DRAFT-FINAL
UNIFORM FEDERAL POLICY - QUALITY ASSURANCE PROJECT PLAN
UNDERGROUND STORAGE TANK PRELIMINARY ASSESSMENT/SITE INSPECTION
SITES CTU006 ; CTU007; CTU008 ; AND CTU011, CTU012, CTU013
GENERAL MITCHELL AIR RESERVE STATION , WISCONSIN

MIDWEST GROUP
BASE REALIGNMENT AND CLOSURE ENVIRONMENTAL CONSTRUCTION OPTIMIZATION SERVICES (BECOS) CONTRACT

Contract Number: FA890316D0053
Task Order: FA890321F1088

Prepared for:

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June 2022

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ABBREVIATIONS AND ACRONYMS

°C	degrees Celsius
>	greater than
≥	greater than or equal to
<	less than
≤	less than or equal to
%	percent
±	plus or minus
µg	microgram
µg/kg	micrograms per kilogram
µg/L	micrograms per liter
µg/m^3	micrograms per meter cubed
AFB	Air Force Base
AFCEC	Air Force Civil Engineering Center
AFRTC	Air Force Reserve Training Center
ARS	Air Reserve Station
B.A.	Bachelor of Arts
B.S.	Bachelor of Science
bgs	below ground surface
BRAC	Base Realignment and Closure
CCB	continuing calibration blank
CCV	continuing calibration verification
CFR	Code of Federal Regulations
CHMM	Certified Hazardous Materials Manager
COC	chain of custody
COPC	contaminant of potential concern
CPM	Certified Project Manager
CTI	CTI and Associates, Inc.
CUES	CTI-URS Environmental Services, LLC
CY	cubic yard
DL	detection limit
DNR	Department of Natural Resources
DO	dissolved oxygen
DOT	Department of Transportation
DOC	dissolved organic carbon
DoD	United States Department of Defense
DPT	direct-push technology
DQO	data quality objective
DRO	diesel range organics
EDD	electronic data deliverable

Eurofins ETA	Eurofins Environment Testing America
EICP	extracted ion current profile
ELAP	Environmental Laboratory Accreditation Program
ERPIMS	Environmental Resources Program Information Management System
FAA	Federal Aviation Administration
FM	Field Manager
ft	foot or feet
gal	gallon
GC	gas chromatograph
GC/MS	gas chromatograph/ mass spectrometer (instrument) or gas chromatography/ mass spectrometry (analysis)
GMIA	General Mitchell International Airport
GRO	gasoline range organics
HASP	health and safety plan
HCl	hydrochloric acid
ICAL	initial calibration
ICP/MS	inductively-coupled plasma/ mass spectrometry
ICV	initial calibration verification
ID	identification or ion detector
IDW	investigation-derived waste
ILHR	Wisconsin Department of Industry, Labor, and Human Relations
IRP	Installation Restoration Program
IS	internal standard
LCS	laboratory control spike
LOD	limit of detection
LOQ	limit of quantitation
LUC	Land Use Controls
MB	method blank
MCL	maximum contaminant level
MD	matrix duplicate
mL	milliliters
mm	millimeters
MPC	measurement performance criteria
MS	matrix spike
MSD	matrix spike duplicate
NA	not applicable
ORP	oxidation reduction potential
OWS	oil-water separator
PA	Preliminary Assessment
PAH	polycyclic aromatic hydrocarbons

PAL	project action limit
PARCCS	precision, accuracy, representativeness, comparability, completeness, and sensitivity
pH	potential hydrogen
PID	photo-ionization detector
PM	Project Manager
POL	Petroleum, Oil, and Lubricants
PPE	personal protective equipment
PSL	Project Screening Level
PVC	polyvinyl chloride
QA	quality assurance
QC	quality control
r^2	regression factor
RPD	relative percent difference
RRT	relative retention time
RSD	relative standard deviation
RT	retention time
RTW	Reserve Training Wing
S2BVM	Manual Stage 2B Validation
S4VM	Manual Stage 4 Validation
SAIC	Science Applications International Corporation
SI	site inspection
SOP	standard operating procedure
SSHO	Site Safety and Health Officer
SW-846 6020B	Metals by Ion Chromatography/ Mass Spectrometry
SW-846 8260D	Volatile Organic Compounds by Gas Chromatography/ Mass Spectrometry
SW-846 8270 SIM	Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry with Select Ion Monitoring
TCG	Troop Carrier Group
TO	Task Order
TOC	total organic carbon
U.S.	United States
USCS	Unified Soil Classification System
UST	underground storage tank
UFP-QAPP	Uniform Federal Policy – Quality Assurance Project Plan
URS	URS Corporation
USEPA	United States Environmental Protection Agency
QSM	Quality System Manual
VI	vapor intrusion
VOC	volatile organic compound

WAC	Wisconsin Administrative Code
WDNR	Wisconsin Department of Natural Resources

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INTRODUCTION

This Uniform Federal Policy – Quality Assurance Project Plan (UFP-QAPP) presents the technical approach, sampling and analysis plan, project data quality objectives (DQOs), and the quality assurance (QA)/quality control (QC) processes that will be conducted during site inspection (SI) activities at three former underground storage tank (UST) locations located at the former General Mitchell Air Reserve Station (ARS), including:

- CTU006 (UST 212),
- CTU007 (UST 215 [Tank 2]),
- CTU008 (UST219); and,
- CTU011, CTU012, and CTU013 (UST 8002).

This UFP-QAPP has been prepared under a contract between the United States (U.S.) Air Force Civil Engineer Center (AFCEC) and CTI-URS Environmental Services, LLC (CUES); Contract No. FA890316D0053, Task Order (TO): FA890321F1088.

The procedures outlined in this UFP-QAPP will serve to verify that 1) the DQOs for this project are met, (2) the field sampling protocols are documented and reviewed in a consistent manner, and (3) the data collected are scientifically valid and defensible. This document has been prepared using the Optimized UFP-QAPP Worksheets that were developed by the Intergovernmental Data Quality Task Force comprised of the U.S. Environmental Protection Agency (USEPA), U.S. Department of Defense (DoD), and U.S. Department of Energy in March 2012.

Project Purpose and Scope

The primary objective of this TO is to complete a Site Inspection (SI) at each of the four sites to confirm the presence and nature, or absence, in soil, groundwater and soil vapor of contaminants of potential concern (COPC) at concentrations exceeding the project screening levels (PSLs). Contamination was documented during tank removal activities conducted in 1994, 1995 and 1998. The SI will include soil borings; installing temporary monitoring wells; installing temporary soil vapor probes; and collecting soil, groundwater and soil vapor samples. COPCs at the sites include volatile organic compounds (VOCs), polynuclear aromatic hydrocarbons (PAHs), and/or lead. The PSLs, listed in Worksheets 15B through 15F, are Wisconsin Department of Natural Resources (WDNR) soil, groundwater and vapor action limits.

History and Background

Former General Mitchell ARS is located approximately 7 miles south of downtown Milwaukee, Wisconsin, on the southern portion of General Mitchell International Airport (GMIA) (**Figure 1**). The base was initially established in February 1952 by the U.S. Air Force as General Billy Mitchell Field when the 924th Reserve Training Wing (RTW) was activated at GMIA. The 924th RTW was redesignated as the 438th Fighter Bomber in July 1952 and then redesignated again to the 247th Air Force Reserve Training Center (AFRTC). In November 1957, the 247th AFRTC was deactivated when the 440th Troop Carrier Wing was transferred to GMIA where C-119 aircraft were assigned to the wing. The C-119 aircraft were replaced in 1971 by C-130A aircraft.

The 440th Tactical Airlift Wing was developed from the 440th Troop Carrier Group (TCG) which began in Baer Field, Indiana in 1943. The TCG was deactivated in October 1945 and was reactivated in August 1947 as a reserve organization (440th TCG), and then expanded in 1949. The mission of the 440th TAG was to combat-airlift support, paratroop and equipment drops, airlift of troops and equipment to forward areas, and aeromedical evacuation (SAIC, 1991).

General Mitchell ARS officially closed on 2 February 2008 as part of the 2005 Base Realignment and Closure (BRAC) Commission, and all personnel in the 440th Airlift Wing at General Mitchell ARS were transferred to Pope Air Force Base (AFB) in North Carolina. A redevelopment plan was conducted in 2008 by RKG Associates, Inc. to create future uses for the buildings located on General Mitchell ARS. Currently, the former General Mitchell ARS is home to Milwaukee County's MKE Regional Business Park. The MKE Regional Business Park leases hangar and office space in support of GMIA and other customers.

Summary of Preliminary Assessment

Figure 1 shows the location of the former ARS on GMIA. The ARS is located along the southern border of the airport's property line, directly west of runway 19-R and 1-L and east of South Howell Avenue and includes approximately 102 acres consisting mostly of concrete/asphalt drive pads, buildings and aircraft hangars. **Figure 2** shows the locations of Sites CTU006, CTU007, CTU008, and CTU011, CTU012, and CTU013 at the former General Mitchell ARS. The Site locations are spread out across the former ARS property, residing approximately in the property's center and along the north and south borders.

Site CTU006 (UST 212)

Site CTU006 is the location of former 1,000-gallon (gal) UST 212 that contained diesel fuel for an emergency generator. UST 212 was located between Buildings 212 and 209 which are in the center of the former General Mitchell ARS property, west of the main concrete drive pad. UST 212 was installed in 1982 and removed in October 1995 after the tank was determined to be out of compliance for the lack of leak detection on the piping. The Wisconsin Department of Industry, Labor, and Human Relations (ILHR) (presently known as the Wisconsin Department of Workforce Development since 1996) regulated this tank system under regulation ILHR 10 which requires leak detection for tank and pipes by 1992. With the tank being out of compliance and also no longer needed, it was decided to remove the tank entirely which occurred in October 1995.

During the excavation for UST 212 removal, soil impacts were encountered in a small area along the UST piping that runs to the generator, but no soil was removed or tested outside of obtaining photoionization detector (PID) measurements. The tank excavation depth was 10 feet (ft) and the piping excavation depth was between 2 and 3 ft deep. No groundwater was encountered while excavating and the soil was recorded as a fine sand.

Site CTU007 (UST 215)

Site CTU007 formerly held a 15,000-gal UST (UST 215, or Tank 2 in this area) that contained back-up fuel for the central heat plant (Building 215). There were three tanks previously in this area, which is centrally

located on the former General Mitchell ARS, two 15,000-gal tanks, and one 20,000-gal tank. The two 15,000-gal tanks (Tank 2 and Tank 3) were placed south of former Building 215 and north of 5th Avenue below a grassy area and the 20,000-gal tank (Tank 1) was placed in line with the other two tanks, but south of 5th Avenue below a grassy area. Tanks 2 and 3 were installed in 1956 and Tank 1 was installed in 1982, all three tanks were removed in March 1998. The tanks were removed for the same reason stated for UST 212 (requirements under regulation ILHR 10 were not met due to lack of leak detection in the tank's piping).

Discolored soils and a noticeable petroleum-like odor was observed during the excavation of Tank 2 in the area of the tank's piping run. Soil samples were collected in this area and analytical results indicated concentrations of diesel range organics (DRO) to be 48 milligrams per kilogram (mg/kg). It was recommended in the Tank Closure Report for Tank 2 (UST 215) created in 1998 that approximately 250 cubic yards (CY) of impacted soil in the area of the piping run be removed in order for the site to meet cleanup criteria and WDNR requirements. No additional soil has yet been excavated at this site and the excavation in March 1998 was backfilled using clean fill (#2). Perched groundwater was encountered during this excavation.

Site CTU008 (UST 219)

Site CTU008 was the location of a 6,000-gal UST (UST 219) that contained diesel fuel for vehicles before the tank was removed in October 1995. The tank was installed in 1976 near the northern border of the former General Mitchell property line, directly east of Building 219 in a grassy area. The tank was removed for the same reason stated for UST 212 and 215 (requirements under regulation ILHR 10 were not met due to lack of leak detection in the tank's piping).

