter the unique LaboratorySampleID into this field</p> <p>Do not append suffixes to the ClientSampleID for dilutions, reanalyses, or re-extracts (the Analysis_Type field is used for this distinction). For example, MW01<u>DL</u> and MW01<u>RE</u> are not allowed</p> <p>Parent sample records must exist for each MS and MSD. If an MS/MSD is shared between two EDDs, records for the MS/MSD and its parent sample must exist in the Sample Analysis table for both EDDs.</p>	Text	25	NO
Collected	<p><u>For radiochemistry methods</u> the Date of sample collection. Refer to the date format for radiochemistry methods at the end of this table.</p> <p><u>For all other methods</u> the Date and Time of sample collection. Refer to the date/time format at the end of this table.</p> <p>Leave this field blank for Method Blank, LCS, and LCSD</p>	Date/Time	16*	NO
MatrixID	Sample matrix (i.e., AQ, SO, etc.)	Text	10	YES (See Table 4)
LabSampleID	<p>Laboratory tracking number for field samples and lab generated QC samples such as method blank, LCS, and LCSD.</p> <p>There are no restrictions for the LabSampleID except field length and that the LabSampleID must be unique for a given field sample or lab QC sample and method.</p>	Text	25	NO
QCType	This record identifies the type of quality control sample QC (i.e., Duplicate, LCS, Method Blank, MS, or MSD). <u>For regular samples, leave this field blank.</u>	Text	10	YES (See Table 4)
ShippingBatchID	Unique identifier assigned to a cooler or shipping container used to transport client or field samples. Links all samples to a cooler or shipping container. No entry for method blanks, LCS, and LCSD. This field is optional.	Text	25	NO
Temperature	<p>Temperature (in centigrade degrees) of the sample as received.</p> <p><u>This field is not required for radiochemistry methods.</u></p>	Numeric	10	NO

Table 3
Field Description for the Sample Analysis (A3 file)

This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Standard Value List
LabAnalysisRefMethodID	Laboratory reference method ID. The method ID may be an EPA Method number or laboratory identifier for a method such as a SOP number, however; values used for Laboratory Method IDs are specified by the project and must in the in standard value list for method IDs.	Text	25	YES (Specified by the project)
PreparationType	Preparation Method Number (i.e., 3010A, 3510C, 3550C, 5030B, etc.) For analytical procedures that do not have a specific preparation method number, use "Gen Prep".	Text	25	YES (See Table 4)
AnalysisType	Defines the type of analysis such as initial analysis, dilution, re-analysis, etc. This field provides distinction for sample records when multiple analyses are submitted for the same sample, method, and matrix, for example: dilutions, re-analyses, and re-extracts.	Text	10	YES (See Table 4)
Prepared	<u>For radiochemistry leave this field blank.</u> For all other methods enter the date and time of sample preparation or extraction. Refer to the date/time format at the end of this table.	Date/Time	16*	NO
Analyzed	<u>For radiochemistry methods</u> the date of sample analysis. Refer to the date format for radiochemistry methods at the end of this table. <u>For all other methods</u> the date and time of sample analysis. Refer to the date and time format at the end of this table.	Date/Time	*	NO
LabID	Identification of the laboratory performing the analysis.	Text	7	NO
QCLevel	The level of laboratory QC associated with the analysis reported in the EDD. If only the Analytical Results Table (A1) and the Sample Analysis Table (A3) information are submitted for the sample, enter "COA". If the Laboratory Instrument Table (A2) information is also submitted for the sample, enter "COCAL"	Text	6	YES (See Table 4)
ResultBasis	Indicates whether results associated with this sample records are reported as wet or percent moisture corrected. This field is only required for soils and sediments. Enter "WET" if results are not corrected for percent moisture. Enter "DRY" if percent moisture correction is applied to results.	Text	3	YES (See Table 4)
TotalOrDissolved	This field indicates if the results related to this sample record are reported as a total or dissolved fraction. This field is only required for metal methods. For all other methods leave this field blank.	Text	3	YES (See Table 4)
Dilution	Dilution of the sample aliquot. Enter "1" for method blanks, LCS, and LCSD, or if the field samples was analyzed without dilution.	Numeric	10	NO
HandlingType	Indicates the type of leaching procedure, if applicable (i.e., SPLP, TCLP, WET). Leave this field blank if the sample analysis was <u>not</u> performed on a leachate.	Text	10	YES (See Table 4)

Table 3
Field Description for the Sample Analysis (A3 file)

This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Standard Value List
HandlingBatch	<p>Unique laboratory identifier for a batch of samples prepared together in a leaching procedure (i.e., SPLP, TCLP, or WET preparation). The HandlingBatch links samples with leaching blanks.</p> <p>Leave this field blank if the sample analysis was <u>not</u> performed on a leachate</p>	Text	12	NO
LeachateDate	<p>Date and time of leaching procedure (i.e., date for SPLP, TCLP, or WET preparation). Refer to the date and time format at the end of this table.</p> <p>Leave this field blank if the sample analysis was <u>not</u> performed on a leachate</p>	Date /Time	16*	NO
Percent_Moisture	Percent of sample composed of water. Enter for soil and sediment samples only.	Numeric	10	NO
MethodBatch	<p>Unique laboratory identifier for a batch of samples of similar matrices analyzed by one method and treated as a group for matrix spike, matrix spike duplicate, or laboratory duplicate association</p> <p>The method batch links the matrix spike and/or matrix spike duplicate or laboratory duplicates to associated samples. Note, the MethodBatch association may coincide with the PreparationBatch association. The MethodBatch is specifically used to link the MS/MSD and/or DUP to associated samples.</p>	Text	12	NO
PreparationBatch	<p>Unique laboratory identifier for a batch of samples prepared together for analysis by one method and treated as a group for method blank, LCS and LCSD association.</p> <p>The PreparationBatch links method blanks and laboratory control samples (blank spikes) to associated samples. Note, the PreparationBatch association may coincide with the MethodBatch association but the PreparationBatch specifically links the Method Blank and LCS to associated samples.</p>	Text	12	NO
RunBatch	<p><u>For radiochemistry methods leave this field blank.</u></p> <p><u>For all other methods</u> the RunBatch is the unique identifier for a batch of analyses performed on one instrument under the control of one initial calibration and initial calibration verification. The RunBatch links both the initial calibration and initial calibration verification to subsequently analyzed and associated continuing calibrations, field samples, and QC analyses. For GC/MS methods, the RunBatch also links a BFB or DFTPP tune. A distinct RunBatch must used with every new initial calibration within a method</p> <p>The value entered in this field links a particular sample/method/analysis type record to a set of associated initial calibration and initial calibration verification records from Table A2.</p> <p>This field is only required if the A2 table is included with the EDD.</p>	Text	12	NO

Table 3
Field Description for the Sample Analysis (A3 file)

This table contains information related to analyses of field samples and laboratory QC samples (excluding calibrations and tunes) on a sample level for environmental chemical analyses including radiochemistry

Field Name	Field Name Description	Field Type	Field Length	Standard Value List
AnalysisBatch	<p><u>For radiochemistry methods</u> leave this field blank.</p> <p><u>For all other methods</u> the AnalysisBatch is the unique identifier for a batch of analyses performed on one instrument and under the control of a continuing calibration or continuing calibration verification. The AnalysisBatch links the continuing calibration or calibration verification to subsequently analyzed and associated field sample and QC analyses. For GC/MS methods, the AnalysisBatch also links the BFB or DFTPP tune. A distinct AnalysisBatch must be used with every new continuing calibration or continuing calibration verification within a method</p> <p>The value entered in this field links a particular sample/method/analysis type record to a set of associated continuing calibration records in the Laboratory Instrument table.</p> <p>This field is only required if the A2 table is included with the EDD.</p>	Text	12	NO
LabReportingBatch	Unique laboratory identifier for the EDD. This is equivalent to the sample delivery group, lab work number, login ID, etc. The LabReportingBatch links all records in the EDD reported as one group. The value entered in this field must be the same in all records.	Text	12	NO
LabReceipt	Date and time the sample was received in the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/Time	16*	
LabReported	Date and time hard copy reported delivered by the lab. A time value of 00:00 may be entered. Refer to the date/time format at the end of this table.	Date/Time	16*	

* For radiochemistry methods format Date as MM/DD/YYYY (where MM = two digit month, DD = two digit day, and YYYY = four digit year)

For all other methods format Date and Time as MM/DD/YYYY hh:mm YYYY (where MM = two digit month, DD = two digit day, and YYYY = four digit year, hh = hour in 24 hour format, and mm = minutes)