The excavation depth was 11 ft in the tank area and between 2 and 3 ft along the piping length during the tank removal process. Groundwater was encountered during excavation between approximately 10 and 11 ft. Impacted soil was observed at the bottom of the excavation, 11 ft below ground surface (bgs), and no discoloration was observed on the sides of the excavation. A groundwater grab sample was collected at this time. Soil in this area was noted as sand with traces of fines and gravel. The soil sample collected near the east end of UST 219 had an elevated PID reading which indicates that a leak may have occurred in this area. The laboratory analysis of this sample (S-1) indicated a concentration of benzene at 31 micrograms per kilogram (ug/kg) which exceeds the current WDNR soil-to-groundwater pathway RCL of 5.1 ug/kg. Other VOCs were detected but at concentrations below all RCLs.

Sites CTU011, CTU012, and CTU013 (UST 8002)

Site CTU011, CTU012 and CTU013 is located in the POL area, which is in the southeastern corner of the former General Mitchell ARS property. This site contained a UST system (UST 8002) comprised of:

- Three 5,000-gal fuel USTs (one tank stored leaded gasoline, one tank held unleaded gasoline and a third tank held diesel fuel;
- JP-4 fuel piping that ran from each tank to a fueling island/dispensers that was south of the USTs; and,

- One 550-gal oil-water separator (OWS) UST that was installed in 1982.

The entire UST system was removed in September and October 1994 due to the same reason stated for UST 212, 215, and 219 (requirements under regulation ILHR 10 were not met due to lack of leak detection in the tank's piping), but two spills were reported in this area prior to removal. WDNR responded to a JP-4 jet fuel spill on 16 April 1991 and a spill involving approximately 10 gallons of acetic acid (20 percent [%] solution) was reported to the City of Milwaukee Environmental Health Department on 7 April 1993. The spill involving acetic acid was remediated using absorbent pads and baking soda (Harenda, 1994).

During the UST system removal, petroleum impacted soil was observed (soil staining and strong odor) under the southern edge of the concrete drive pad in the site's area on 18 August 1994. A soil sample was collected in this area using a PID and was analyzed for DRO and gasoline range organics (GRO). The soil sample analytical results indicated a DRO concentration of 46 mg/kg and a GRO concentration of 2,400 mg/kg with the historical generic Wisconsin Residual Contaminant Levels (RCL) for GRO and DRO being 100 mg/kg (for coarse-grained soil; 250 mg/kg for fine grained soil). There are currently no RCLs for GRO or DRO in Wisconsin. According to historical documentation, it was believed that a prior spill occurred in this area impacted the gravel fill below the concrete pad and the native clay soils below the gravel fill. Over-excavation of the observed impacted area was conducted in September 1994, but the extents of impact were not explored. More impacted soil (petroleum odors and staining) was found in the excavation of the four USTs and the abandonment of approximately 100 ft of the JP-4 underground pipeline. The majority of petroleum impacted soil was believed to be removed at the time of over-excavation with a total of approximately 5,800 tons of soil removed and disposed of.

QAPP WORKSHEETS #1 & #2, TITLE AND APPROVAL PAGE

Site Name/Project Name: UST PA/SI for Sites CTU006 (UST 212), CTU007 (UST 215 [Tank 2]), CTU008 (UST 219), CTU011, CTU012, and CTU013 (UST 8002).

Site Location: General Mitchell ARS, Milwaukee, Wisconsin

Contract/Work Assignment Number: FA890316D0053, TO: FA890321F1088

Lead Organization:

Air Force Civil Engineer Center
2261 Hughes Avenue, Building 171, Suite 155
Joint Base San Antonio – Lackland Texas 78236
Project Manager: Kay Grosinske
Email: Kay.Grosinske@us.af.mil

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Signature Date

Preparer:

CUES Environmental Services, LLC
34705 West 12 Mile Road, Suite 230
Farmington Hills, Michigan 48331
CUES Project Manager: Jeremy Bennett
Email: jbennett@cticompanies.com

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Signature Date

Relevant Plans and Reports :

Air Force Civil Engineering Center (AFCEC), 2021. *Statement of Work for Architect-Engineering (A-E) Services to Support Underground Storage Tank (UST) Preliminary Assessment/Site Inspection (PA/SI)*. Former General Mitchell Air Reserve Station. July 2021.

Department of Defense (DoD), 2019a. *Consolidated Quality Systems Manual (QSM) for Environmental Laboratories. DoD Quality Systems Manual Version 5.3*.

DoD, 2019b. *General Data Validation Guidelines*. 4 November.

DoD, 2020. *Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by GC/MS*. 11 May.

Hallberg, George R., 1978. *Standard Procedures for Evaluation of Quarternary Materials in Iowa, Part 5 - Standard Weathering Zone Terminology for the Description of Quarternary Sediments in Iowa, Iowa Geological Survey, Technical Information Series, No. 8*.

Harenda Enterprises (Harenda), 1994. *Phase One Site Assessment Report for Underground Storage Tank Removed at 440th Airlift Support Group, 300 East College Avenue, Milwaukee, WI, 53097*. Former General Mitchell Air Reserve Station. November 1994.

Science Applications International Corporation (SAIC), 1991. *Final Remedial Investigation Report, Volume I*. Former General Mitchell Air Reserve Station. October 1991.

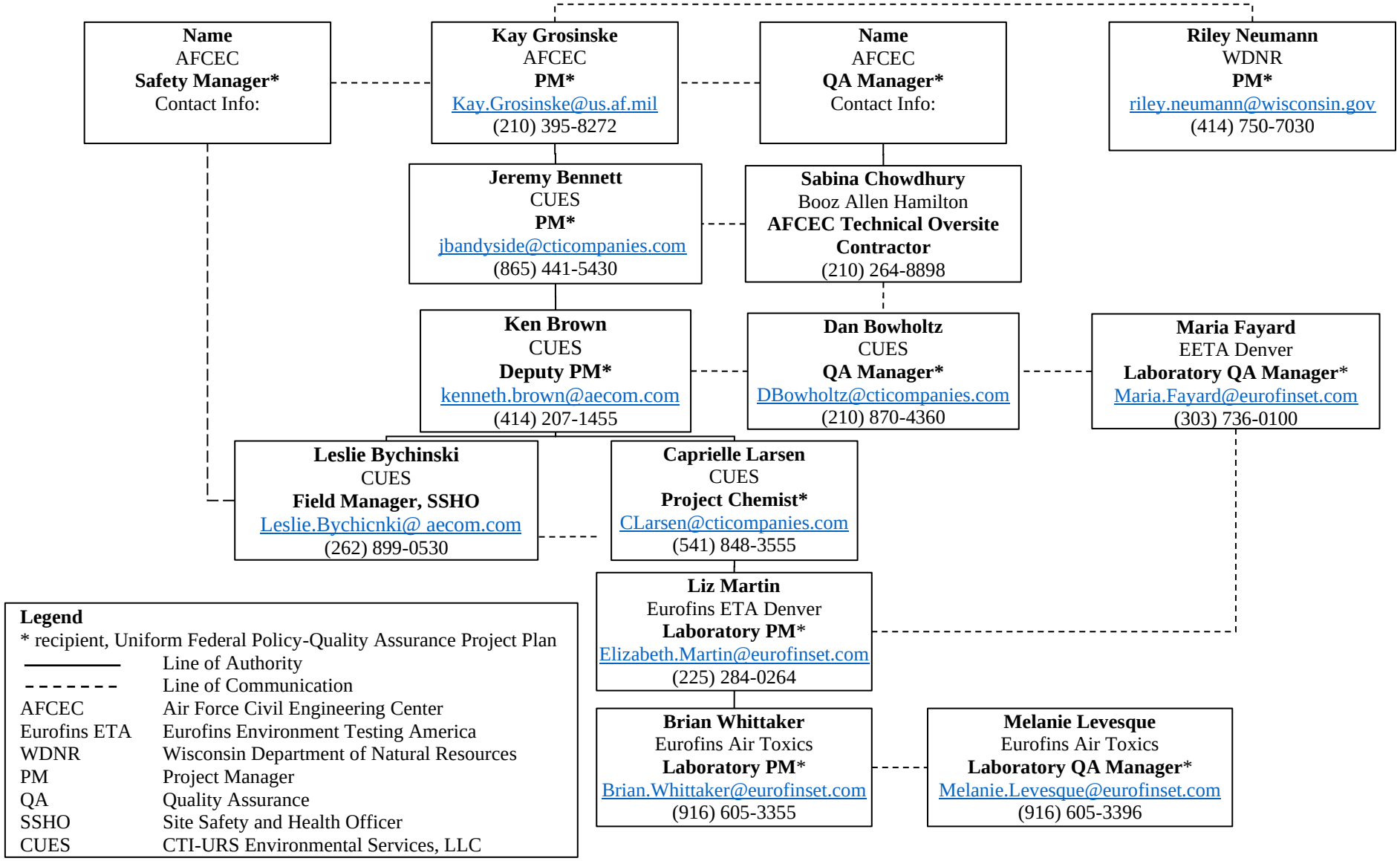
Tony's Cement Works (TCW), 1995. *Underground Storage Tank Removal*. General Mitchell International Airport, Milwaukee, Wisconsin. November 1995.

United States Environmental Protection Agency (USEPA), 1992. *Guide to Management of Investigation-Derived Wastes*. 9345.3-03FS. April.

USEPA, 1996. *Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures*. EPA/540/S-95/504. April.

USEPA, 2002. Guidance on Choosing a Sampling Design for Environmental Data Collection, for Use in Developing a Quality Assurance Project Plan. EPA/240/R-02/005. December.

QAPP WORKSHEET #3 & #5: PROJECT ORGANIZATION AND QAPP DISTRIBUTION



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

Organization: Prime Contractor, CUES

Name	Project Title/Role	Education/Experience	Special Training/Certifications	Signature/Date*
Jeremy Bennett	Project Manager	B.S. Environmental Science / 20 years	CHMM, CPM	
Ken Brown	Deputy Project Manager	B.S. Environmental Science / 29 years		
Leslie Bychinski	Field Manager / SSHO	M.S. Hydrogeology / 2 years		
Dan Bowholtz	Quality Assurance Manager	B.S., M.S Civil Engineering/ 30+ years	P.E., USACE CQM Training	
Caprielle Larsen	Project Chemist, Data Manager	B.A. Chemistry/ 7 years	ERPToolsX Training 2021, 2022	
Rachael Brill	Project Engineer	B.A. Biosystems Engineering/5 years	EIT	

Notes:
*Signatures indicate personnel have read and agree to implement this Uniform Federal Policy-Quality Assurance Project Plan (UFP-QAPP) as written.
B.A. – Bachelor of Arts
B.S. – Bachelor of Science
CHMM – Certified Hazardous Materials Manager
CPM – Certified Project Manager
CUES – CTI-URS Environmental Services, LLC
CQM – Construction Quality Manager
EIT – Engineer-in-Training
M.S. – Master of Science
NA – not applicable
SSHO – Site Safety and Health Officer
USACE – United States Army Corps of Engineers

Organization: Analytical Laboratory, Eurofins Environment Testing America in Denver, Colorado

Name	Project Title/Role	Education/Experience	Special Training/ Certifications	Signature/Date*
Liz Martin	Laboratory Project Manager	B.S. Biology/ 15 years project management, 7 years laboratory	NA (none required)	
Maria Fayard	Quality Assurance Manager	B.S. Biology/ 12 years Quality Assurance experience	NA (none required)	

Notes:
*Signatures indicate personnel have read and agree to implement this UFP-QAPP as written.
B.S. – Bachelor of Science
NA – not applicable

Organization: Analytical Laboratory, Eurofins Air Toxics in Folsom, Colorado

Name	Project Title/Role	Education/Experience	Special Training/ Certifications	Signature/Date*
Brian Whittaker	Laboratory Project Manager	15 years laboratory experience/ 7 years Project Management	NA (none required)	
Melanie Levesque	Quality Assurance Manager	M.S. Chemistry/ 23 years of experience	NA (none required)	
Sepideh Saeed	Business Unit Manager	B.S. Biochemistry/ 24 years of experience	NA (none required)	

Notes:
*Signatures indicate personnel have read and agree to implement this UFP-QAPP as written.
B.S. – Bachelor of Science
NA – not applicable
M.S. – Master of Science