Table 4
Standard Value List

Field Name	Standard Value	Standard Value Description
Analysis_Type	DL	Dilution of the original sample
	DL2	Second dilution of the original sample
	DL3	Third dilution of the original sample
	DL4	Fourth dilution of the original sample
	RE	Reanalysis/re-extraction of sample
	RE2	Second reanalysis/re-extraction of sample
	RE3	Third reanalysis/re-extraction of sample
	RE4	Fourth reanalysis/re-extraction of the original sample
	RES	The initial or original sample.
Analyte_Name	Refer to QAPP and Project Library	Analyte names are specified by the project and entered into the library for each method and matrix. Analyte Names used in project libraries must first exist in the standard value table. The same holds true for the ClientAnalyteID
Analyte_Type	IS	Internal standard as defined per CLP usage
	SPK	Spiked analyte
	SURR	Surrogate as defined as per CLP usage
	TIC	Tentatively identified compound for GC/MS analysis
	TRG	Target compound
Detection_Limit_Type ¹	CRDL	Contract required detection limit
	IDL	Instrument detection limit
	MDA	Minimum detectable activity
	MDL	Method detection limit
Handling_Type ²	WET	Wet leaching procedure
	SPLP	Synthetic Precipitation Leaching Procedure
	TCLP	Toxicity Characteristic Leaching Procedure
Lab_Analysis_Ref_Method_ID	Refer to QAPP and Project Library	Method IDs are specified by the project and entered into the library. Methods used in project libraries must first exist in the standard value table
Lab_Qualifiers ³	*	INORG: Duplicate analysis was not within control limits
	*	ORG: Surrogate values outside of contract required QC limits
	+	INORG: Correlation coefficient for the method of standard additions (MSA) was less than 0.995
	A	ORG: Tentatively identified compound (TIC) was a suspected aldol-condensation product
	B	INORG: Value less than contract required detection limit, but greater than or equal to instrument detection limit
	B	ORG: Compound is found in the associated blank as well as in the sample
	C	ORG: Analyte presence confirmed by GC/MS
	D	Result from an analysis at a secondary dilution factor
	E	INORG: Reported value was estimated because of the presence of interference
	E	ORG: Concentrations exceed the calibration range of the instrument
	H	Analysis performed outside method or client-specified holding time requirement
	J	Estimated value
	M	INORG: Duplicate injection precision was not met
	N	INORG: Spiked sample recovery was not within control limits
	N	ORG: Presumptive evidence of a compound
	P	ORG: Difference between results from two GC columns unacceptable (>25% Difference)
	S	Reported value was determined by the method of standard additions (MSA)
	U	Compound was analyzed for, but not detected. Analyte result was below the Reporting Limit.
	W	INORG: Post digestion spike was out of control limits
X	Reserved for a lab-defined data qualifier	
Y	Reserved for a lab-defined data qualifier	
Z	Reserved for a lab-defined data qualifier	
Matrix_ID	AIR	Air
	AQ	Water
	ASH	Ash

Table 4
Standard Value List

Field Name	Standard Value	Standard Value Description
Matrix_ID (continued)	BIOTA	Biological matter
	FILTER	Filter
	LIQUID	Non-aqueous liquid
	OIL	Oil
	SED	Sediment
	SLUDGE	Sludge
	SO	Soil
	SOLID	Non-soil/sediment solid
	TISSUE	Tissue
	WASTE	Waste
	WIPE	Wipe
Preparation_Type ⁴	3005A	Acid Digestion of Waters for Total Recoverable or Dissolved Metals by FLAA or ICP
	3010A	Acid of Aqueous Samples and Extracts for Total Metals by FLAA or ICP
	3015	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts
	3020A	Acid Digestion of Aqueous Samples and Extracts for Total Metals by GFAA
	3031	Acid Digestion of Oils for Metals Analysis by AA or ICP
	3050B	Acid Digestion of Sediments, Sludges, and Soils
	3051	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils and Oils
	3052	Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices
	3060A	Alkaline Digestion for Hexavalent Chromium
	3510C	Separatory Funnel Liquid-Liquid Extraction
	3520C	Continuous Liquid-Liquid Extraction
	3535	Solid Phase Extraction
	3540C	Soxhlet Extraction
	3541	Automated Soxhlet Extraction
	3545	Pressurized Fluid Extraction
	3550B	Ultrasonic Extraction
	3560	Supercritical Fluid Extraction of Total Recoverable Petroleum Hydrocarbons
	5030B	Purge and Trap for Aqueous Samples
	5035	Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples
	7470A	Acid digestion of waters for Mercury analysis
	7471A	Acid digestion of soils and solids for Mercury analysis
	Gen Prep	Generic preparation type when a preparation method ID does not exist (used mostly for general chemistry methods)
QC_Level	COA	Certificate of Analysis (accuracy and precision, no calibration)
	COACAL	Certificate of Analysis (accuracy and precision including calibration)
QC_Type	MB	Analytical control consisting of all reagents and standards that is carried through the entire procedure (Method Blank)
	CV	(Calibration Verification) Analytical standard run at a specified frequency to verify the calibration of the analytical system
	CCV	(Continuing Calibration Verification) Analytical standard run every 12 hours to verify the calibration of the GC/MS system
	DUP	A second aliquot of a sample that is treated the same as the original aliquot to determine the precision of the method
	IC	(Initial Calibration) Analysis of analytical standards for a series of different specified concentrations
	ICV	(Initial Calibration Verification) Analytical standard run at a specified frequency to verify the accuracy of the initial calibration of the analytical system
	IPC	(Instrument Performance Check) Analysis of DFTPP or BFB to evaluate the performance of the GC/MS system
	LCS	(Laboratory Control Sample) A control sample of known composition
	LCSD	(Laboratory Control Sample Duplicate) A duplicate control sample of known composition
	MS	(Matrix Spike) Aliquot of a matrix spiked with known quantities and subjected to the entire analytical procedure to measure recovery
	MSD	(Matrix Spike Duplicate) A second aliquot of the same matrix as the matrix spike that is spiked in order to determine the precision of the method
Reporting_Limit_Type ¹	CRDL	Contract- required detection limit
	CRQL	Contract- required quantitation limit

Table 4
Standard Value List

Field Name	Standard Value	Standard Value Description
Reporting_Limit_Type (continued)	PQL	Practical quantitation limit
	SQL	Sample quantitation limit
	RDL	Reportable detection limit
Result_Basis	DRY	Result was calculated on a dry weight basis
	WET	Result was calculated on a wet weight basis
Result_Units ⁵	ug/L	Micrograms per liter
	mg/L	Milligrams per liter
	ug/Kg	Micrograms per kilogram
	mg/Kg	Milligrams per kilogram
	pg/L	Picograms per liter
	ng/Kg	Nanograms per kilogram
Total_Or_Dissolved	DIS	Dissolved
	TOT	Total

- 1 Additional Detection Limit Types and Reporting Limit Types may be used. These must be added to the application standard values.
- 2 Additional Handling Types (leachate procedures) may be used. These must be added to the application standard values
- 3 Additional Lab Qualifiers may be used, or listed Lab Qualifiers may be used in a different manner than described in this table. New lab qualifiers must be added to the application standard value tables. NOTE: The “U” Lab Qualifier must be used for all non-detects.
- 4 Additional Preparation Types may be used. These must be added to the application standard value tables.
- 5 Additional Result Units may be used. The project library specifies the reporting limit used for each method and matrix

Note: if new standard values are used then these standard values must be entered in the software standard values for both the lab and contractor. The application will automatically update the standard values tables if an importing library contains standard values (method, client analyte ID, and analyte name) that do not exist in the software importing the new library.

Table 5

Required Fields in the Analytical Results Table for GC/MS, GC, and HPLC Methods

Field	GC/MS Methods			GC and HPLC Methods		
	Regular Sample*	MS/MSD	Method Blank, LCS/LCSD	Regular Sample*	MS/MSD	Method Blank, LCS/LCSD
Client_Sample_ID	X	X	X	X	X	X
Lab_Analysis_Ref_Method_ID	X	X	X	X	X	X
Analysis_Type	X	X	X	X	X	X
Lab_Sample_ID	X	X	X	X	X	X
Lab_ID	X	X	X	X	X	X
Client_Analyte_ID	X	X	X	X	X	X
Analyte_Name	X	X	X	X	X	X
Result	X	X	X	X	X	X
Result_Units	X	X	X	X	X	X
Lab_Qualifiers	Q	Q	Q	Q	Q	Q
Detection Limit	X	X	X	X	X	X
Detection_Limit_Type	X	X	X	X	X	X
Retention_Time	T		T			
Analyte_Type	X	X	X	X	X	X
Percent_Recovery	S	R	R	S	R	R
Relative_Percent_Difference		D	D		D	D
Reporting_Limit	X	X	X	X	X	X
Reporting_Limit_Type	X	X	X	X	X	X
Reportable_Result	X	X	X	X	X	X

Key

- X Required Field
- D Required field for spiked compounds in the LCSD and MSD only
- Q Required field if laboratory has qualified result. The “U” qualifier MUST be entered if the result is non-detect.
- R Required field if Analyte_Type = “SPK” or “SURR”
- S Required field for surrogate compounds only
- T Required field for tentatively identified compounds by GC/MS only
- * Also includes Equipment Blanks, Field Blanks, and Trip Blanks

Table 6
Required Fields in the Analytical Results Table for ICAP, AA, and IC Methods

Field	ICAP and AA Methods			IC and Wet Chemistry Methods		
	Regular Sample*	Sample Duplicate, MS/MSD	Method Blank, LCS/LCSD	Regular Sample*	Sample Duplicate MS/MSD	Method Blank, LCS/LCSD
Client_Sample_ID	X	X	X	X	X	X
Lab_Analysis_Ref_Method_ID	X	X	X	X	X	X
Analysis_Type	X	X	X	X	X	X
Lab_Sample_ID	X	X	X	X	X	X
Lab_ID	X	X	X	X	X	X
Client_Analyte_ID	X	X	X	X	X	X
Analyte_Name	X	X	X	X	X	X
Result	X	X	X	X	X	X
Result_Units	X	X	X	X	X	X
Lab_Qualifiers	Q	Q	Q	Q	Q	Q
Detection_Limit	X	X	X	X	X	X
Detection_Limit_Type	X	X	X	X	X	X
Retention_Time						
Analyte_Type	X	X	X	X	X	X
Percent_Recovery		S	S		S	S
Relative_Percent_Difference		R	R		R	R
Reporting_Limit	X	X	X	X	X	X
Reporting_Limit_Type	X	X	X	X	X	X
Reportable_Result	X	X	X	X	X	X

Key

- X Required field
- Q Required field if laboratory has qualified result. The “U” qualifier MUST be entered if the result is non-detect
- R Required field for spiked compounds in LCSD or MSD, or target compounds in the Sample Duplicate only
- S Required field if Analyte_Type = “SPK”
- * Also includes Trip Blanks, Equipment Blanks, and Field Blanks

Table 7
Required Fields in the Laboratory Instrument Table

Field	GC/MS Tunes		Initial Calibration				Initial Calibration Verification				Calibration Verification, Continuing Calibration
	VOA	SVOA	GC/MS	GC HPLC	ICP/AA	IC*	GC/MS	GC HPLC	ICP/AA	IC*	ALL METHODS
Instrument_ID	X	X	X	X	X	X	X	X	X	X	X
QC_Type	X	X	X	X	X	X	X	X	X	X	X
Analyzed	X	X	X	X	X	X	X	X	X	X	X
Alternate_Lab_Analysis_ID	X	X	X	X	X	X	X	X	X	X	X
Lab_Analysis_ID	X	X					X	X	X	X	X
Lab_Analysis_Ref_Method_ID	X	X	X	X	X	X	X	X	X	X	X
Client_Analyte_ID	X	X	X	X	X	X	X	X	X	X	X
Analyte_Name	X	X	X	X	X	X	X	X	X	X	X
Run_Batch	X	X	X	X	X	X	X	X	X	X	X
Analysis_Batch	C	C									X
Lab_Reporting_Batch	X	X	X	X	X	X	X	X	X	X	X
Percent_Relative_Standard_Deviation			X	X							
Correlation_Coefficient			B	B	X	X					
Relative_Response_Factor			X				X				M
Percent_Difference							X	X	X	X	X
Peak_ID_01	X	X									
Percent_Ratio_01	X	X									
Peak_ID_02	X	X									
Percent_Ratio_02	X	X									
Peak_ID_03	X	X									
Percent_Ratio_03	X	X									
Peak_ID_04	X	X									
Percent_Ratio_04	X	X									
Peak_ID_05	X	X									
Percent_Ratio_05	X	X									
Peak_ID_06	X	X									
Percent_Ratio_06	X	X									
Peak_ID_07	X	X									
Percent_Ratio_07	X	X									
Peak_ID_08	X	X									
Percent_Ratio_08	X	X									
Peak_ID_09	X	X									
Percent_Ratio_09	X	X									
Peak_ID_10		X									
Percent_Ratio_10		X									
Peak_ID_11		X									
Percent_Ratio_11		X									
Peak_ID_12		X									
Percent_Ratio_12		X									
Peak_ID_13		X									
Percent_Ratio_13		X									

Key

- X Required field (some fields are not applicable to some General (Wet) Chemistry tests)
- B Required field if reporting best fit
- C Required field if BFB or DFTPP associated with a continuing calibration only
- M Required field for GC/MS continuing calibration only

*IC Includes Ion Chromatography and Classical or Wet Chemistry methods. Methods such as pH, Conductivity, and others do not use traditional calibration procedures, ; therefore, some fields marked as a required field under the "IC" column do not apply for these methods.

Table 8
Required Fields in the Sample Analysis Table

Field	GC, GC/MS, HPLC Methods		ICAP and AA Methods		IC and Wet Chemistry Methods	
	Method Blanks, LCS/LCSD	Regular Samples*, Sample Duplicate, MS/MSD	Method Blanks, LCS/LCSD	Regular Samples*, Sample Duplicate, MS/MSD	Method Blanks, LCS/LCSD	Regular Samples*, Sample Duplicate, MS/MSD
Client_Sample_ID	X	X	X	X	X	X
Collected		X		X		X
Matrix_ID	X	X	X	X	X	X
Lab_Sample_ID	X	X	X	X	X	X
QC_Type	X	Q	X	Q	X	X
Shipping_Batch_ID		X		X		X
Temperature		X				X
Lab_Analysis_Ref_Method_ID	X	X	X	X	X	X
Preparation_Type	X	X	X	X	X	X
Analysis_Type	X	X	X	X	X	X
Prepared	A	A	X	X	N	N
Analyzed	X	X	X	X	X	X
Lab_ID	X	X	X	X	X	X
QC_Level	X	X	X	X	X	X
Results_Basis		S		S		S
Total_Or_Dissolved			W	W		
Dilution	X	X	X	X	X	X
Handling_Type	L	L	L	L	L	L
Handling_Batch	L	L	L	L	L	L
Leachate_Date	L	L	L	L	L	L
Percent Moisture		S		S		S
Method_Batch	X	X	X	X	X	X
Preparation_Batch	X	X	X	X	X	X
Run_Batch	C	C	C	C	C	C
Analysis_Batch	C	C	C	C	C	C
Lab_Reporting_Batch	X	X	X	X	X	X
Lab_Receipt		X		X		X
Lab_Reported	X	X	X	X	X	X

Key

- X Required field
- A Required field for samples prepared by methanol extraction
- C Required field if Instrument Calibration Table (A2) is included in EDD
- L Required field if analysis performed on SPLP, TCLP, or WET extracts
- N Required field only for samples that require preparation before analysis
- Q Required field for Sample Duplicate, MS, and MSD only
- S Required field if "Matrix_ID" = "SO" or "SED"
- W Required field for aqueous samples only
- * Includes Trip Blanks, Equipment Blanks, and Field Blanks

Instructions For EDF File Specifications

1. The upload EDF function will allow you to upload Lab data for one or more sites.
2. To create a valid EDF upload you must create several documents and zip them up into a single file for uploading.
3. There are two ways of creating the ZIP file for uploading. Option one is:
 - A Quality Control Limits file with the name: **EDFCL.TXT**
This file contains control limit information concerning the QC results.
[Click here](#) for field information guidelines
 - A Quality Control file with the name: **EDFQC.TXT**
This file contains the quality assurance information associated with the results file
[Click here](#) for field information guidelines
 - A Results file with the name: **EDFRES.TXT**
This file contains the analytical results generated by the laboratory
[Click here](#) for field information guidelines
 - A Sample file with the name: **EDFSAMP.TXT**
This file contains the administrative information associated with sampling points
[Click here](#) for field information guidelines
 - A Test file with the name: **EDFTEST.TXT**
This file contains information concerning the analytical test associated with the sample
[Click here](#) for field information guidelines
4. Each file in the uploaded zip, except the narrative, must be in either [FIXED-LENGTH](#) or CSV format.
5. Once you have all the files, zip them together using a program like [winzip](#). The EDF upload will only accept ZIP files.
6. [Click here](#) to download an example of a properly made EDF upload. The files are in FIXED-LENGTH format.
7. **Delete the first row of the spreadsheet if it contains the column headings. Column headings are not allowed in the submitted file.**
8. Once you have a ZIP file with all the necessary files in it you are ready to upload.

EDFCL.TXT

Quality Control Limits File Field Guidelines

<u>Field</u>	<u>Fixed- Length Pos.</u>	<u>Field Name</u>	<u>Required</u>	<u>Description</u>
1	1-4	LABCODE	YES	The code identifying the laboratory that analyzes the sample
2	5-6	MATRIX	YES	The code identifying the sample matrix as determined by the laboratory

3	7-13	ANMCODE	YES	The code identifying the method of analysis
4	14-20	EXMCODE	YES	The code identifying the method of preparation
5	21-32	PARLABEL	YES	The code or CAS number identifying the analyte
6	33-40	CLREVDATE	YES	The date a control limit is established
7	41-46	CLCODE	YES	The code identifying the type of a quality control limit
8	47-50	UPPERCL	YES	The upper control limit of a quality control criterion
9	51-54	LOWERCL	NO	The lower control limit of a quality control criterion
10	55-294	(PROCEDURE_NAME)	NO	The method title as defined by the analysis laboratory
11	295-319	(LAB_METH_GRP)	NO	The unique identifier for a group of methods as defined by the laboratory
12	320-344	(METH_DESIGN_ID)	NO	The unique identifier for the design of an analytical method

NOTE: You cannot give NULL values for required fields

NOTE: Fields in parentheses can be omitted

EDFQC.TXT

QUALITY CONTROL FILE FIELD GUIDELINES

<u>Field</u>	<u>Fixed- Length Pos.</u>	<u>Field Name</u>	<u>Required</u>	<u>Description</u>
1	1-2	MATRIX	YES	The code identifying the sample matrix as determined by the laboratory
2	3-6	LABCODE	YES	The code identifying the laboratory that analyzes the sample
3	7-16	LABLOTCTL	YES	The unique identifier for a preparation and handling batch
4	17-23	ANMCODE	YES	The code identifying the method of analysis
5	24-35	PARLABEL	YES	The code or CAS number identifying the analyte

6	36-38	QCCODE	YES	The code identifying the type of sample
7	39-50	LABQCID	YES	The unique identification number assigned to the sample by the laboratory
8	51-62	LABREFID	YES	The laboratory sample ID of the quality control sample
9	63-76	EXPECTED	YES	The target result for a quality control sample or surrogate spike
10	77-86	UNITS	YES	The units for the parameter value measurement
11	87-326	(PROCEDURE_NAME)	NO	The method title as defined by the analysis laboratory
12	327-351	(LAB_METH_GRP)	NO	The unique identifier for a group of methods as defined by the laboratory
13	352-376	(METH_DESIGN_ID)	NO	The unique identifier for the design of an analytical method

NOTE: You cannot give NULL values for required fields

NOTE: Fields in parentheses can be omitted

EDFRES.TXT

RESULTS FILE FIELD GUIDELINES

<u>Field</u>	<u>Fixed- Length Pos.</u>	<u>Field Name</u>	<u>Required</u>	<u>Description</u>
1	1-2	MATRIX	YES	The code identifying the sample matrix
2	3-4	LABCODE	YES	The code identifying the laboratory that analyzes the sample
3	7-18	LABSAMPID	YES	The unique identification number assigned to the sample by the laboratory
4	19-21	QCCODE	YES	The code identifying the type of sample
5	22-28	ANMCODE	YES	The code identifying the method of analysis
6	29-35	EXMCODE	YES	The code identifying the method of preparation
7	36-37	PVCCODE	YES	The code identifying whether a sample result is a primary or a confirmatory value
8	38-45	ANADATE	YES	The date the sample is analyzed
9	46-47	RUN_NUMBER	YES	The numeric code distinguishing multiple or repeat analysis of a sample by the same method on the same day
10	48-59	PARLABEL	YES	The code or CAS number identifying the analyte
11	60-73	PARVAL	YES	The analytical value for a compound, analyte, or physical parameter