QAPP WORKSHEET #6: COMMUNICATION PATHWAYS

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Regulatory agency interface	AFCEC	Kay Grosinske, BRAC Base Environmental Coordinator	(210) 395-8272 Kay.Grosinske@us.af.mil	Communication with AFCEC will be on-going, throughout the project. Requests for document review will be made via email or hard copy.
UFP-QAPP modification	CUES	Jeremy Bennett, Project Manager	(865) 441-5430 jbennett@cticompanies.com	The CUES PM will communicate with the AFCEC PM and the CUES field team regarding modification of the project UFP-QAPP. Once approved, the revised UFP-QAPP will be distributed to recipients designated in Worksheet #3 & 5.
Onsite/real time UFP-QAPP minor modifications	CUES	Jeremy Bennett, Project Manager	(865) 441-5430 jbennett@cticompanies.com	CUES PM will communicate with AFCEC PM regarding any issues that may result in deviations from the approved UFP-QAPP. Examples are: unable to use the UFP-QAPP specified sampling method like low-flow groundwater sampling from a well with slow recharge, a situation that calls for deviation from UFP-QAPP. Communication will be via telephone to make real-time decision. Where appropriate, verbal communications will be documented and included in the final report.
	CUES	Leslie Bychinski, Field Manager	(262) 899-0530 Leslie.Bychicnki@ aecom.com	
Delays/changes in field work	CUES	Jeremy Bennett, Project Manager	(865) 441-5430 jbennett@cticompanies.com	CUES Field Staff will communicate with CUES PM regarding any issues that may result in delays or changes in the fieldwork. Examples are: breakdown of field analytical instruments (e.g., water quality meter); break down of sampling equipment, equipment not available, etc. The Field Manager will be responsible to inform CUES PM of any problems with activities under their control that will influence the specified field activity schedules. They will inform CUES PM of the length of anticipated delays, or any changes they would like to recommend that will expedite the resolution of the issues to minimize the delay. CUES Field Manager will communicate with CUES PM to discuss the issues and concurrence/approval of the resolutions recommended. CUES PM will (i) either approve the resolutions recommended at the time or (ii) consult with AFCEC PM and AFCEC QAO, for approval of recommendations made, by telephone to expedite continuation of the field work. Where appropriate, verbal communications will be documented and included in the final report.
		Leslie Bychinski, Field Manager	(262) 899-0530 Leslie.Bychicnki@ aecom.com	
Stop Work Order	CUES	Jeremy Bennett, Project Manager	(865) 441-5430 jbennett@cticompanies.com	CUES PM and CUES Field Manager/SSHO will have the authority to stop work if non-compliance to field QC protocols as specified in the QAPP occurs or if there is a breach of safety protocols as specified in Health and Safety Plan. CUES SSHO will communicate with CUES PM informing them: (i) of stop work order, (ii) circumstances under which the order was given, and (iii) the anticipated time when the field work will resume. CUES PM will notify the AFCEC PM about the stop work order. Communications will include circumstances under which the order was given, corrective action(s) being implemented and anticipated timeframe within which the field work will resume. To expedite resumption of field activities, the communication will be via telephone or e-mail. All communication will be documented and included in the final report.
		Leslie Bychinski, Field Manager	(262) 899-0530 Leslie.Bychicnki@ aecom.com	
Sample Receipt Variance	Eurofins ETA Denver	Liz Martin, Laboratory PM	Elizabeth.Martin@eurofinset.com (225) 284-0264	Within 24 hours of sample receipt, Eurofins ETA will notify CUES Project Chemist and PM via telephone or email.
Laboratory Quality Control Variances, Analytical Issues	Eurofins ETA Denver	Liz Martin, Laboratory PM	Elizabeth.Martin@eurofinset.com (225) 284-0264	The Eurofins ETA PM will communicate with CUES Project Chemist and PM, via telephone or e-mail, any issues that may impact data quality or may result in delay in project schedule. Examples are: missed holding times, out of control QC recoveries due to matrix effect, etc. Eurofins ETA will work with CUES Project Chemist and PM to determine corrective action. CUES PM will communicate with AFCEC PM to discuss the issues with analytical protocols and decisions made. Where appropriate, the communication will be documented and included in the project analytical data package.

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Analytical Corrective Actions	Eurofins ETA Denver	Kimberly Drag, Laboratory QAO	Kimberly.Drag@eurofinset.com (704) 607-7735	Eurofins ETA Denver will initiate and oversee corrective action investigations and implementation, for issues originating at the Denver laboratory. Eurofins ETA will inform CUES within 24 hours of initiating a corrective action investigation and will submit the corrective action report via email within one week of the investigation. If Eurofins ETA Denver is notified of a corrective action by Eurofins Air Toxics, they will notify the CUES team within 24 hours of being informed.
Sample Receipt Variance	Eurofins Air Toxics	Brian Whittaker, Laboratory PM	Brian.Whittaker@eurofinset.com (916) 605-3396	Within 24 hours of sample receipt, Eurofins Air Toxics will notify Eurofins ETA Denver Lab PM via telephone or email, who will relay the information to the CUES team within 24 hours of receiving notification.
Laboratory Quality Control Variances, Analytical Issues	Eurofins Air Toxics	Brian Whittaker, Laboratory PM	Brian.Whittaker@eurofinset.com (916) 605-3396	The Eurofins Air Toxics PM will notify the Eurofins ETA PM. In turn, the Eurofins ETA PM will communicate with CUES Project Chemist and PM, via telephone or e-mail, any issues that may impact data quality or may result in delay in project schedule. Examples are: missed holding times, out of control QC recoveries due to matrix effect, etc. Eurofins ETA will work with CUES Project Chemist and PM to determine corrective action. CUES PM will communicate with AFCEC PM to discuss the issues with analytical protocols and decisions made. Where appropriate, the communication will be documented and included in the project analytical data package.
Analytical Corrective Actions	Eurofins Air Toxics	Melanie Levesque	Melanie.Levesque@eurofineset.com (916) 605-3396	Eurofins Air Toxics will initiate and oversee corrective action investigations and implementation, for issues originating at their facility. Eurofins Air Toxics will inform Eurofins ETA Denver within 24 hours of initiating a corrective action investigation and will submit the corrective action report via email within one week of the investigation.
Data Verification Issues (incomplete records/forms)	CUES	Caprielle Larsen, Project Chemist	clarsen@cticompanies.com (541) 848-3555	The CUES Project Chemist will resolve verification issues via email with the laboratory within one week of identification.
Data Validation Issues (non-compliance with DoD QSM)	CUES	Caprielle Larsen, Project Chemist	clarsen@cticompanies.com (541) 848-3555	The CUES Project Chemist will resolve data validation or DoD QSM non-compliance issues via email with the laboratory, within one week of identification. Resolution may include requesting a corrective action investigation.

Notes:
AFCEC – Air Force Civil Engineering Center
BRAC – Base Realignment and Closure
CUES – CTI-URS Environmental Services, LLC
Denver – Denver, Colorado
DoD QSM – United States Department of Defense Quality Systems Manual
Eurofins ETA – Eurofins Environment Testing America
PM – Project Manager
QAO – Quality Assurance Officer
QC – quality control
SSHO – Site Safety and Health Officer
UFP-QAPP – Uniform Federal Policy-Quality Assurance Project Plan

QAPP WORKSHEET #9: PROJECT PLANNING SESSION SUMMARY

Date of Session: 19 October 2021

Location: Conference Call

Purpose: Discuss project controls/setup, personnel introductions and project background and objectives at Former General Mitchell ARS sites. Fieldwork is anticipated to begin in Spring 2022.

Participants:

Name	Organization	Title/Role	Email/Phone
Cybelle Castillo	AFCEC	Contracting Officer	cybelle.castillo.1@us.af.mil
Dave Gibson	AFCEC	Contracting Officer Representative	david.gibson.2@us.af.mil
Kay Grosinske	AFCEC	BRAC Environmental Coordinator	Kay.Grosinske@us.af.mil (210) 395-8272
Sabina Chowdhury	Booz Allen Hamilton	AFCEC Technical Oversight Contractor	chowdhury_sabina@bah.com (210) 264-8898
Jeremy Bennett	CUES	CTI Project Manager	jbennett@cticompanies.com (865) 441-5430
Matt Handyside	CUES	CTI Business Unit Manager	mhandyside@cticompanies.com (248) 229-5892
Kenneth Brown	CUES	AECOM Project Manager	Kenneth.Brown@aecom.com (865) 441-5430
Paul Sklar	CUES	AECOM Technical Lead	paul.sklar@aecom.com (414) 477-1505
Emily Storm	CUES	CTI Project Engineer	estorm@cticompanies.com

Notes:
AFCEC – Air Force Civil Engineering Center
BRAC – Base Realignment and Closure
CUES – CTI-URS Environmental Services, LLC

10:00 am Eastern Standard Time (EST) - Jeremy Bennett (JB) opened the call. Cybelle Castillo (CC) and JB shared presentation materials with the group via email, which are included as **Attachment 1 and 2**, respectively.

AFCEC began the meeting with introductions, including: Cybelle Castillo (Contracting Officer [CO]), Kayla Hernandez (KH) (Contract Specialist [CS]) was not able to join the call, Dave Gibson (Contracting Officer Representative [COR]), Kay Grosinske (BRAC Environmental Coordinator [BEC]), Sabina Chowdhury (GEITA Support). Once AFCEC introductions were made, CC began the AFCEC presentation.

AFCEC Slide 4: included a discussion on contractual details, including contract number, task order (TO) Number, award amount, contract type, and period of performance (PoP). CC made note that all correspondence (e.g., email) shall include the contract number and/or TO number in the subject line.

AFCEC Slide 5: related to the preparation and submittal of CPSMRs, which shall be prepared monthly. Each CPSMR should be sent to CC, KH, DG, KG, and SC.

AFCEC Slide 6: included a discussion on notification requirements. The major emphasis was that all critical issues should be orally communicated to the COR immediately with written communication following as soon as possible. In addition, if a PoP extension is required, CUES must notify the CO, CS and COR in writing at least 60 days prior to PoP end date. The notice can be included with the CPSMR. All email correspondence shall include the CS and COR.

AFCEC Slide 9: the awarding CO was MSgt Jennifer Dantzler; however, she has been reassigned and CC is the administrative CO. KH was the awarding CS and remains in this role. DG is the COR, Kay Grosinske is the BEC and technical lead on the AFCEC side of the project. If immediate communication is necessary, he can be reached on his cell phone at: 210-367-2625.

AFCEC Slide 10: Related to invoicing. the best process is to send the draft invoice with the CPSMR to the CO, CS, and COR for approval. Once approved, it can be uploaded into the WAWF system for final approval.

AFCEC Slide 11: project closeout will include a report, release of claims, and a final invoice. The final invoice shall include "final invoice" and a zero balance.

Following the AFCEC presentation, JB begins introductions for CUES, including Chris Winkeljohn (Program Manager) was not able to make the call, JB (CTI Project Manager [PM]), Matt Handyside (CTI Business Unit Manager), Caprielle Larsen (CTI Project Chemist) was not able to attend the meeting, Emily Storm (CTI Project Engineer), Ken Brown (KB) (AECOM PM), Paul Sklar (AECOM Technical Lead). JB then indicated that he would serve as the overall PM and be the main POC for the contract and then KB will be more of the day-to-day PM. KB added that he would be more of the technical PM.

CUES Slide 4: summarized project background and objectives. The project includes conducting PA/SIs at four UST sites (UST 212, 215, 219, and 8002). Investigations will be conducted at these sites to determine the overall extent of any soil or groundwater impacts and determine the path forward. The PA will be conducted to assess site conditions, identify data gaps, and evaluate the necessary steps to achieve Site Closure (SC). Part of the evaluation will be to determine if additional data collection is necessary to support SC or if current data would allow for administrative closure. Separate administrative site closure packages will also be prepared for sites CTU001 (OWS 104), CTU014 (OWS 308), and CTU015 (West Ditch).