12	74-75	PARVQ	YES	The code identifying the qualifier of an analytical result
13	76-84	LABDL	NO	The laboratory-established method detection limit
14	85-93	REPDL	NO	The laboratory-established method detection limit, adjusted for the particular sample preparation
15	94-96	REPDLVQ	YES	The code identifying the type of reporting limit
16	97-108	PARUN	NO	The uncertainty of a measured value due to a measuring technique
17	109-118	UNITS	YES	The units for the parameter value measurement
18	119-125	RT	NO	The retention time of a tentatively identified compound (TIC), reported in minutes
19	126-135	DILFAC	YES	The numeric factor indicating the level of sample dilution
20	136-143	CLREVDAT	NO	The date a control limit is established
21	144-155	SRM	YES	The code identifying the standard reference material used in the analysis
22	156-175	LNOTE	NO	The code identifying notes pertaining to analytical performance irregularities that apply to a single analyte
23	176-415	(PROCEDURE_NAME)	NO	The method title as defined by the analysis laboratory
24	416-440	(LAB_METH_GRP)	NO	The unique identifier for a group of methods as defined by the laboratory
25	441-465	(METH_DESIGN_ID)	NO	The unique identifier for the design of an analytical method

NOTE: You cannot give NULL values for required fields

NOTE: Fields in parentheses can be omitted

EDFSAMP.TXT

SAMPLE FILE FIELD GUIDELINES

<u>Field</u>	<u>Fixed-Length Pos.</u>	<u>Field Name</u>	<u>Required</u>	<u>Description</u>
1	1-10	LOCID	YES	The Unique identifier for the samples location.
2	11-18	LOGDATE	YES	The Date a field sample is collected
3	19-22	LOGTIME	YES	The time a filed sample is collected (24-hour military time)
4	23-26	LOGCODE	YES	The code identifying the company collecting the sample

5	27-51	SAMPID	YES	The unique identifier representing a sample
6	52-53	MATRIX	YES	The code identifying the sample matrix as determined by the laboratory
7	54-78	PROJNAME	YES	The identification assigned to the project by the org. performing the work
8	79-85	LABWO	YES	A delivery order number associated with the contract
9	86-97	GLOBAL_ID	YES	The unique identifier for a regulated facility or site
10	98-101	LABCODE	YES	The code identifying the laboratory that analyzes the sample
11	102-126	(COOLER_ID)	NO	The unique identifier representing a cooler used to transport samples
12	152-153	(COC_MATRIX)	NO	The Code identifying the sample matrix as noted on the chain-of-custody
13	154-178	(DQO_ID)	NO	The unique identifier representing the data quality objectives

NOTE: You cannot give NULL values for required fields

NOTE: Fields in parentheses can be omitted

EDFTEST.TXT

TEST FILE FIELD GUIDELINES

<u>Field</u>	<u>Fixed- Length Pos.</u>	<u>Field Name</u>	<u>Required</u>	<u>Description</u>
1	1-10	LOCID	NO	The unique identifier for the samples location
2	11-18	LOGDATE	YES	The date a field sample is collected
3	19-22	LOGTIME	YES	The time that a field sample is collected, recorded using 24-hour military time
4	23-26	LOGCODE	YES	The code identifying the company collecting the samples or performing field tests
5	27-51	SAMPID	YES	The unique identifier representing a sample, assigned by the consultant, as submitted to the laboratory on a chain-of-custody
6	52-53	MATRIX	YES	The code identifying the sample matrix as determined by the laboratory
7	54-57	LABCODE	YES	The code identifying the laboratory that analyzes the sample
8	58-69	LABSAMPID	YES	The unique identification number assigned to the sample by the laboratory
9	70-72	QCCODE	YES	The code identifying the type of sample
10	73-79	ANMCODE	YES	The code identifying the method of analysis

11	80-80	MODPARLIST	YES	A field indicating whether the parameter list of an analytical method has been modified
12	81-87	EXMCODE	YES	The code identifying the method of preparation
13	88-97	LABLOTCTL	YES	The unique identifier for a preparation and handling batch
14	98-107	LCHMETH	NO	The code identifying the method of leaching performed
15	108-115	ANADATE	YES	The date the sample is analyzed
16	116-123	EXTDATE	YES	The date that a sample is prepared for analysis
17	124-125	RUN_NUMBER	YES	The numeric code distinguishing multiple or repeat analysis of a sample by the same method on the same day
18	126-133	RECDATE	YES	The date the sample is received by the laboratory doing the analysis
19	134-149	COCNUM	NO	The number assigned to the chain-of-custody
20	150-150	BASIS	YES	The code used to distinguish whether a sample is reported as dry or wet weight, filtered or not filtered
21	151-165	PRESCODE	NO	The code identifying the type of preservative added to the sample
22	166-169	SUB	YES	The code identifying the subcontracted laboratory
23	170-177	REP_DATE	NO	The date of the laboratory report
24	178-197	LAB_REPNO	NO	The unique identifier for the laboratory report, assigned by the laboratory
25	198-200	APPRVD	NO	The initials of the individual approving the laboratory report
26	201-220	LNOTE	NO	The code identifying notes pertaining to analytical performance irregularities that apply to the entire test
27	221-245	(REQ_METHOD_GRP)	NO	The unique identifier for the method or group of methods requested by the client for analysis of the sample
28	246-485	(PROCEDURE_NAME)	NO	The method title as defined by the analysis laboratory
29	486-510	(LAB_METH_GRP)	NO	The unique identifier for a group of methods as defined by the laboratory
30	511-535	(METH_DESIGN_ID)	NO	The unique identifier for the design of an analytical method
31	536-550	(CLEANUP)	NO	The code identifying the method of cleanup performed

NOTE: You cannot give NULL values for required fields

NOTE: Fields in parentheses can be omitted

ATTACHMENT D

Three Phase Quality Control Process and Documentation

**INVESTIGATION, MONITORING, O&M PROJECTS
PREPARATORY PHASE INSPECTION COVER SHEET**

Contract No.: _____

Date: _____

Task No.: _____

Location/Project: _____

A. Key Personnel Present:

	<u>Name</u>	<u>Position</u>	<u>Company</u>
1.	_____	_____	_____
2.	_____	_____	_____
3.	_____	_____	_____
4.	_____	_____	_____
5.	_____	_____	_____
6.	_____	_____	_____
7.	_____	_____	_____
8.	_____	_____	_____
9.	_____	_____	_____
10.	_____	_____	_____

B. Submittals:

1. Review submittals and/or submittal register. Have all applicable submittals been approved?

Yes___ No___

If No, what items have not been submitted?

- a. _____
- b. _____
- c. _____

USACE Representative Signature

Quality Control Manager Signature

INVESTIGATION, MONITORING, O&M PROJECTS: PREPARATORY PHASE INSPECTION CHECKLIST

Assessment Activity	Assessment Mechanism	Person(s) Responsible	Response Action	Completed by/Date
Have planning documents been prepared in accordance with the statement of work, regulatory requirements, and contract requirements?	Quality control review of document by Project Manager and QC reviewer.	Project Manager, QC Reviewer	Modify document as directed by reviewers	
Prior to project activities: Have planning documents been read by appropriate project personnel (including subcontractors) before work is conducted.	Documentation (e.g., sign-off form, note to file, email acknowledgement) that document has been read and requirements are understood.	Subcontractors as required. Project Manager, Task Manager, and Project Chemist to check signoff and forms.	Direct project personnel to read relevant documents.	
Prior to project activities: Has required preliminary work (e.g., clearance activities, permits, site access) been completed in accordance with project plan.	Comparison of information obtained from preliminary work completion assessment as specified in the project planning document(s).	Project Manager, Safety and Health Officer, QC Manger/Reviewer, Task Manager, Project Chemist, Field Staff	Delay startup if necessary preliminary work has not been completed. Implement corrective actions by directing appropriate personnel or subcontractors to complete necessary preliminary work.	
Prior to project activities: Are staff and subcontractors prepared to implement project activities according to planning documents?	Review and discussion of planned activities prior to implementation.	Project Manager, Safety and Health Officer, Quality Control System Manager, Task Manager, Project Chemist, Field staff.	Delay startup if staff and subcontractors are not prepared to implement activities <i>in</i> accordance with specification.	
Prior to project activities: Is necessary field equipment available and in acceptable working order?	Compare field equipment list with planned activities. Compare field equipment calibration documentation with project goals specified in the SAP.	Project Manager, Quality Control System Manager, Task Manager, Project Chemist, Field staff.	Delay startup if equipment is unavailable or not in proper working order. Implement corrective actions to include use of alternate equipment, or recalibration of available equipment.	

**INVESTIGATION, MONITORING, O&M PROJECTS
INITIAL PHASE INSPECTION COVER SHEET**

Contract No.: _____

Date: _____

Task No.: _____

Location/Project: _____

Description and Location of Work Inspected: _____

A. Key Personnel Present:

	<u>Name</u>	<u>Position</u>	<u>Company</u>
1.	_____	_____	_____
2.	_____	_____	_____
3.	_____	_____	_____
4.	_____	_____	_____
5.	_____	_____	_____
6.	_____	_____	_____
7.	_____	_____	_____
8.	_____	_____	_____
9.	_____	_____	_____
10.	_____	_____	_____

Quality Control Manager Signature

INVESTIGATION, MONITORING, O&M PROJECTS: INITIAL PHASE INSPECTION CHECKLIST

Assessment Activity	Assessment Mechanism	Person(s) Responsible	Response Action	Completed by/Date
Beginning of project activity: Is work being performed according to project plans?	Conduct field and laboratory audits.	Project Manager, Quality Control System Manager, Task Manager, Project Chemist, Field staff.	Stop work if audits indicate significant deviation from project plan. Implement immediate or long- term corrective actions. Communicate deficiencies to USACE Project Manager.	
Early phase of project: Have necessary audits been performed?	Review project phase and check to see if required audits have <i>been</i> satisfactorily completed.	Project Manager, Project Manager, Quality Control System Manager	Stop work if reviewer decides that absence of audit jeopardizes successful implementation of project plans. Immediately schedule necessary audits.	
Ongoing throughout project: Are daily quality control reports being prepared according to contract requirements?	Review Content and delivery schedules of daily quality control reports.	Project Manager, Task Manager, Project Chemist, Project Staff	Correct deficiencies in reports or reporting delays.	
Ongoing throughout project: Do project plans adequately address any changes in project activities or goal?	Compare data gathered to assess conformance to the project plan and conceptual site model.	Project Manager, Safety and Health Officer, Quality Control System Manager, Task Manager, Project Chemist, Field staff.	Stop work if assessor decides that project plan deficiencies are significant. Implement corrective action to include modification of project plans. Notify USACE Project Manager.	
Ongoing throughout project: Do project plans adequately address any changes in project activities or goals?	Compare data gathered to assess conformance to the conceptual site model, data quality objectives, and project plan.	Project Manager, Quality Control System Manager, Task Manager, Project Chemist, data users and evaluators.	Propose additional data collection activities to fill data gaps. Notify USACE Project Manager. Revise or update planning documents as appropriate.	

**INVESTIGATION, MONITORING, O&M PROJECTS
FOLLOW-UP PHASE INSPECTION COVER SHEET**

Contract No.: _____
Task No.: _____
Location/Project: _____

Date: _____

Project/Area of Inspection: _____

A. Key Personnel Present:

	<u>Name</u>	<u>Position</u>	<u>Company</u>
1.	_____	_____	_____
2.	_____	_____	_____
3.	_____	_____	_____
4.	_____	_____	_____
5.	_____	_____	_____
6.	_____	_____	_____
7.	_____	_____	_____
8.	_____	_____	_____
9.	_____	_____	_____
10.	_____	_____	_____

B. Definable Features of Work:

Status of Inspection:

Quality Control Manager

INVESTIGATION PROJECT FOLLOW-UP PHASE INSPECTION CHECKLIST

Assessment Activity	Assessment Mechanism	Person(s) Responsible	Response Action	Completed by/Date
Reporting phase of project: Have data reports been prepared in accordance with project plans?	Compare data reports to specifications detailed in planning documents.	Project Manager, Quality Control Manager, Task Manager, Project Chemist, data users and evaluators.	Revise documents and reports as appropriate.	
After draft report submittal or project completion: Are reports adequate to meet client and regulatory agency requirements?	Review client and agency comments. Prepare responses to comments.	Project Manager, Quality Control Manager, Task Manager, Project Chemist, data users and evaluators.	Revise documents and reports as appropriate.	
Have other definable features of work been completed in accordance to project requirements	Compare definable features of work with project requirements.	Project Manager, Quality Control Manager	Complete definable feature of work as required.	

ATTACHMENT E

Analytical Laboratory Certifications



CERTIFICATE OF ACCREDITATION

The ANSI National Accreditation Board

Hereby attests that

Eurofins Air Toxics, LLC

**180 Blue Ravine Road
Folsom, CA 95630**

Fulfills the requirements of

ISO/IEC 17025:2017

and

U.S. Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories (DoD QSM V5.3)

In the field of

TESTING

This certificate is valid only when accompanied by a current scope of accreditation document.
The current scope of accreditation can be verified at www.anab.org.

R. Douglas Leonard Jr., VP, PILR SBU

Expiry Date: 27 April 2022
Certificate Number: ADE-1451



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



**SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017 AND U.S.
DEPARTMENT OF DEFENSE (DOD) QUALITY SYSTEMS MANUAL
FOR ENVIRONMENTAL LABORATORIES (DOD QSM V5.3)**

Eurofins Air Toxics, LLC

180 Blue Ravine Road
Folsom, CA 95630
Melanie Levesque
(916) 605-3396

TESTING

Valid to: **April 27, 2022**

Certificate Number: **ADE-1451**

Environmental

Air and Emissions		
Technology	Method	Analyte
GC/FID/dual TCD	ASTM D1945 Mod	Acetylene
GC/FID/dual TCD	ASTM D1945 Mod	Carbon Dioxide
GC/FID/dual TCD	ASTM D1945 Mod	Carbon Monoxide
GC/FID/dual TCD	ASTM D1945 Mod	Ethane
GC/FID/dual TCD	ASTM D1945 Mod	Ethylene
GC/FID/dual TCD	ASTM D1945 Mod	Helium
GC/FID/dual TCD	ASTM D1945 Mod	Hydrogen
GC/FID/dual TCD	ASTM D1945 Mod	Isobutane
GC/FID/dual TCD	ASTM D1945 Mod	Isopentane
GC/FID/dual TCD	ASTM D1945 Mod	Methane
GC/FID/dual TCD	ASTM D1945 Mod	n-Butane
GC/FID/dual TCD	ASTM D1945 Mod	Neopentane

Air and Emissions		
Technology	Method	Analyte
GC/FID/dual TCD	ASTM D1945 Mod	Nitrogen
GC/FID/dual TCD	ASTM D1945 Mod	n-Pentane
GC/FID/dual TCD	ASTM D1945 Mod	Oxygen
GC/FID/dual TCD	ASTM D1945 Mod	Propane
GC/FID/dual TCD	ASTM D1946 Mod	Carbon Dioxide
GC/FID/dual TCD	ASTM D1946 Mod	Carbon Monoxide
GC/FID/dual TCD	ASTM D1946 Mod	Ethane
GC/FID/dual TCD	ASTM D1946 Mod	Ethylene
GC/FID/dual TCD	ASTM D1946 Mod	Helium
GC/FID/dual TCD	ASTM D1946 Mod	Hydrogen
GC/FID/dual TCD	ASTM D1946 Mod	Methane
GC/FID/dual TCD	ASTM D1946 Mod	Nitrogen
GC/FID/dual TCD	ASTM D1946 Mod	Oxygen
GC/FID/PID	TO-3 Mod	TPH(GRO)
GC/FID/PID	TO-3 Mod	TPH(JP4)
GC/MS	TO-15 (Full Scan/SIM)	1,1,1-Trichloroethane
GC/MS	TO-15 (Full Scan/SIM)	1,1,2,2-Tetrachloroethane
GC/MS	TO-15 (Full Scan/SIM)	1,1,2-Trichloroethane
GC/MS	TO-15 (Full Scan/SIM)	1,1-Dichloroethane
GC/MS	TO-15 (Full Scan/SIM)	1,1-Dichloroethene
GC/MS	TO-15 (Full Scan)	1,2,3-Trichlorobenzene
GC/MS	TO-15 (Full Scan/SIM)	1,2,3-Trichloropropane
GC/MS	TO-15 (Full Scan/SIM)	1,2,4-Trichlorobenzene

Air and Emissions		
Technology	Method	Analyte
GC/MS	TO-15 (Full Scan/SIM)	1,2,4-Trimethylbenzene
GC/MS	TO-15 (Full Scan/SIM)	1,2-Dibromoethane (EDB)
GC/MS	TO-15 (Full Scan/SIM)	1,2-Dichlorobenzene
GC/MS	TO-15 (Full Scan/SIM)	1,2-Dichloroethane
GC/MS	TO-15 (Full Scan/SIM)	1,2-Dichloropropane
GC/MS	TO-15 (Full Scan/SIM)	1,2-Dichlorotetrafluoroethane (Freon 114)
GC/MS	TO-15 (Full Scan/SIM)	1,3,5-Trimethylbenzene
GC/MS	TO-15 (Full Scan/SIM)	1,3-Butadiene
GC/MS	TO-15 (Full Scan/SIM)	1,3-Dichlorobenzene
GC/MS	TO-15 (Full Scan/SIM)	1,4-Dichlorobenzene
GC/MS	TO-15 (Full Scan/SIM)	1,4-Dioxane
GC/MS	TO-15 (Full Scan)	2,2,4-Trimethylpentane
GC/MS	TO-15 (Full Scan/SIM)	2-Butanone (MEK)
GC/MS	TO-15 (Full Scan)	2-Chlorotoluene
GC/MS	TO-15 (Full Scan/SIM)	2-Hexanone
GC/MS	TO-15 (Full Scan/SIM)	2-Propanol
GC/MS	TO-15 (Full Scan)	3-Chloropropene
GC/MS	TO-15 (Full Scan)	4-Isopropyltoluene (p-Cymene)
GC/MS	TO-15 (Full Scan/SIM)	4-Methyl-2-pentanone (MIBK)
GC/MS	TO-15 (Full Scan/SIM)	Acetone
GC/MS	TO-15 SIM	Acetonitrile
GC/MS	TO-15 (Full Scan/SIM)	Acrolein
GC/MS	TO-15 SIM	Acrylonitrile

Air and Emissions		
Technology	Method	Analyte
GC/MS	TO-15 (Full Scan/SIM)	alpha-Chlorotoluene
GC/MS	TO-15 (Full Scan)	alpha-Methyl Styrene
GC/MS	TO-15 (Full Scan/SIM)	Benzene
GC/MS	TO-15 (Full Scan/SIM)	Bromodichloromethane
GC/MS	TO-15 (Full Scan/SIM)	Bromoform
GC/MS	TO-15 (Full Scan/SIM)	Bromomethane
GC/MS	TO-15 (Full Scan)	Butane
GC/MS	TO-15 (Full Scan)	Butyl Benzene
GC/MS	TO-15 (Full Scan)	Carbon disulfide
GC/MS	TO-15 (Full Scan/SIM)	Carbon tetrachloride
GC/MS	TO-15 (Full Scan/SIM)	Chlorobenzene
GC/MS	TO-15 (Full Scan/SIM)	Chlorodibromomethane
GC/MS	TO-15 (Full Scan/SIM)	Chloroethane
GC/MS	TO-15 (Full Scan/SIM)	Chloroform
GC/MS	TO-15 (Full Scan/SIM)	Chloromethane
GC/MS	TO-15 (Full Scan/SIM)	cis-1,2-Dichloroethene
GC/MS	TO-15 (Full Scan/SIM)	cis-1,3-Dichloropropene
GC/MS	TO-15 (Full Scan/SIM)	Cyclohexane
GC/MS	TO-15 (Full Scan)	Cumene
GC/MS	TO-15 (Full Scan)	Dibromomethane
GC/MS	TO-15 (Full Scan/SIM)	Dichlorodifluoromethane (Freon 12)
GC/MS	TO-15 (Full Scan)	Ethanol
GC/MS	TO-15 (Full Scan/SIM)	Ethyl Acetate