CUES Slide 5: outlined the statement of work that includes the following:

- Task 1 – Project Management (WBS, PPC, CPSMRs, and meetings). Meetings will include the kickoff meeting, two PMRs that will be attended either virtually or in-person, two on-site technical meetings, and eight BCT meetings via teleconference and two on-site BCT meetings.
- Task 2 – TO Planning (on-site scoping visit and the QPP, which will include the work plan, field sampling plan, sampling and analysis plan, and health and safety plan).
- Task 3 – PA/SI
- Task 4 – Reporting

CUES Slide 6: discussion of project deliverables, which includes CPSMRs, meeting minutes, QPP, PA/SI Report, GIS SDFIE geodatabase files, and ERPIMs submittals.

CUES Slide 7: the team agreed on the review time assumptions, which include 30 days for AFCEC initial review, 14 days for AFCEC backcheck, and 45 days for Wisconsin DNR review. KG noted that there is not a DIMOA agreement and there is a fee associated with each report. Fieldwork is anticipated to begin in Spring 2022, PA/SI Report is anticipated to be completed in Summer/Fall 2022, and site closure packages in Winter 2022 through Fall 2023.

JB and KG agreed that a scoping meeting will be scheduled at another time. Site access/coordination activities will include contacting Brian Growth as he currently gets us access to most places. We will also need to include the Airport Environmental Manager (Greg Failey) to help us with access to buildings and areas around the buildings.

JB closes the call at 11:30 a.m.

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QAPP WORKSHEET #10: PRELIMINARY CONCEPTUAL SITE MODEL

Installation Description and Characteristics

The Former General Mitchell ARS (former ARS) is approximately 102 acres and operated from 1952 to 1995. The former ARS is situated on the southern portion of the General Mitchell International Airport (GMIA) property, located 7 miles south of downtown Milwaukee. GMIA is owned and operated by Milwaukee County. The ARS was formerly operated concurrently with GMIA. Individual site areas which are the subject of this SI are approximately 1,000-3,500 square feet (ft).

Physical Profile

Topography – The approximate elevation of the former ARS is approximately 700 ft mean sea level (msl). The former installation including the former UST areas are relatively flat.

Vegetation – Low grass vegetation is present adjacent to concrete and building locations. Aquatic vegetation is present on banks surrounding drainage ditches.

Surface Water – The former ARS is located is located within the Oak Creek Watershed. Surface water drains to the north or south towards drainage ditches. Drainage ditches flow beneath the runways and ultimately to Oak Creek, which is located east of the airport. Oak Creek is located approximately one mile east of the eastern edge of the former ARS and discharges to Lake Michigan.

Soils – Soil boring logs are not available for the study area. Based on review of UST removal reports, depending upon the specific area, soils at the former ARS within the upper 20 feet of the subsurface consist of mixtures of poorly graded fine to coarse sand with gravel or silty clay, silt and clay. Some areas of mainly clay soil may make identification of the water table difficult. Fill soil (topsoil) is present in unpaved areas.

Geology – The surficial geology consists of glacial diamicton belonging to the Oak Creek Formation which has a minimum thickness of approximately 100 ft in the GMIA vicinity. The Oak Creek Formation is approximately 90 percent silt and clay. Discontinuous sandy lenses are present throughout the formation and between glacial till deposits. Bedrock is comprised of the Niagara Dolomite and is expected to occur between 100 ft and 150 ft below ground surface.

Hydrogeology – Shallow, perched groundwater is typically found in thin, discontinuous lenses that can consist of mixtures of silty sand, silty clay or gravelly sand. The discontinuous nature of the lenses tends to restrict the horizontal and vertical movement of perched groundwater. Perched groundwater typically occurs within 15 ft of the ground surface. Recent work at the GMIA Fire Department encountered shallow groundwater at depths between 3 and 15 ft below ground. Overall shallow groundwater flow is generally to the east-northeast across the GMIA. Average horizontal hydraulic gradients of approximately 0.002 ft/ft were measured in monitoring wells installed in other areas of the former ARS. Productive groundwater zones are not found within the Oak Creek Formation but tend to occur at the weathered bedrock interface and within the bedrock underlying the region.

Meteorology – The average annual rainfall is approximately 30-inches per year.

Release Profile

The former USTs at each site were removed due to the tanks being out of compliance by lacking a leak detection mechanism for the piping. All USTs previously held fuel of some sort.

Contaminants of Potential Concern – Contaminants of Potential Concern (COPCs) include volatile organic compounds (VOCs), polynuclear aromatic hydrocarbons (PAHs) and lead.

Media of Potential Concern – Soil, groundwater and soil vapor.

Potential and Confirmed Releases

CTU006 is centrally located in the former General Mitchell ARS property (TCW, 1995). CTU006 (UST212) is considered a potential release site based on soil characteristics observed during the UST removal process. UST 212 is a former 1,000-gallon (gal) UST that was located between Buildings 212 and 209 and contained diesel fuel. The UST was installed in 1982 and removed in October 1995. During the UST removal, soil contamination was encountered in a small area along the UST piping. No soil has been removed (AFCEC, 2021).

CTU007 is located near the northwestern corner of the General Mitchell ARS property (TCW, 1995). CTU007 (UST 215, Tank 2) is considered a potential release site based on soil characteristics observed during the UST removal process. UST 215 is a former 15,000-gal UST (Tank 2) that contained fuel oil and was located near Building 215. Tank 2 was installed in 1956 and removed in March 1998. Evidence of soil contamination was observed during the Tank 2 removal along the UST piping run, but no soil was removed at this time (AFCEC, 2021).

CTU008 is located along the northern border of the General Mitchell ARS property line (TCW, 1995). CTU008 (UST 219) is considered a potential release site based on soil characteristics observed during the UST removal process. UST 219 is a former 6,000-gal UST near Building 219 that held fuel for vehicles. The final tank at Building 219, UST 219, was installed in 1976 and removed in October 1995 when contaminated soil was encountered but not removed (AFCEC, 2021).

CTU011, CTU012, and CTU013 (UST 8002) is the location of a confirmed release. This location contained three USTs, all 5,000-gal capacity, near the southeastern corner of the former ARS, where a UST system consisting of the three USTs, a JP-4 service line, and one 550-gal oil-water separator were formerly located (also referred to as the Petroleum, Oil, and Lubricants [POL] Area). All three USTs were installed in 1982. JP-4 jet fuel spills (16 April 1991 and 12 November 1993) and acetic acid spills (7 April 1993) were reported in this area prior to the removal of the three USTs and JP-4 line in September and October 1994. Contamination was discovered along the JP-4 pipe runs, around the USTs, and under the former concrete drive pads during the removal process. Over excavation of the affected area occurred in September and October 1994 when approximately 5,800 tons of impacted soil was removed (AFCEC, 2021).

Contaminant Migration Pathways – Contaminants are sorbed to the soil matrix near the release locations. Migration of dissolved-phase contaminants deeper in the soil column can occur over time, potentially reaching shallow groundwater. Gravity-driven migration of non-aqueous phase liquids (NAPL), if present in the unsaturated zone, can also transport contaminants downward. Dissolved-phase contaminants can migrate vertically and horizontally

in groundwater. Vertical migration can result in transport of contaminants to deeper groundwater if downward vertical gradients are present. When petroleum-related contaminants are exposed on the ground surface, transport to, and discharge into nearby surface water bodies (ditches, creeks) or storm water outfall locations can occur. Dissolved-phase transport of contaminants via groundwater to surface water features can also occur. Vapor-phase contaminants, if present, can migrate in soil and shallow groundwater beneath buildings and can enter buildings through vapor intrusion.

Exposure Pathways and Potential Receptors – Exposure pathways include direct contact with contaminated soil and groundwater via inhalation, ingestion and dermal contact, or via inhalation of vapor-phase VOCs or particulates. As access to the sites are restricted, direct contact exposure to the general public is unlikely but construction worker exposure is possible. Buildings are present nearby USTs 212 and 219 and the vapor intrusion pathway is possible at these locations. Potable water to the area is supplied by the City of Milwaukee, therefore, exposure (i.e. ingestion) to contaminated groundwater is unlikely. Surface water bodies (creeks) could be considered receptors if impacted shallow groundwater is discharging to these bodies.

Secondary Pathways – Disposal of excavated soil can be a secondary migration pathway.

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QAPP WORKSHEET #11: PROJECT/DATA QUALITY OBJECTIVES

The USEPA's seven-step DQO process was used to plan this project and prepare this UFP-QAPP.

Step 1. State the Problem

As discussed in the introduction, several former USTs were used to store gasoline and/or diesel fuel at sites UST 212, UST 215, UST 219, and UST 8002. Impacted soil was only removed at UST 8002 in the POL Area. Impacted soils were found along the UST piping at sites UST 212, UST 215, UST 219 while impacted soil was encountered along the UST pipe runs, around the UST itself, and below the former concrete drive pads in the POL area at site UST 8002 (AFCEC, 2021). Because impacted soils were left in place at these sites and formal investigations have not yet been conducted, an SI will be conducted to confirm the presence and nature, or absence of contamination.

Step 2. Identify the Goals of the Study

The primary objectives of the study are to collect soil, groundwater, and soil vapor samples at each of the former UST sites to confirm the presence and extent within the study area of COPCs in the potentially affected media. COPCs concentrations will be compared to PSLs as described in Worksheet #15. If COPC concentrations are identified in soil, groundwater, and/or soil vapor above the applicable PSLs, then it will be determined that COPC impacts are present at the site and additional actions will be recommended. If COPC concentrations are below applicable PSLs, then the data will be used to support the confirmation of administrative closure.

Step 3. Identify Information Inputs

The following data and information needs are required to achieve the project goals:

- Collection and laboratory analysis of soil and groundwater within each former UST site, and soil vapor samples at the UST 212 and UST 219 sites;
- As-built drawings of the utility infrastructure may be used to evaluate the potential for COPC migration and secondary sources.

Step 4. Define the Boundary of the Study

The physical boundaries of the SI will be restricted to the horizontal and vertical limits of proposed sampling areas encompassing the former UST sites, as summarized below.

- Site CTU006 (UST 212) is centrally located on the former General Mitchell ARS property, just west of the concrete drive pad (**Figure 2**). UST 212 (1,000-gal) was located between Buildings 212 and 209 (**Figure 3**).
- Site CTU007 (UST 215) is also centrally located on the former General Mitchell ARS between B Street and Building 217 (**Figure 2**). Three USTs were previously located in this area: Tank 1 (20,000-gal) was placed in a grassy area south of 5th Avenue and Tanks 2 and 3 (15,000-gal) were placed in a grassy area south of former Building 215 and north of 5th Avenue (**Figure 4**).
- Site CTU008 (UST 219) is positioned along the northern border of the former General Mitchell ARS property line, north of 2nd Avenue (**Figure 2 and 5**). UST 219 (6,000-gal) was formerly located on site CTU008 in the grassy area between Buildings 219 and 220, just north of 2nd Avenue (**Figure 5**).

- Site CTU011, CTU012 and CTU013 is located in the POL Area (UST 8002), which is along the southern border of the former General Mitchell ARS property line (**Figure 2**). The former UST system (three 5,000-gal tanks, JP-4 fuel line, 550-gal oil-water separator and fueling island/dispensers) at this site was located near the western edge of the concrete pad in the POL Area, east of the southern portion of D Street. The three USTs and oil-water separator were placed in the grassy area north of the large concrete pad with the JP-4 line running under the concrete from the USTs to the south where it connected to a fueling island with dispensers (**Figure 6**).

Within these sites, a total of 33 soil borings will be advanced to characterize surface and subsurface soil to a maximum depth of 20 ft bgs. A total of 14 newly installed temporary monitoring wells will be installed to characterize groundwater at the sites. In addition, up to six soil vapor sampling points will be installed for the collection of soil vapor samples because they will be closer to the source of contamination and will indicate whether soil vapors have migrated near buildings. If the soil vapor samples indicate the presence of volatile contaminants, subslab samples may be collected inside nearby buildings. Samples will be selectively analyzed for VOCs, PAHs, and lead. See Worksheet 17 for details.

Step 5. Define the Analytic Approach

Soil, groundwater, and soil vapor samples will be collected and selectively analyzed for:

- VOCs using USEPA Method 8260D (all sites);
- VOCs (soil vapor) using USEPA Method TO-15 (UST 212 and 219)
- PAHs using USEPA Method 8270 with Select Ion Monitoring (all sites); and/or
- Lead using 6020B (UST 8002 only for UST formerly containing gasoline).