Air and Emissions		
Technology	Method	Analyte
GC/MS	TO-15 (Full Scan/SIM)	Ethylbenzene
GC/MS	TO-15 (Full Scan/SIM)	Hexachlorobutadiene
GC/MS	TO-15 (Full Scan/SIM)	Methylene Chloride
GC/MS	TO-15 (Full Scan/SIM)	m,p-Xylene
GC/MS	TO-15 (Full Scan/SIM)	Naphthalene
GC/MS	TO-15 (Full Scan)	n-Butanol (1-Butanol)
GC/MS	TO-15 (Full Scan/SIM)	n-Heptane
GC/MS	TO-15 (Full Scan/SIM)	n-Hexane
GC/MS	TO-15 (Full Scan)	Nonane
GC/MS	TO-15 (Full Scan)	n-Pentane
GC/MS	TO-15 (Full Scan)	n-Propylbenzene
GC/MS	TO-15 (Full Scan)	Octane
GC/MS	TO-15 (Full Scan/SIM)	o-Xylene
GC/MS	TO-15 (Full Scan/SIM)	p-Ethyltoluene
GC/MS	TO-15 (Full Scan/SIM)	Propylene
GC/MS	TO-15 (Full Scan)	sec-Butylbenzene
GC/MS	TO-15 (Full Scan/SIM)	Styrene
GC/MS	TO-15 (Full Scan)	tert-Butyl Alcohol
GC/MS	TO-15 (Full Scan)	tert-Butyl Benzene
GC/MS	TO-15 (Full Scan/SIM)	tert-Butyl methyl ether (MTBE)
GC/MS	TO-15 (Full Scan/SIM)	Tetrachloroethylene
GC/MS	TO-15 (Full Scan/SIM)	Tetrahydrofuran
GC/MS	TO-15 (Full Scan/SIM)	Toluene

Air and Emissions		
Technology	Method	Analyte
GC/MS	TO-15 (Full Scan/SIM)	trans-1,2-Dichloroethene
GC/MS	TO-15 (Full Scan/SIM)	trans-1,3-Dichloropropene
GC/MS	TO-15 (Full Scan/SIM)	Trichloroethene
GC/MS	TO-15 (Full Scan/SIM)	Trichlorofluoromethane (Freon 11)
GC/MS	TO-15 (Full Scan)/SIM	Trichlorotrifluoroethane (Freon 113)
GC/MS	TO-15 (Full Scan/SIM)	Vinyl Acetate
GC/MS	TO-15 (Full Scan)	Vinyl Bromide
GC/MS	TO-15 (Full Scan/SIM)	Vinyl chloride
GC/MS	TO-17 (WMS/RAD130) Mod	1,1,1-Trichloroethane
GC/MS	TO-17 (WMS/RAD130) Mod	1,1,2,2-Tetrachloroethane
GC/MS	TO-17 (WMS/RAD130) Mod	1,1,2-Trichloroethane
GC/MS	TO-17 (WMS/RAD130) Mod	1,1-Dichloroethane
GC/MS	TO-17 (WMS/RAD130) Mod	1,1-Dichloroethene
GC/MS	TO-17 (WMS/RAD130) Mod	1,2,4-Trimethylbenzene
GC/MS	TO-17 (WMS/RAD130) Mod	1,2-Dichlorobenzene
GC/MS	TO-17 (WMS/RAD130) Mod	1,2-Dichloroethane
GC/MS	TO-17 (WMS/RAD130) Mod	1,3,5-Trimethylbenzene
GC/MS	TO-17 (WMS/RAD130) Mod	1,3-Dichlorobenzene
GC/MS	TO-17 (WMS/RAD130) Mod	1,4-Dichlorobenzene
GC/MS	TO-17 (WMS/RAD130) Mod	2-Butanone (MEK)
GC/MS	TO-17 (WMS/RAD130) Mod	4-Methyl-2-pentanone (MIBK)
GC/MS	TO-17 (WMS/RAD130) Mod	Benzene
GC/MS	TO-17 (WMS/RAD130) Mod	Carbon tetrachloride

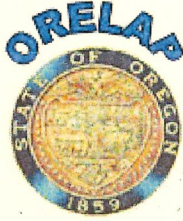
Air and Emissions		
Technology	Method	Analyte
GC/MS	TO-17 (WMS/RAD130) Mod	Chlorobenzene
GC/MS	TO-17 (WMS/RAD130) Mod	Chloroform
GC/MS	TO-17 (WMS/RAD130) Mod	cis-1,2-Dichloroethene
GC/MS	TO-17 (WMS/RAD130) Mod	Cyclohexane
GC/MS	TO-17 (WMS/RAD130) Mod	Ethanol
GC/MS	TO-17 (WMS/RAD130) Mod	Ethyl Acetate
GC/MS	TO-17 (WMS/RAD130) Mod	Ethylbenzene
GC/MS	TO-17 (WMS/RAD130) Mod	m,p-Xylene
GC/MS	TO-17 (WMS/RAD130) Mod	Naphthalene
GC/MS	TO-17 (WMS/RAD130) Mod	n-Heptane
GC/MS	TO-17 (WMS/RAD130) Mod	n-Hexane
GC/MS	TO-17 (WMS/RAD130) Mod	o-Xylene
GC/MS	TO-17 (WMS/RAD130) Mod	Propylbenzene
GC/MS	TO-17 (WMS/RAD130) Mod	Styrene
GC/MS	TO-17 (WMS/RAD130) Mod	tert-Butyl methyl ether (MTBE)
GC/MS	TO-17 (WMS/RAD130) Mod	Tetrachloroethylene
GC/MS	TO-17 (WMS/RAD130) Mod	Toluene
GC/MS	TO-17 (WMS/RAD130) Mod	trans-1,2-Dichloroethene
GC/MS	TO-17 (WMS/RAD130) Mod	Trichloroethene
GC/MS	TO-17 (WMS) Mod	Vinyl chloride

Note:

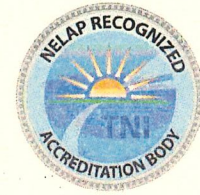
1. This scope is formatted as part of a single document including Certificate of Accreditation No. ADE-1451.



R. Douglas Leonard Jr., VP, PILR SBU



OREGON Environmental Laboratory Accreditation Program



Eurofins Air Toxics, LLC
CA300005

NELAP Recognized

180 Blue Ravine Road, Ste. B
Folsom, CA 95630

IS GRANTED APPROVAL BY ORELAP UNDER THE 2009 TNI STANDARDS, TO PERFORM ANALYSES ON ENVIRONMENTAL SAMPLES IN MATRICES AS LISTED BELOW :

<i>Air</i>	<i>Drinking Water</i>	<i>Non Potable Water</i>	<i>Solids and Chem. Waste</i>	<i>Tissue</i>
Chemistry				

AND AS RECORDED IN THE LIST OF APPROVED ANALYTES, METHODS, ANALYTICAL TECHNIQUES, AND FIELDS OF TESTING ISSUED CONCURRENTLY WITH THIS CERTIFICATE AND REVISED AS NECESSARY.

ACCREDITED STATUS DEPENDS ON SUCCESSFUL ONGOING PARTICIPATION IN THE PROGRAM AND CONTINUED COMPLIANCE WITH THE STANDARDS.

CUSTOMERS ARE URGED TO VERIFY THE LABORATORY'S CURRENT ACCREDITATION STATUS IN OREGON.

Travis Bartholomew
Oregon State Public Health Laboratory
ORELAP Program Manager
7202 NE Evergreen Parkway, Suite 100
Hillsboro, OR 97124

EFFECTIVE DATE : 10/18/2020

EXPIRATION DATE : 10/17/2021

Certificate No : CA300005 - 014





OREGON

Environmental Laboratory Accreditation Program

ORELAP Fields of Accreditation

ORELAP ID: CA300005

EPA CODE: CA00933

Certificate: CA300005 - 014



Eurofins Air Toxics, LLC

180 Blue Ravine Road, Ste. B

Folsom, CA 95630

Issue Date: 10/18/2020 Expiration Date: 10/17/2021

As of 10/18/2020 this list supersedes all previous lists for this certificate number.

MATRIX	Reference	Code	Analyte	Code	Description
--------	-----------	------	---------	------	-------------

Air

	40 CFR Part 50 Appendix J			10000507	Determination of Particulate Matter as PM10 PARTICULATE MATTER AS PM10 IN THE ATMOSPHERE
		3950	Particulates <10 um		
	ASTM D1945 03			30024443	Natural Gas by Gas Chromatography
		4938	2-Methylbutane (Isopentane)		
		4942	2-methylpropane (Isobutane)		
		4323	Acetylene		
		3755	Carbon dioxide		
		3780	Carbon monoxide		
		4747	Ethane		
		4752	Ethene		
		1767	Helium		
		1772	Hydrogen		
		4926	Methane		
		3853	Natural Gas		
		5007	n-Butane		
		9511	Neopentane		
		1843	Nitrogen		
		5028	n-Pentane		
		5029	n-Propane		
		3895	Oxygen		
	ASTM D1946- 90			30024465	Reformed Gas by Gas Chromatography
		3755	Carbon dioxide		
		3780	Carbon monoxide		
		4747	Ethane		
		4752	Ethene		
		1767	Helium		
		1772	Hydrogen		
		4926	Methane		
		1843	Nitrogen		
		3895	Oxygen		
	EPA 325B 2013			10277437	Sorbent Tubes Coupled with Thermal Desorption and GC/MS
		9318	1,3-Butadiene		
		4375	Benzene		
		4765	Ethylbenzene		
		5240	m+p-xylene		
		5005	Naphthalene		



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Air	EPA Method	Method Number	Method Name	Method Code	Method Description
	EPA 325B 2013	4855	n-Hexane		
		5250	o-Xylene		
		5100	Styrene		
		5140	Toluene		
		5170	Trichloroethene (Trichloroethylene)		
	EPA TO-12	3860	Non-methane organics	10248201	Non-Methane Organic Compounds by GC/FID
	EPA TO-13A	5795	2-Chloronaphthalene	10248405	Polycyclic Aromatic Hydrocarbons in Ambient Air by GC/MS
		6385	2-Methylnaphthalene		
		5500	Acenaphthene		
		5505	Acenaphthylene		
		5555	Anthracene		
		5575	Benzo(a)anthracene		
		5580	Benzo(a)pyrene		
		5590	Benzo(g,h,i)perylene		
		5600	Benzo(k)fluoranthene		
		5585	Benzo[b]fluoranthene		
		5855	Chrysene		
		5895	Dibenz(a,h)anthracene		
		6265	Fluoranthene		
		6270	Fluorene		
		6315	Indeno(1,2,3-cd)pyrene		
		5005	Naphthalene		
		6615	Phenanthrene		
		6665	Pyrene		
	EPA TO-13A SIM	5795	2-Chloronaphthalene	10248449	Polycyclic Aromatic Hydrocarbons in Ambient Air by GC/MS SIM
		6385	2-Methylnaphthalene		
		5500	Acenaphthene		
		5505	Acenaphthylene		
		5555	Anthracene		
		5575	Benzo(a)anthracene		
		5580	Benzo(a)pyrene		
		5590	Benzo(g,h,i)perylene		
		5600	Benzo(k)fluoranthene		
		5585	Benzo[b]fluoranthene		
		5855	Chrysene		
		5895	Dibenz(a,h)anthracene		
		6265	Fluoranthene		
		6270	Fluorene		



OREGON

Environmental Laboratory Accreditation Program



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Air	EPA TO-13A SIM	6315	Indeno(1,2,3-cd) pyrene
		5005	Naphthalene
		6615	Phenanthrene
		6665	Pyrene
	EPA TO-14A	10248609	Volatile Organic Compounds with SUMMA canister and GC/MS
		5160	1,1,1-Trichloroethane
		5110	1,1,2,2-Tetrachloroethane
		5195	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
		5165	1,1,2-Trichloroethane
		4630	1,1-Dichloroethane
		4640	1,1-Dichloroethylene
		5155	1,2,4-Trichlorobenzene
		5210	1,2,4-Trimethylbenzene
		4585	1,2-Dibromoethane (EDB, Ethylene dibromide)
		4695	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon-114)
		4610	1,2-Dichlorobenzene
		4635	1,2-Dichloroethane (Ethylene dichloride)
		4655	1,2-Dichloropropane
		5215	1,3,5-Trimethylbenzene
		9318	1,3-Butadiene
		4615	1,3-Dichlorobenzene
		4620	1,4-Dichlorobenzene
		4735	1,4-Dioxane (1,4- Diethyleneoxide)
		4836	1-Propene (Propylene)
		4410	2-Butanone (Methyl ethyl ketone, MEK)
		4860	2-Hexanone (MBK)
		4542	4-Ethyltoluene
		4995	4-Methyl-2-pentanone (MIBK)
		4315	Acetone
		4375	Benzene
		5635	Benzyl chloride
		4395	Bromodichloromethane
		4400	Bromoform
		4450	Carbon disulfide
		4455	Carbon tetrachloride
		4475	Chlorobenzene
		4575	Chlorodibromomethane
		4485	Chloroethane (Ethyl chloride)
		4505	Chloroform
		4705	cis & trans-1,2-Dichloroethene
		4680	cis-1,3-Dichloropropene



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Air	EPA TO-14A	4555	Cyclohexane
		4625	Dichlorodifluoromethane (Freon-12)
		4750	Ethanol
		4765	Ethylbenzene
		4835	Hexachlorobutadiene
		4895	Isopropyl alcohol (2-Propanol, Isopropanol)
		4900	Isopropylbenzene (Cumene)
		5240	m+p-xylene
		4950	Methyl bromide (Bromomethane)
		4960	Methyl chloride (Chloromethane)
		5000	Methyl tert-butyl ether (MTBE)
		4975	Methylene chloride (Dichloromethane)
		5005	Naphthalene
		4825	n-Heptane
		4855	n-Hexane
		5090	n-Propylbenzene
		5250	o-Xylene
		5100	Styrene
		5115	Tetrachloroethylene (Perchloroethylene)
		5120	Tetrahydrofuran (THF)
		5140	Toluene
		4685	trans-1,3-Dichloropropylene
		5170	Trichloroethene (Trichloroethylene)
		5175	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)
		5235	Vinyl chloride
		5260	Xylene (total)
	EPA TO-15	10248803	VOCs collected in Canisters by GC/MS
		5160	1,1,1-Trichloroethane
		5110	1,1,1,2-Tetrachloroethane
		5195	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
		5165	1,1,2-Trichloroethane
		4630	1,1-Dichloroethane
		4640	1,1-Dichloroethylene
		5180	1,2,3-Trichloropropane
		5182	1,2,3-Trimethylbenzene
		5155	1,2,4-Trichlorobenzene
		5210	1,2,4-Trimethylbenzene
		4585	1,2-Dibromoethane (EDB, Ethylene dibromide)
		4695	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon-114)
		4610	1,2-Dichlorobenzene



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Air	EPA TO-15	4635	1,2-Dichloroethane (Ethylene dichloride)
		4655	1,2-Dichloropropane
		5215	1,3,5-Trimethylbenzene
		9318	1,3-Butadiene
		4615	1,3-Dichlorobenzene
		4676	1,3-Diethylbenzene
		4620	1,4-Dichlorobenzene
		4735	1,4-Dioxane (1,4- Diethyleneoxide)
		4917	1-Butene
		4833	1-Pentene
		4836	1-Propene (Propylene)
		5220	2,2,4-Trimethylpentane
		4666	2,2-Dimethylbutane
		4667	2,3,4-Trimethylpentane
		4669	2,3-Dimethylbutane
		4671	2,3-Dimethylpentane
		4672	2,4-Dimethylpentane
		4410	2-Butanone (Methyl ethyl ketone, MEK)
		4535	2-Chlorotoluene
		4538	2-Ethyltoluene
		4860	2-Hexanone (MBK)
		4934	2-Methyl-2-Butene
		4937	2-Methylbutadiene (Isoprene)
		4938	2-Methylbutane (Isopentane)
		4939	2-Methylheptane
		4946	2-Methylhexane
		4941	2-Methylpentane (Isohexane)
		4942	2-methylpropane (Isobutane)
		4531	3-Ethyltoluene (1-Methyl-3-ethylbenzene)
		4529	3-Methyl-1-Butene
		4532	3-Methylheptane
		4533	3-Methylhexane
		4534	3-Methylpentane
		4542	4-Ethyltoluene
		4910	4-Isopropyltoluene (p-Cymene)
		4913	4-Methyl-1-Pentene
		4995	4-Methyl-2-pentanone (MIBK)
		4300	Acetaldehyde
		4315	Acetone
		4320	Acetonitrile
		4323	Acetylene
		4325	Acrolein (Propenal)
		4340	Acrylonitrile



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Air	EPA TO-15		
		4355	Allyl chloride (3-Chloropropene)
		4357	alpha-Methylstyrene
		4375	Benzene
		5635	Benzyl chloride
		4390	Bromochloromethane
		4395	Bromodichloromethane
		4400	Bromoform
		4450	Carbon disulfide
		4455	Carbon tetrachloride
		4475	Chlorobenzene
		4575	Chlorodibromomethane
		4577	Chlorodifluoromethane (Freon-22)
		4485	Chloroethane (Ethyl chloride)
		4505	Chloroform
		4525	Chloroprene (2-Chloro-1,3-butadiene)
		4705	cis & trans-1,2-Dichloroethene
		4680	cis-1,3-Dichloropropene
		4602	cis-2-Butene
		4604	cis-2-Hexene
		4603	cis-2-pentene
		4555	Cyclohexane
		4562	Cyclopentane
		4563	Cyclopentene
		4595	Dibromomethane (Methylene bromide)
		4625	Dichlorodifluoromethane (Freon-12)
		4627	Dichlorofluoromethane (Freon 21)
		4725	Diethyl ether
		4747	Ethane
		4750	Ethanol
		4752	Ethene
		4755	Ethyl acetate
		4765	Ethylbenzene
		4835	Hexachlorobutadiene
		4895	Isopropyl alcohol (2-Propanol, Isopropanol)
		4900	Isopropylbenzene (Cumene)
		5240	m+p-xylene
		4950	Methyl bromide (Bromomethane)
		4960	Methyl chloride (Chloromethane)
		4990	Methyl methacrylate
		5000	Methyl tert-butyl ether (MTBE)
		4965	Methylcyclohexane
		4966	Methylcyclopentane
		4975	Methylene chloride (Dichloromethane)



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Air	EPA TO-15		
		5005	Naphthalene
		5007	n-Butane
		4435	n-Butylbenzene
		5875	n-Decane
		4825	n-Heptane
		4855	n-Hexane
		5026	n-Nonane
		5027	n-Octane
		5028	n-Pentane
		5029	n-Propane
		5090	n-Propylbenzene
		6747	n-Undecane
		5250	o-Xylene
		5253	p-Diethylbenzene
		4440	sec-Butylbenzene
		5100	Styrene
		4420	tert-Butyl alcohol
		4445	tert-Butylbenzene
		5115	Tetrachloroethylene (Perchloroethylene)
		5120	Tetrahydrofuran (THF)
		5140	Toluene
		4685	trans-1,3-Dichloropropylene
		4607	trans-2-Butene
		4606	trans-2-Hexene
		4608	trans-2-pentene
		5170	Trichloroethene (Trichloroethylene)
		5175	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)
		5225	Vinyl acetate
		5230	Vinyl bromide (Bromoethane)
		5235	Vinyl chloride
		5260	Xylene (total)
	EPA TO-15 GC/MS SIM	10248858	VOCs collected in Canisters by GC/MS SIM
		5160	1,1,1-Trichloroethane
		5110	1,1,2,2-Tetrachloroethane
		5185	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
		5165	1,1,2-Trichloroethane
		4630	1,1-Dichloroethane
		4640	1,1-Dichloroethylene
		5180	1,2,3-Trichloropropane
		5155	1,2,4-Trichlorobenzene
		5210	1,2,4-Trimethylbenzene