The soil sample from UST 8002 will be analyzed for lead as this UST reportedly contained leaded gasoline.

WDNR groundwater, soil and vapor standards can be found at the following locations and are included in Worksheet 17:

- Wisconsin Administrative Code Chapter NR140 Groundwater Quality Standards: https://docs.legis.wisconsin.gov/code/admin_code/nr/100/140
- Wisconsin Administrative Code Chapter NR720 Soil Residual Contaminant Levels: https://docs.legis.wisconsin.gov/code/admin_code/nr/700/720 and Soil Residual Contaminant Levels Determinations using the USEPA Regional Screening Level Web Calculator
- WDNR Publication RR-890, Soil Residual Contaminant Level Determinations Using The U.S. EPA Regional Screening Level Web Calculator, January 2018; https://dnr.wi.gov/DocLink/RR/RR800.pdf

For this SI, sample concentrations will be compared to the following WDNR screening values:

- Soil: Non-Industrial Direct Contact and Soil-Groundwater Pathway RCLs
- Groundwater: Public Health Groundwater Quality Standards; and,
- Soil Vapor: Residential and Small Commercial Structures Vapor Risk Screening Levels.

Step 6. Specify Performance or Acceptance Criteria

Data will meet UFP-QAPP-specified precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS) requirements, as detailed below.

- The Field Manager (FM) will verify UFP-QAPP-specified field procedures are followed. Samples will be collected from pre-determined locations (Worksheet #18) with accompanying field quality control (QC [Worksheet #20]) in accordance with Standard Operating Procedures (SOPs) (Worksheet #21), using appropriate sample containers and methods of preservation (Worksheet #19 & 30). Deviations will be documented immediately and communicated via pathways described in Worksheet #6.
- Field data will be reviewed by the CUES Field Manager in comparison to UFP-QAPP Worksheets #19 & 30, #20, #21, #22, and #25. Deviations will be documented immediately upon discovery, and the project team will evaluate impacts to PARCCS elements.
- The subcontract laboratory will adhere to analytical SOPs (Worksheet #23), method-specified acceptance criteria, and requirements of the DoD Quality Systems Manual (QSM) (DoD, 2019a) Version 5.3 or later (Worksheets #23-25 and #28).
- The CUES Project Chemist will perform Stage 2B data validation on 100% of analytical results from field samples, as defined in the DoD General Data Validation Guidelines (DoD, 2019b). Validation will be informed by the DoD QSM, the DoD General Data Validation Guidelines, and DoD Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by gas chromatography/mass spectrometry (GC/MS) (DoD, 2020), and worksheets in this UFP-QAPP (Worksheets #12, #15, #19 & 30, #24, #28, and #34-36).
- Results of the data validation report will be used by the project team to determine data usability, in accordance with Worksheet #37. Data usability will be evaluated based on the desired usage of the data and the specific impacts indicated by validation qualifiers.
    - Data points qualified as being potentially biased high or impacted by blank contamination (i.e., J+, B, or Q) may be considered estimated maximum concentrations within the samples they represent.
    - Detected results qualified as being potentially biased low (i.e., J- and sometimes Q) may be considered minimum concentrations, and therefore may be used as evidence to confirm the presence of a contaminant.
- The data completeness goal is 90% usable data, in accordance with Worksheet #12.

Step 7: Develop the Plan for Obtaining Data

Data will be generated from the analysis of soil, groundwater, and soil vapor samples that will be collected during the SI at sites UST 212, 215, 219, and 8002 as specified in Worksheet #17. Sampling locations are based on historical data, chosen because they potentially represent the highest COPCs and locations with the greatest likelihood to contain PSL exceedances.

Requirements for the analytical design are found in Worksheets #15, #19 & 30, #20, and #24-28.

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QAPP WORKSHEET #12A: MEASUREMENT PERFORMANCE CRITERIA

Matrix: Groundwater and Soil
Analytical Group or Method: Lead by USEPA SW-846 6020B
Concentration Level: Low

Data Quality Indicator	QC Sample or Measurement Performance Activity	Measurement Performance Criteria
Overall Precision	Field Duplicates / Non-Spiked Laboratory Duplicates	Soil: RPD ≤ 40%, Groundwater: RPD ≤ 20% RPD is applicable when both results are detected above the LOQ. Otherwise, the absolute value of the difference between results should be less than the LOQ.
Bias/Contamination	Field Blank / Equipment Rinsate Blank / Trip Blank	Detected results ≤ ½ LOQ, or the detection in the blank is < five times the concentration in the associated field sample.
Bias/Contamination	Method Blank	≤ ½ LOQ
Accuracy/Bias (ideal matrix)	LCS (recovery only)	Soil: 84 – 118%, Groundwater: 88 – 115%
Accuracy/Bias (project matrix)	MS (recovery only)	Soil: 84 – 118%, Groundwater: 88 – 115%. Not valid if the parent sample concentration is >4 times the spike concentration.
Precision	MS Duplicate/ LCS Duplicate	RPD ≤ 20%
Precision	Dilution Test	A five-fold dilution must agree within ±10% of the original measurement. Note: the dilution test is required if the MS/MSD fails and the native undiluted sample concentration is > 50 times the LOQ. The dilution should be performed on the parent sample of the failed MS/MSD if possible.
Accuracy	Post Digestion Spike	Recovery within 80 to 120% of expected results. Note: The post digestion spike is required if the MS/MSD fails and the native unspiked sample concentration is < 50 times the LOQ. The spike should be performed on the parent sample of the failed MS/MSD if possible.
Sensitivity	Internal Standard	Per sample: Intensity within 30 to 120% of the internal standard in the ICAL.
Sensitivity	LOQ of undiluted samples.	Laboratory's LOQ for non-dilute samples should meet PQL Goal criteria in Worksheet #15.
Accuracy	Interference Check Solution	ICS-A: Absolute value of concentration for all non-spiked project analytes <1/2 LOQ, unless they are a verified trace impurity from one of the spiked analytes. ICS-AB: Within ± 20% of true value.
Completeness (field)	Planned verses Collected Samples	90% of planned field and QC samples are successfully collected along with field parameters specified in Worksheet #17, and data are not rejected due to field errors e.g., gross contamination. Calculated as number of collected samples divided by number of planned samples multiplied by 100%.
Completeness (analytical)	Received vs. Accepted Results	90% of analytical results are valid (not rejected) in accordance with Worksheet #36. Calculated as number of valid results divided by number of expected results, multiplied by 100%.

Notes:
≤ – less than or equal to
< – less than
> – greater than
% – percent
± – plus or minus
ICS-A – interference check solution A
ICS-AB – interference check solution AB
LCS – laboratory control sample
LOQ – limit of quantitation
MS – matrix spike
MSD – matrix spike duplicate
PQL – project quantitation limit
QC – quality control
SW-846 6020B – Inductively Coupled Plasma – Mass Spectrometry
USEPA – United States of America Environmental Protection Agency
RPD – relative percent difference

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QAPP WORKSHEET #12B: MEASUREMENT PERFORMANCE CRITERIA

Matrix: Soil and groundwater
Analytical Group or Method: VOCs by USEPA SW-846 8260D
Concentration Level: Low

Data Quality Indicator	QC Sample or Measurement Performance Activity	Measurement Performance Criteria
Overall Precision	Field Duplicates / Non-Spiked Laboratory Duplicates	RPD ≤ 20% when both results are detected above the LOQ. Otherwise, the absolute value of the difference between results should be less than the LOQ.
Bias/Contamination	Field Blank / Equipment Rinsate Blank / Trip Blank	Detected results ≤ ½ LOQ, or the detection in the blank is less than five times the concentration in the associated field sample.
Bias/Contamination	Method Blank	≤ ½ LOQ
Accuracy/Bias (ideal matrix)	LCS (recovery only)	Recoveries within DoD QSM Version 5.3 Table C-24
Accuracy/Bias (project matrix)	MS (recovery only)	Recoveries within DoD QSM Version 5.3 Table C-24. Not valid if the parent sample concentration is >4 times the spike concentration.
Precision	MS Duplicate/ LCS Duplicate	RPD ≤ 20%
Sensitivity	Internal Standards	Per sample: Area counts ±50% from the ICAL midpoint standard. On days when the ICAL is not performed, ±50% from the daily initial CCV.
Sensitivity	LOQ of undiluted samples.	Laboratory's LOQ for non-dilute samples should meet PQL Goal criteria in Worksheet #15.
Accuracy	Surrogate Compounds	Per sample: Recoveries within DoD QSM Version 5.3 Table C-24. If the surrogate compound is not found in Table C-24, use laboratory's statistically derived limits.
Completeness (field)	Planned verses Collected Samples	90% of planned field and QC samples are successfully collected along with field parameters specified in Worksheet #17, and data are not rejected due to field errors (e.g., gross contamination). Calculated as number of collected samples divided by number of planned samples multiplied by 100%.
Completeness (analytical)	Planned vs. Accepted Results	90% of analytical results are valid (not rejected) in accordance with Worksheet #36. Calculated as number of valid results divided by number of expected results, multiplied by 100%.

Notes:
≤ – less than or equal to
> – greater than
% – percent
± – plus or minus
CCV – continuing calibration verification
CVOC – chlorinated volatile organic compound
DoD QSM – United States Department of Defense Quality Systems Manual
ICAL – initial calibration
LCS – laboratory control sample
MS – matrix spike
QC – quality control
RPD – relative percent difference
SW-846 8260D – Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry
USEPA – United States of America Environmental Protection Agency

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QAPP WORKSHEET #12C: MEASUREMENT PERFORMANCE CRITERIA

Matrix: Soil and groundwater
Analytical Group or Method: PAHs by USEPA SW-846 8270 SIM
Concentration Level: Low

Data Quality Indicator	QC Sample or Measurement Performance Activity	Measurement Performance Criteria
Overall Precision	Field Duplicates / Non-Spiked Laboratory Duplicates	RPD ≤ 20% when both results are detected above the LOQ. Otherwise, the absolute value of the difference between results should be less than the LOQ.
Bias/Contamination	Field Blank / Equipment Rinsate Blank	Detected results ≤ ½ LOQ, or the detection in the blank is less than five times the concentration in the associated field sample.
Bias/Contamination	Method Blank	≤ ½ LOQ
Accuracy/Bias (ideal matrix)	LCS (recovery only)	Recoveries within DoD QSM Version 5.3 Table C-42
Accuracy/Bias (project matrix)	MS (recovery only)	Recoveries within DoD QSM Version 5.3 Table C-42. Not valid if the parent sample concentration is >4 times the spike concentration.
Precision	MS Duplicate/ LCS Duplicate	RPD ≤ 20%
Sensitivity	LOQ of undiluted samples.	Laboratory's LOQ for non-dilute samples should meet PQL Goal criteria in Worksheet #15.
Accuracy	Surrogate Compound	Per sample: Recoveries within laboratory's statistically derived limits.
Completeness (field)	Planned verses Collected Samples	90% of planned field and QC samples are successfully collected along with field parameters specified in Worksheet #17, and data are not rejected due to field errors (e.g., gross contamination). Calculated as number of collected samples divided by number of planned samples multiplied by 100%.
Completeness (analytical)	Planned vs. Accepted Results	90% of analytical results are valid (not rejected) in accordance with Worksheet #36. Calculated as number of valid results divided by number of expected results, multiplied by 100%.

Notes:
≤ – less than or equal to
> – greater than
% – percent
± – plus or minus
CCV – continuing calibration verification
DoD QSM Version 5.3 – United States Department of Defense Quality Systems Manual Version 5.3
ICAL – initial calibration
LCS – laboratory control sample
LOQ – limit of quantitation
MS – matrix spike
PQL – project quantitation limit
QC – quality control
RPD – relative percent difference
SW-846 8270 SIM – Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry with Select Ion Monitoring
USEPA – United States Environmental Protection Agency

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QAPP WORKSHEET #12D: MEASUREMENT PERFORMANCE CRITERIA

Matrix: Soil vapor
Analytical Group or Method: VOCs by USEPA Compendium Method TO-15
Concentration Level: Low

Data Quality Indicator	QC Sample or Measurement Performance Activity	Measurement Performance Criteria
Overall Precision	Field Duplicates / Non-Spiked Laboratory Duplicates	RPD ≤ 20% when both results are detected above the LOQ. Otherwise, the absolute value of the difference between results should be less than the LOQ.
Bias/Contamination	Method Blank	≤ ½ LOQ
Accuracy/Bias (ideal matrix)	LCS (recovery only)	Recoveries within DoD QSM Version 5.3 Table C-42
Precision	LCS Duplicate	RPD ≤ 20%
Sensitivity	LOQ of undiluted samples.	Laboratory's LOQ for non-dilute samples should meet PQL Goal criteria in Worksheet #15.
Accuracy	Surrogate Compound	Per sample: Recoveries within laboratory's statistically derived limits.
Completeness (field)	Planned verses Collected Samples	90% of planned field and QC samples are successfully collected along with field parameters specified in Worksheet #17, and data are not rejected due to field errors (e.g., gross contamination). Calculated as number of collected samples divided by number of planned samples multiplied by 100%.
Completeness (analytical)	Planned vs. Accepted Results	90% of analytical results are valid (not rejected) in accordance with Worksheet #36. Calculated as number of valid results divided by number of expected results, multiplied by 100%.

Notes:
≤ – less than or equal to
> – greater than
% – percent
± – plus or minus
CCV – continuing calibration verification
Compendium Method TO-15 – Determination of Volatile Organic Compounds in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/ Mass Spectrometry
DoD QSM – United States Department of Defense Quality Systems Manual
ICAL – initial calibration
LCS – laboratory control sample
LOQ – limit of quantitation
PQL – project quantitation limit
QC – quality control
RPD – relative percent difference
USEPA – United States Environmental Protection Agency

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QAPP WORKSHEET #13: SECONDARY DATA USES AND LIMITATIONS

Data Type	Source	Data uses relative to current project	Factors affecting the reliability of data limitations on data use
Previous Observations in Soil During UST Removal (Site CTU006)	TCW, 1995. *Underground Storage Tank Removal*. General Mitchell International Airport, Milwaukee, Wisconsin. November 1995.	Identification of areas where impacted soil was encountered during excavation activities, specific information about the UST at site CTU006 (UST 212).	None.
Previous Observations in Soil During UST Removal (Site CTU007)	TCW, 1995. *Underground Storage Tank Removal*. General Mitchell International Airport, Milwaukee, Wisconsin. November 1995.	Identification of areas where impacted soil was encountered during excavation activities, specific information about the UST at site CTU007 (UST 215).	None.
Previous Observations in Soil During UST Removal (Site CTU008)	TCW, 1995. *Underground Storage Tank Removal*. General Mitchell International Airport, Milwaukee, Wisconsin. November 1995.	Identification of areas where impacted soil was encountered during excavation activities, specific information about the UST at site CTU008 (UST 219).	None.
Previous Observations in Soil During UST System Removal (Site CTU011, CTU012 and CTU013)	Harenda, 1994. *Phase One Site Assessment Report for Underground Storage Tank Removed at 440th Airlift Support Group, 300 East College Avenue, Milwaukee, WI, 53097*. Former General Mitchell Air Reserve Station. November 1994.	Identification of areas where impacted soil was encountered during excavation activities, specific information about the POL Area UST system and a sequence of events that occurred during removal.	None.
Previous Soil Sampling Results	Harenda, 1994. *Phase One Site Assessment Report for Underground Storage Tank Removed at 440th Airlift Support Group, 300 East College Avenue, Milwaukee, WI, 53097*. Former General Mitchell Air Reserve Station. November 1994.	Identification of chemicals of concern, sampling locations, and sampling results at site CTU011, CTU012 and CTU013.	None.

Notes:
CSM – conceptual site model
TCW – Tony's Cement Works, Inc.
UST – Underground Storage Tank

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QAPP WORKSHEET #14/16: PROJECT TASKS & SCHEDULE

Activity	Responsible Party	Planned Start Date	Planned Completion Date	Deliverable(s)	Deliverable Due Date
Task Order Kickoff Meeting	CUES	Completed	10/19/2021	Kickoff Meeting Minutes	10/19/2021
UFP-QAPP	CUES	In progress	7/30/2022	2022 Draft, Draft-Final and Final versions of UFP-QAPP	7/30/2022
Site Inspection	CUES	9/12/22	9/30/2022	Field Reports and Updates for sampling and well installation activities	9/30/2022
PA/SI Report	CUES	10/12/2022	5/24/2023	Draft, Draft-Final and Final versions of PA/SI Report	5/24/2023
CTU001 (OWS 104) Site Closure Letter	CUES	8/1/2022	1/30/2023	Draft, Draft-Final and Final versions of CTU001 (OWS 104) Site Closure Letter	1/30/2023
CTU014 (OWS 308) Site Closure Letter	CUES	9/12/2022	3/13/2023	Draft, Draft-Final and Final versions of CTU014 (OWS 308) Site Closure Letter	3/13/2023
CTU015 (West Ditch) Site Closure Letter	CUES	10/24/2022	4/24/2023	Draft, Draft-Final and Final versions of CTU015 (West Ditch) Site Closure Letter	4/24/2023

Notes:
CUES – CTI-URS Environmental Services, LLC
PA – Preliminary Assessment
SI – Site Inspection
UFP-QAPP – Uniform Federal Policy-Quality Assurance Project Plan

Site Inspection Activities

The approach to characterizing environmental media at UST 212, 215, 219, and 8002 includes the collection of surface and subsurface soil samples, installation of monitoring wells for the collection of groundwater samples, and the collection of soil vapor samples (Worksheet #18). Fieldwork will be conducted in accordance with SOPs included in **Appendix A**. Additional details related to this SI can be found in **Worksheet #17 and #20**. **Figure 7** provides a schedule of project activities.

Pre-Mobilization Planning

Prior to site mobilization, the following planning and coordination activities will be conducted:

- GMIA Regulations - The work will be performed on a secure airport and the contractor personnel must become familiar with and comply with GMIA regulations. All required badges, passes, and vehicle permits will be acquired with proper authority.
- Federal Aviation Administration (FAA) Obstruction Evaluation – A *Notice of Proposed Construction or Alteration Form* (FAA 7460-1) will be filed with the FAA if equipment height extends above an imaginary surface extending outward and upward at a slope of 100:1 for a horizontal distance of 20,000 ft from the nearest point of the closest runway surface to the planned sample location.
- Utility Clearances – Prior to the commencement of drilling activities, underground utility lines will be located and protected. Drilling operations will not begin until all underground hazards have been located. To assist with utility location, utility maps will be requested from GMIA showing the locations of utilities and other infrastructure features at each site. The following will be conducted as part of this task:
    - Notifying Wisconsin's utility safety notification system (Diggers Hotline); and,
    - Completing 3rd party utility locate and mark-out at all proposed drilling locations.

After pre-mobilization activities are complete, site access obtained, and a determination letter from the FAA obtained (as applicable), mobilization activities can occur. Demobilization will occur once activities associated with the SI are completed and the investigation areas have been restored to original conditions per concurrence of the Airport Manager.

Field Instruments and Equipment

Instruments used to collect, generate, or measure environmental data will be calibrated daily and in such a matter that accuracy and reproducibility of results are consistent with the manufacturer's specifications. Field instruments for this purpose will have unique identifiers and calibration readings for each instrument will be logged on calibration forms before use in the field. The FM or designee will be responsible for performing and documenting calibration and performance verification records for all instruments in the field. Calibration forms are provided in **Appendix B**.

Soil Boring Advancement/Abandonment and Soil Sample Collection

To confirm the presence and nature, or absence of COPCs at the former UST sites, and to characterize subsurface conditions, soils borings will be advanced to the water table (approximately 10 ft bgs) or to

maximum depth of 20 ft bgs using direct push technology (DPT). Each of the borings will be lithologically logged and soil samples collected for chemical analysis. Soil boring locations will be selected based on the findings of the scoping visit and will be biased toward potential source areas, spill locations, and along potential migration pathways.

Soil cores will be collected continuously, visually screened for evidence of contamination (e.g., discoloring or staining), and will be logged, classified, and geologically interpreted by a geologist to include the amount of sample recovery, consistency or density, matrix color (using a Munsell soil color chart), field screened using a PID, classification using the Unified Soil Classification System (USCS) per ASTM D2488, field moisture, plasticity, cohesiveness, primary sedimentary structure, observed secondary features, weathering zone abbreviation (Hallberg, 1978), and depositional interpretation.

At each soil boring location, up to two discrete soil samples may be collected to evaluate COPC concentration distribution. Soil samples will be biased toward intervals exhibiting impacts (e.g., soil staining, elevated PID measurement). If field observations do not indicate the presence of impacts, then samples will be located at depth intervals related to the depths of the former USTs and the 1-ft interval just above the saturated zone. Soil samples will be shipped to the contracted off-site laboratory for analysis of VOCs using USEPA Method 8260B, PAHs using USEPA Method 8270 with Select Ion Monitoring, and/or lead using USEPA Method 6020B. A summary of soil samples to be collected during this TO is outlined in **Table 1**.

Table 1. Soil Sampling Summary

Site ID	No. Borings	Depth (ft bgs)	No. Soil Samples	Analysis
CTU006 (UST212)	7	20	14	VOCs, PAHs
CTU007 (UST 215)	7	20	14	VOCs, PAHs
CTU008 (UST219)	7	20	14	VOCs, PAHs
CTU011, CTU012, and CTU013 (UST 8002)	12	20	24	VOCs, PAHs, Lead

Notes:
ft bgs – feet below ground surface
PAHs – polynuclear aromatic hydrocarbons
VOCs – volatile organic compounds

Soil samples will be collected in accordance with SOP 3, *Soil Sampling* (**Appendix A**). Borings that are not completed as monitoring wells will be abandoned in accordance with the state-specific regulatory requirements and SOP 11, *Borehole Abandonment* (**Appendix A**). Soil samples and lithologic descriptions will be recorded on WDNR's Soil Boring Log (Form 4400-122) (**Appendix B**). Soil sample information will be recorded on Soil Sample Collection Forms (**Appendix B**). A summary of proposed soil samples (e.g., number, locations, equipment to be used, and depths) are provided on **Worksheets #17-1 and #17-2**.

Monitoring Well Installation

At select soil boring locations, Wisconsin Administrative Code (WAC) NR141-compliant groundwater monitoring wells will be installed using hollow-stem drilling techniques. Monitoring wells will be installed in accordance with WAC, Chapter NR 141. Monitoring wells will be constructed as water table wells such that the well screens will intersect the water table, assumed to be at a depth 10 ft bgs.

Monitoring wells will be constructed using 2-inch inside diameter schedule 40 polyvinyl chloride casing that will be flush threaded and will have a threaded end cap with O-ring seal installed. Well screens will be 10-ft long, 2-inch inside diameter factory-cut 0.01-inch slotted pipe. Well screen and riser will be placed in a manner to avoid threaded sections of riser being present at the top of casing elevation. A filter pack consisting of 10/20 mesh, commercially available, clean silica sand with uniform sorting, or similar size compatible with the well slot size, will be installed in the annulus around the well screen at a minimum of 0.5 ft below the well cap to a minimum height of 2 ft above the top of screen. An annular seal will be placed above the filter pack and will consist of a minimum of 2 ft of bentonite. Following placement, the annular seal will be allowed to hydrate for a minimum of 12 hours prior to grout installation. After hydration of the annular seal, a high solids-bentonite grout will be used to fill the boring annulus from the top of the bentonite seal to within approximately 1 to 2 ft of ground surface. The grout will be allowed to cure for a minimum of 24 hours prior to installation of the concrete pad and vaults.

The wells will be completed as flush-mount wells with well pad construction consisting of a minimum 2-ft by 2-ft by 4-inches thick concrete pad with 8-inch minimum diameter bolt-down manhole. The concrete shall be sloped away from the protective casing to promote surface drainage away from the well. The top of well casing will be cut using a rotary tool to ensure the top of the riser is smooth, even, and parallel to ground surface. The top of riser will be capped with watertight, lockable compression well caps with keyed alike Master locks or equivalent. Boring logs and monitoring well completion diagrams will be generated for each well. **Table 2** summarizes proposed monitoring wells.