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Air	EPA TO-15 GC/MS SIM	4585	1,2-Dibromoethane (EDB, Ethylene dibromide)
		4695	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon-114)
		4610	1,2-Dichlorobenzene
		4635	1,2-Dichloroethane (Ethylene dichloride)
		4655	1,2-Dichloropropane
		5215	1,3,5-Trimethylbenzene
		9318	1,3-Butadiene
		4615	1,3-Dichlorobenzene
		4620	1,4-Dichlorobenzene
		4735	1,4-Dioxane (1,4- Diethyleneoxide)
		4836	1-Propene (Propylene)
		4410	2-Butanone (Methyl ethyl ketone, MEK)
		4860	2-Hexanone (MBK)
		4542	4-Ethyltoluene
		4995	4-Methyl-2-pentanone (MIBK)
		4315	Acetone
		4320	Acetonitrile
		4325	Acrolein (Propenal)
		4340	Acrylonitrile
		4375	Benzene
		5635	Benzyl chloride
		4395	Bromodichloromethane
		4400	Bromoform
		4455	Carbon tetrachloride
		4475	Chlorobenzene
		4575	Chlorodibromomethane
		4485	Chloroethane (Ethyl chloride)
		4505	Chloroform
		4645	cis-1,2-Dichloroethylene
		4680	cis-1,3-Dichloropropene
		4555	Cyclohexane
		4625	Dichlorodifluoromethane (Freon-12)
		4755	Ethyl acetate
		4765	Ethylbenzene
		4795	Ethylene oxide
		4835	Hexachlorobutadiene
		4895	Isopropyl alcohol (2-Propanol, Isopropanol)
		5240	m+p-xylene
		4950	Methyl bromide (Bromomethane)
		4960	Methyl chloride (Chloromethane)
		5000	Methyl tert-butyl ether (MTBE)



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Air	EPA TO-15 GC/MS SIM	4975	Methylene chloride (Dichloromethane)
		5005	Naphthalene
		4825	n-Heptane
		4855	n-Hexane
		5250	o-Xylene
		5100	Styrene
		5115	Tetrachloroethylene (Perchloroethylene)
		5120	Tetrahydrofuran (THF)
		5140	Toluene
		4700	trans-1,2-Dichloroethylene
		4685	trans-1,3-Dichloropropylene
		5170	Trichloroethene (Trichloroethylene)
		5175	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)
		5225	Vinyl acetate
		5235	Vinyl chloride
	EPA TO-17	10312206	Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes
		5160	1,1,1-Trichloroethane
		5195	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
		5165	1,1,2-Trichloroethane
		4630	1,1-Dichloroethane
		4640	1,1-Dichloroethylene
		5155	1,2,4-Trichlorobenzene
		5210	1,2,4-Trimethylbenzene
		4695	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon-114)
		4610	1,2-Dichlorobenzene
		4635	1,2-Dichloroethane (Ethylene dichloride)
		4655	1,2-Dichloropropane
		5215	1,3,5-Trimethylbenzene
		9318	1,3-Butadiene
		4615	1,3-Dichlorobenzene
		4620	1,4-Dichlorobenzene
		4735	1,4-Dioxane (1,4- Diethyleneoxide)
		6380	1-Methylnaphthalene
		5220	2,2,4-Trimethylpentane
		4410	2-Butanone (Methyl ethyl ketone, MEK)
		4860	2-Hexanone (MBK)
		4938	2-Methylbutane (Isopentane)
		6385	2-Methylnaphthalene
		4542	4-Ethyltoluene
		4995	4-Methyl-2-pentanone (MIBK)



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Air	EPA TO-17	5500	Acenaphthene
		5505	Acenaphthylene
		5555	Anthracene
		4375	Benzene
		4450	Carbon disulfide
		4455	Carbon tetrachloride
		4475	Chlorobenzene
		4485	Chloroethane (Ethyl chloride)
		4505	Chloroform
		4645	cis-1,2-Dichloroethylene
		4555	Cyclohexane
		4765	Ethylbenzene
		6265	Fluoranthene
		6270	Fluorene
		4835	Hexachlorobutadiene
		4895	Isopropyl alcohol (2-Propanol, Isopropanol)
		4900	Isopropylbenzene (Cumene)
		5240	m+p-xylene
		5000	Methyl tert-butyl ether (MTBE)
		4965	Methylcyclohexane
		4975	Methylene chloride (Dichloromethane)
		5005	Naphthalene
		4825	n-Heptane
		4855	n-Hexane
		5090	n-Propylbenzene
		5250	o-Xylene
		6615	Phenanthrene
		6665	Pyrene
		5100	Styrene
		5115	Tetrachloroethylene (Perchloroethylene)
		5140	Toluene
		4700	trans-1,2-Dichloroethylene
		5170	Trichloroethene (Trichloroethylene)
		5175	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)
		5235	Vinyl chloride
		5260	Xylene (total)
	EPA TO-17 Modified 2	10312217	Hydrocarbons in Ambient Air Using WMS Passive Sampling Tubes
		5160	1,1,1-Trichloroethane
		5110	1,1,2,2-Tetrachloroethane
		5165	1,1,2-Trichloroethane
		4630	1,1-Dichloroethane



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Air	EPA TO-17 Modified 2		
		4640	1,1-Dichloroethylene
		5150	1,2,3-Trichlorobenzene
		5155	1,2,4-Trichlorobenzene
		5210	1,2,4-Trimethylbenzene
		4610	1,2-Dichlorobenzene
		4635	1,2-Dichloroethane (Ethylene dichloride)
		5215	1,3,5-Trimethylbenzene
		4615	1,3-Dichlorobenzene
		4620	1,4-Dichlorobenzene
		9546	1,4-Dithiane
		4410	2-Butanone (Methyl ethyl ketone, MEK)
		4995	4-Methyl-2-pentanone (MIBK)
		6698	alpha-Pinene
		4375	Benzene
		4455	Carbon tetrachloride
		4475	Chlorobenzene
		4505	Chloroform
		4645	cis-1,2-Dichloroethylene
		4555	Cyclohexane
		6208	d-Limonene
		4750	Ethanol
		4755	Ethyl acetate
		4765	Ethylbenzene
		6774	Halothane (2-Bromo-2-chloro-1,1,1-trifluoroethane)
		5240	m+p-xylene
		4960	Methyl chloride (Chloromethane)
		4990	Methyl methacrylate
		5000	Methyl tert-butyl ether (MTBE)
		5005	Naphthalene
		4825	n-Heptane
		4855	n-Hexane
		5090	n-Propylbenzene
		5250	o-Xylene
		5100	Styrene
		5115	Tetrachloroethylene (Perchloroethylene)
		5140	Toluene
		4700	trans-1,2-Dichloroethylene
		5170	Trichloroethene (Trichloroethylene)
		5235	Vinyl chloride



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Air	EPA TO-3	10312400	Method for the Determination of Volatile Organic Compounds in Ambient Air Using Cryogenic Preconcentration Techniques and Gas Chromatography With Flame Ionization and Electron Capture Detection
	9408 Gasoline range organics (GRO)		
MADEP APH		90017188	Air-Phase Petroleum Hydrocarbons
MADEP APH	9318 1,3-Butadiene		
	3792 APH Aliphatics C5-C8		
	3793 APH Aliphatics C9-C12		
	3794 APH Aromatics C9-C10		
	4375 Benzene		
	4765 Ethylbenzene		
	5240 m+p-xylene		
	5000 Methyl tert-butyl ether (MTBE)		
	5005 Naphthalene		
	5250 o-Xylene		
	5140 Toluene		
Modified EPA TO-17 Passive RAD130 Tube 2		60032351	The Determination of Hydrocarbons in Air Via RAD130 RADIELLO Passive Sample Tubes
	5160 1,1,1-Trichloroethane		
	5110 1,1,2,2-Tetrachloroethane		
	5165 1,1,2-Trichloroethane		
	4630 1,1-Dichloroethane		
	4640 1,1-Dichloroethylene		
	5210 1,2,4-Trimethylbenzene		
	4610 1,2-Dichlorobenzene		
	4635 1,2-Dichloroethane (Ethylene dichloride)		
	5215 1,3,5-Trimethylbenzene		
	4615 1,3-Dichlorobenzene		
	4620 1,4-Dichlorobenzene		
	4410 2-Butanone (Methyl ethyl ketone, MEK)		
	4995 4-Methyl-2-pentanone (MIBK)		
	4375 Benzene		
	4455 Carbon tetrachloride		
	4475 Chlorobenzene		
	4505 Chloroform		
	4645 cis-1,2-Dichloroethylene		
	4555 Cyclohexane		
	4750 Ethanol		
	4755 Ethyl acetate		
	4765 Ethylbenzene		
	5240 m+p-xylene		



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Air	Modified EPA	5000	Methyl tert-butyl ether (MTBE)
	TO-17 Passive	5005	Naphthalene
	RAD130 Tube 2	4825	n-Heptane
		4855	n-Hexane
		5090	n-Propylbenzene
		5250	o-Xylene
		5100	Styrene
		5115	Tetrachloroethylene (Perchloroethylene)
		5140	Toluene
		4700	trans-1,2-Dichloroethylene
		5170	Trichloroethene (Trichloroethylene)
		5235	Vinyl chloride