Table 2. Monitoring Well Construction Summary

Site ID	No. Monitoring Wells	Proposed Depth (ft bgs)	Proposed Screen Interval (ft bgs)
CTU006 (UST212)	3	20	9-19
CTU007 (UST 215)	3	20	9-19
CTU008 (UST219)	3	20	9-19
CTU011, CTU012, and CTU013 (UST 8002)	5	20	9-19

Notes:
ft – foot or feet
bgs – below ground surface

Well Development

A minimum of 24 hours following monitoring well installation and surface completion activities, each monitoring well will be developed to remove sediment from the well. Development will include surging the well with an appropriately sized surge block followed by pumping with a submersible pump capable of removing sediment from the bottom of the well. Wells will be developed until water quality parameters (temperature, specific conductance, pH, dissolved oxygen (DO), and oxidation-reduction potential [ORP]) have stabilized and turbidity has stabilized or is below 10 nephelometric turbidity units.

Static Water Level Measurements

Prior to initiating groundwater sampling activities, static water levels will be measured in existing and newly installed monitoring wells at each site to evaluate the direction of groundwater flow. Water level measurements will be measured from the top of well casing to an accuracy of 0.01 ft using an electronic water level indicator.

Groundwater Sampling

Groundwater samples will be collected from newly installed monitoring wells using an appropriate pump type based on depth to water and depth of the well. Wells will be purged and sampled using low flow purge and sampling techniques. Low flow purging and sampling rates generally range from 0.1 to 1.0 liters per hour. Drawdown during purging will not exceed 0.33 ft (USEPA, 1996). During purging, water quality parameters (pH, specific conductance, temperature, DO, ORP, and turbidity) will be measured and recorded at regular intervals that will be sufficient in duration so that at least one volume of the flow-through cell (flow cell) has been purged. Water quality parameter measurements will be obtained during purging of the monitoring wells using a continuous flow cell water quality monitoring unit equipped to measure multiple parameters. The flow cell will be attached to the discharge of the pumped well with a polyethylene hose. Parameter probes are secured within the unit that is inserted into the continuous flow-through cell to monitor real-time parameter variation. Turbidity will be measured using a turbidity meter. Water quality meters and the turbidity meter shall be calibrated in accordance with the manufacturer's procedures.

Purging will be considered complete when stabilization of water quality parameters is achieved. Stabilization of field parameters is achieved when three consecutive readings are within the following limits:

- Turbidity – plus or minus 1%;
- Conductivity – plus or minus 3%;
- pH – plus or minus 0.1 unit;
- Temperature – plus or minus 0.5 degree Celsius;
- ORP – plus or minus 10%; and,
- DO – plus or minus 10%.

For slowly recharging wells, the parameters may not stabilize before the well casing is emptied, even when using low flow rates. In this case, purging will be considered complete when one well volume (well casing plus filter pack volume) has been purged from the well and the well goes dry. The well will be allowed to recharge, and sampling must be initiated within 24 hours of purging. The depth to the water level in the well will be measured and recorded immediately prior to sample collection.

Prior to sample collection, the flow-through cell will be disconnected, and the sample bottle will be filled directly from the pump tubing. The samples, field duplicates, and matrix spike/matrix spike duplicate samples will be filled simultaneously. **Table 3** summarizes proposed groundwater samples.

Table 3. Groundwater Sampling Summary

Site ID	No. Groundwater Samples	Analysis
CTU006 (UST212)	3	VOCs, PAHs
CTU007 (UST 215)	3	VOCs, PAHs
CTU008 (UST219)	3	VOCs, PAHs
CTU011, CTU012, and CTU013 (UST 8002)	5	VOCs, PAHs

Vapor Intrusion Sampling

Soil vapor sampling will be conducted via a soil vapor probe within a temporary vapor well. The vapor probe will be installed using DPT to advance a soil boring to a depth of 10 ft bgs. Once an open soil boring has been advanced, a soil vapor probe will be constructed within the boring. The vapor filter will be connected to tubing and placed approximately 3-inches from the bottom of the boring. The bottom of the borehole will then be filled with 6 inches of 20/40 mesh silica sand. The annulus surrounding the prepacked screen interval and 2 ft above the top of the screen will be also filled with 20/40 mesh silica sand. Above the filter pack, the annulus will be sealed with a minimum of two feet of 8/20 mesh granular sodium bentonite consisting of 1 ft of dry granular bentonite to prevent the infiltration of hydrated bentonite followed by 1 ft of hydrated bentonite. Hydrated granular bentonite will be placed to a depth of 2 ft bgs. The annulus from 0 to 2 ft bgs will be filled with Rapid Set® concrete to complete the surface seal. **Table 4** summarizes proposed soil vapor sampling. **Figures 03 and 05** indicate proposed soil vapor probe locations.

Exhibit 1: Typical soil vapor probe construction.

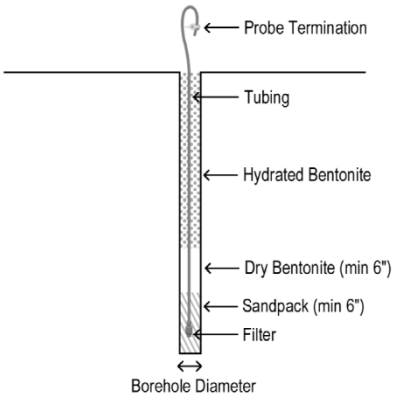

Table 4. Soil Vapor Sampling Summary

Site ID	No. Samples	Analysis
CTU006 (UST212)	3	VOCs via TO15
CTU008 (UST219)	3	VOCs via TO15

Surveying

Sample locations (i.e., soil, groundwater, and soil vapor samples) will have horizontal coordinates determined using a Trimble ProXH Global Positioning System unit, or equivalent device, which is capable of providing horizontal accuracies of 1 meter or less. The coordinates for each sample location will be recorded electronically by trained field personnel and written in the logbook. Locations will be recorded in Universe Transverse Mercator World Geodetic System 84 coordinates.

Wells will be surveyed by a licensed surveyor to enable accurate placement of well locations on a map and to provide data sufficient to calculate groundwater elevations. Horizontal coordinates will be surveyed to the nearest 0.1 ft and referenced to the relevant State Plane Coordinate System using the North American Datum of 1983, as adjusted in 1991. Elevation measurements will be made both at ground surface and at a casing measurement point at each of the wells. Elevations will be surveyed to the nearest 0.01 ft and referenced to the

North American Vertical Datum of 1988. The survey reference point on the monitoring well casing will be marked for future reference by the licensed surveyor.

Investigation-Derived Waste Management

Investigation-derived waste (IDW) will be managed in accordance with the USEPA guidance document entitled *Management of Investigation-Derived Wastes During Site Investigations* (USEPA, 1992).

IDW will consist of soil cuttings from monitoring well installation, soil boring advancement, decontamination water, purge water, disposable personal protective equipment (PPE), and other trash. PPE and other trash will be placed in plastic bags and disposed into sanitary trash containers bound for final disposition at a sanitary landfill. Soil and water IDW will be sampled and containerized in Department of Transportation (DOT)-approved containers pending characterization based on the requirements of the disposal facility. Disposition of these wastes will be determined based upon analytical results and regulations governing these wastes.

Field Quality Control

Equipment blanks will be collected daily when non-dedicated sampling equipment is used, by running water over/through the equipment after decontamination. Preferably, equipment blanks will be collected after sampling the location suspected to be most contaminated. The sampler shall record the locations where samples are collected with non-dedicated equipment on the field worksheet or logbook.

Typically, field blanks will be collected at a frequency of 5% (i.e., one (1) field blank per twenty (20) primary samples).

Field duplicates will be collected at a frequency of 10% (i.e., one (1) field duplicate per ten (10) primary field samples). Field duplicates will be collected from locations indicated on Worksheet #18. If the indicated location cannot be sampled or volume is insufficient for collection of field QC, another location within the site may be selected, as long as the required frequency is achieved. Field duplicate identifications (IDs) will be blinded by replacing the location code with “FD,” per the examples in Worksheet #18.

Additional sample volume for matrix spikes (MSs) and matrix spike duplicates (MSDs) are typically collected at a frequency of 5% (i.e., one (1) MS/MSD pair per twenty (20) primary field samples). MS/MSD sample aliquots may be labelled with the same ID as the parent sample and should be indicated on the laboratory chain of custody by writing “MS/MSD” on parent sample line.

Trip blanks will be supplied by the lab, and one (1) trip blank shall be included in every cooler containing VOC sample aliquots.

Decontamination

This discussion presents procedures for decontamination of personnel and equipment. Decontamination of equipment and personnel will be performed for health and safety precautions to avoid cross contamination of samples collected for chemical analysis, and to limit the migration of contaminants off-site and between on-site work areas.

Equipment Decontamination

Decontamination of equipment will occur within the exclusion zone of the intrusive activities or at central decontamination stations (if required). SOP 15, *Equipment Decontamination* (**Appendix A**) discusses the operating procedures for the decontamination of equipment. Decontamination pads constructed at each site shall be durable, portable, and capable of supporting all equipment to be decontaminated without risk of damage resulting in loss of rinsate. Decontamination pads will also be capable of containing decontamination fluids and transfer into appropriate DOT approved containers for off-site shipment and proper disposal.

Reusable equipment that may come in contact with samples for chemical analysis will be decontaminated between collection of samples. Cleaning will consist of scraping and scrubbing to remove encrusted materials, if necessary, followed by soap (Alconox or similar) and water wash and then a potable water rinse. Alternatively, the equipment may be cleaned with a high-pressure hot water/steam-cleaning unit. Following decontamination, clean equipment will be allowed to air dry prior to obtaining the next sample.

Personnel Decontamination

Decontamination of personnel engaged in the intrusive activities will be performed at personnel decontamination stations established at the edge of the exclusion zones. A personnel decontamination station will also be available at the central decontamination station (if required) for decontamination of field personnel.

Personnel decontamination will take place in both the central decontamination station (if required) and at the edge of exclusion zones prior to leaving these areas. Personnel decontamination will consist primarily of removal of gloves and disposable coveralls (if worn on-site) followed by cleaning of boots and hand washing with soap and water.

Non-reusable equipment and clothing will be collected in plastic trash bags. Disposal of IDW associated with decontamination activities will be in accordance with the *Investigation-Derived Waste Management* Section located in this worksheet (Worksheet #17).

Field Documentation

Field personnel will document field activities, issues, variances, progress, and any other pertinent information. This documentation will be kept on-site during fieldwork. The following sections list the forms/logs which will be used to document field activities.

Field Logbook

Field logbooks will be maintained to record pertinent information. The cover of each field logbook will contain the following information:

- Project name and number;
- Book number;
- Activity type;
- Start date; and,
- Stop date.

Entries to a field logbook will be made and completed daily. At a minimum, the information provided will consist of the following:

- Date;
- Start time;
- Weather;
- Field personnel present;
- Visitors to the site (time, name, and company);
- Level of personnel protection used;
- Type of activity conducted;
- Sampling location;
- Sample identification number;
- Description of sampling point;
- Method of sampling;
- Type of sample;
- Air monitoring readings, if applicable;
- Pertinent field observations;
- Instrument identification numbers;
- Results of field instrument calibration;
- Field measurements;
- Anticipated disposition of sample;
- Description of all related activities; and,
- Signature of the person making the entry.

Measurements made and samples collected will be recorded in indelible ink. No erasures are permitted. If an incorrect entry is made, the data shall be crossed out with a single strike mark and initialed. Entries will be organized into easily understandable tables, if possible.

At each station where a sample is collected or a measurement made, a detailed description of the location of the stations will be recorded. Equipment used to make measurements will be identified, including the date and time on which the equipment was calibrated. In addition, the FM will maintain a daily field summary book. Entries into this book will include:

- Types of activities conducted throughout the day;
- Personnel involved with each activity;
- Description of instructions given to field personnel;
- Health and safety related problems and corrective measures taken;
- Summary of discussions with the Project Manager (PM);
- List of site visitors with purpose for the visit;
- Changes/modifications to sample locations or procedures; and,
- Any other pertinent information related to site activities.

Photographic Records

At a minimum, color photographs will be taken, during important milestones of field activities. Additional photographs may be taken during the activity and at the request of the FM. Photographs will be accompanied with a numbered photograph log that will include the project name, date, and description of activity (e.g., surface soil sampling and corresponding sample identification number).

Sample Documentation

Sample collection logs will be completed while environmental samples are collected for laboratory analysis, as certain field conditions, environments, and other notes may assist in the interpretation of analytical results. Sample collection logs are presented in **Appendix B**.

A sample identification system has been established for sampling activities. Sample identification provides a method for tracking each sample through collection, analysis, and data reduction. Sample labels shall be pre-printed if possible. A Station ID will correspond with a unique location within each site. Sample IDs will be numbered sequentially and represent a unique sample and identify the type of sample (e.g., soil, groundwater, etc.). Nomenclature to be used is presented on **Worksheet #18**.

Sample Packaging and Shipping Requirements

The instructions contained in this section are to be used by field personnel when collecting and handling samples for packing and shipping. On the occasion that field personnel determine that any of the instructions described in this section are inappropriate, inadequate, or impractical and that another procedure must be used, the variance must be documented in the field logbook, along with a description of the circumstances requiring its use.

Once the samples have been collected, it is important that the sampler properly package the samples for shipment/transport and ensure that the samples are sent to the appropriate laboratory as quickly as possible. Sample preservation requirements are specified in **Worksheet #19 & 30**. Prompt and proper packaging of samples will:

- Protect the integrity of samples from changes in composition or concentration caused by bacterial growth or degradation from increased temperatures;
- Reduce the chance of leaking or breaking of sample containers that would result in loss of sample volume, loss of sample integrity, and exposure of personnel to toxic substances; and,
- Verify compliance with shipping regulations.

Prior to shipment, samplers will conduct an inventory of the contents of the shipping cooler or container against the corresponding chain of custody (COC) record when packing for shipment to the laboratory. An inventory will ensure that the proper number of containers have been collected for each analysis of the samples, that the required QA/QC samples and cooler temperature blanks are included, and the correct sample numbers have been assigned to each sample.

After samples are packaged within shipping coolers, the following procedures must be followed:

- The top and bottom of the coolers must be secured with tape;
- Place return address labels clearly on the outside of the cooler;
- The words "Contents – Water Samples" should be clearly marked on the outside of the cooler; and,
- Attach the required COC seals.

Paperwork/COC records will be placed in a plastic bag or pouch and then secured to the underside of the shipping cooler lids (see example on right). Custody seals will be placed on all shipping containers to ensure that tampering or unauthorized opening does not compromise sample integrity. Further information regarding sample handling procedures can be found in SOP 17, *Sample Handling and Custody* (**Appendix A**).

Collected samples will be shipped to the analytical laboratory within 24 hours of collection. Samples will be shipped in accordance with applicable DOT requirements (49 Code of Federal Regulations [CFR] 171 through 49 CFR 178) and USEPA sample-handling, packaging, and shipping methods (40 CFR 262).

Chain of Custody Records

The sampler will be responsible for initiating and completing the COC. The field team members are responsible for the care and custody of the samples collected until the samples are transferred to another individual or shipped to the analytical laboratory. The COC will be signed, with date and time, by the sampler when samples are relinquished to anyone else. The COC will accompany the samples at all times. Individuals who subsequently take possession of the samples will also sign, with date and time, the COC. The only exception to this requirement is that of common carriers (e.g., Federal Express). The shipping document provided by them will suffice for custody. The COC accompany each cooler containing samples sent to the analytical laboratory. Laboratory personnel are responsible for the receipt and entry of samples into the laboratory, which have been submitted under a COC document. Additionally, samples received will be entered into the laboratory COC procedures by properly documenting and maintaining COC from the moment that they take custody of the sample until the sample is properly disposed of. The COC procedures are summarized as follows:

- At the time of sample collection, the COC is completed for the sample collected.
- When the form is full or when all samples have been collected that will fit in a single cooler, the field team members will crosscheck the form for possible errors. Corrections are made to the record with a single strike mark and dated and initialed. All entries will be made in blue or black ink. The COC will be signed when the samples are relinquished.
- If shipping samples to a laboratory off-site, a shipping bill is completed, and the shipping bill number recorded on the COC prior to enclosing inside a clear plastic bag and attaching it to the inside of the cooler lid.

When transferring custody of the samples, the individual relinquishing custody of the samples will verify sample numbers and condition and will document the sample acquisition and transfer by signing, with date and time, the COC. The field sample coordinator will assist the samplers in grouping the samples for shipment to the analytical laboratory and review the completed COC for each cooler. Samples will be packaged for shipment and submitted to the analytical laboratory with a separate COC accompanying each cooler.

Custody seals will be used to ensure that the shipping containers have not been opened during shipment and prior to receipt at the off-site laboratory. The following information will be included on the custody seals:

- Signature of the sample coordinator; and,
- Date when the sample package is sealed.

Seals will be completed using indelible ink. The seals will be affixed to the front and back of the cooler, at the interface of the cooler and the lid. The placement of the seals will be in a manner that breaking the seals would be necessary to open the sample shipping cooler.

In conjunction with data reporting, the analytical laboratory will return the original or a photocopy of the original COC to the field office for inclusion into the project file.

Samples collected will remain in the possession of the sampling crew until shipment. Locked vehicles or trailers will be used for interim storage if necessary. If coolers (used for sample storage) must be left unattended for extended periods of time, signed custody seals will be placed on the front and back of each cooler or the cooler will be stored under lock until shipped to the off-site laboratory.

Sample Receipt Forms

When the analytical laboratory receives the sample coolers, a cooler and/or sample receipt form for the samples will be initialed and faxed back to the job site or submitted to the CUES PM. This form will document the sample condition upon receipt. Receipt nonconformance situations will be initiated using this form.

Data Management

The purpose of data management is to verify that the data collected are accurate and readily accessible to meet the analytical and reporting objectives of the project. The analytical results will be provided by the laboratory in the Environmental Resources Program Information Management System (ERPIMS) lab submittal electronic data deliverable (EDD) format. Laboratory EDDs will be loaded to the ERPToolsX software where it will be checked and supplemented with field-derived data. Final ERPIMS submittal will be complete after all data in ERPToolsX have been loaded, checked, and final review performed by CUES ERPIMS staff.

Data Validation

Data validation will be conducted in accordance with **QAPP Worksheet #36**: *Data Validation Procedures*. The results of data validation will be presented in a Data Validation Report that will be included as an appendix in the groundwater monitoring reports.

Reporting

Reporting of SI results will occur within an SI Report.

QAPP WORKSHEET #15A: PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Analytical Group: Metals, lead
Matrix: Groundwater
Analytical Method: USEPA SW-846 6020B
Concentration: Low
Laboratory: Eurofins Environment Testing America in Denver, Colorado

Analyte	CAS Number	WDNR Enforcement Standard[1] (µg/L)	WDNR Preventative Action Limit[2] (µg/L)	Project Quantitation Limit Goal[3] (µg/L)	Laboratory-Specific		
					Limit of Quantitation[6] (µg/L)	Limit of Detection (µg/L)	Detection Limit (µg/L)
Lead	7439-92-1	15	1.5	LOQ*	3.00	0.700	0.180

Matrix: Soil
Analytical Method: USEPA SW-846 6020B
Concentration: Low
Laboratory: Eurofins Environment Testing America in Denver, Colorado

Analyte	CAS Number	WDNR Soil-Groundwater RCL[4] (µg/kg)	WDNR Direct Contact RCL[5] (µg/kg)	Project Quantitation Limit Goal[3] (µg/kg)	Laboratory-Specific		
					Limit of Quantitation[6] (µg/kg)	Limit of Detection (µg/kg)	Detection Limit (µg/kg)
Lead	7439-92-1	27,000	52,000	13,500	400	70.0	18.2

Notes:
[1] Wisconsin Department of Natural Resources Chapter NR 140 Table 1, Public Health Groundwater Quality Standards Enforcement Standard
[2] Wisconsin Department of Natural Resources Chapter NR140 Table 1, Public Health Groundwater Quality Standards Preventative Action Limit
[3] Whenever possible, the Project Quantitation Limit Goal is equal to or less than one half of the lowest regulatory limit. When analytical sensitivity is not sufficient, the goal is the laboratory's statistically derived limit of quantitation in undiluted samples.
[4] Wisconsin Department of Natural Resources Chapter NR 720, Soil Residual Contaminant Level Worksheet, Soil-to-Groundwater Pathway, December 2018
[5] Wisconsin Department of Natural Resources Chapter NR 720, Soil Residual Contaminant Level Worksheet, Direct Contact (Industrial and Non-Industrial) Pathway, December 2018
*The laboratory's limit of quantitation for this analyte is between the WDNR Enforcement Standard and the Preventative Action Limit. The Project Quantitation Limit Goal is the laboratory's statistically derived limit of quantitation.
[6] Where the Limit Of Quantification (LOQ) is greater than the WDNR Preventive Action Limit, the LOQ will be the Project Screening Level.
µg/kg – micrograms per kilogram
µg/L – micrograms per liter
CAS – chemical abstract service
SW-846 6020B – Inductively Coupled Plasma – Mass Spectrometry
USEPA – United States Environmental Protection Agency
WDNR – Wisconsin Department of Natural Resources

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QAPP WORKSHEET #15B: PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Matrix: Groundwater
Analytical Group: Volatile Organic Compounds
Analytical Method: USEPA SW-846 8260D
Concentration: Low
Laboratory: Eurofins Environment Testing America in Denver, Colorado

Analyte	CAS Number	WDNR Enforcement Standard[1] (µg/L)	WDNR Preventative Action Limit[2] (µg/L)	Project Quantitation Limit Goal[3] (µg/L)	Laboratory-Specific		
					Limit of Quantitation[7] (µg/L)	Limit of Detection (µg/L)	Detection Limit (µg/L)
1,1,1,2-Tetrachloroethane	630-20-6	70	7	3.0	1.0	0.8	0.21
1,1,1-Trichloroethane	71-55-6	200	40	20	1.0	0.4	0.16
1,1,2,2-Tetrachloroethane	79-34-5	0.2	0.02	LOQ†	1.0	0.8	0.21
1,1,2-Trichloroethane	79-00-5	5	0.5	LOQ*	1.0	0.8	0.27
1,1-Dichloroethane	75-34-3	850	85	40	1.0	0.8	0.22
1,1-Dichloroethene	75-35-4	7	0.7	LOQ*	1.0	0.8	0.23
1,2,3-Trichloropropane	96-18-4	60	12	6.0	3.0	0.8	0.33
1,2,4-Trichlorobenzene	120-82-1	70	17	7.0	1.0	0.8	0.21
1,2,4-Trimethylbenzene[4]	95-63-6	480	96	48	1.0	0.4	0.15
1,2-Dibromo-3-Chloropropane	96-12-8	0.2	0.02	LOQ†	5.0	1.6	0.47
1,2-Dibromoethane (EDB)	106-93-4	0.05	0.005	LOQ†	1.0	0.4	0.18
1,2-Dichlorobenzene	95-50-1	600	60	30	1.0	0.4	0.15
1,2-Dichloroethane	107-06-2	5	0.5	LOQ*	1.0	0.4	0.13
1,2-Dichloropropane	78-87-5	5	0.5	LOQ*	1.0	0.4	0.18
1,3,5-Trimethylbenzene[4]	108-67-8	480	96	48	1.0	0.4	0.16
1,3-Dichlorobenzene	541-73-1	600	120	60	1.0	0.4	0.13
1,4-Dichlorobenzene	106-46-7	75	15	7.0	1.0	0.4	0.16
2-Butanone (MEK)	78-93-3	4,000	800	400	6.0	4	2
4-Methyl-2-pentanone (MIBK)	108-10-1	500	50	25	5.0	3.2	0.98
Acetone	67-64-1	9,000	1,800	900	10	6.4	1.9
Benzene	71-43-2	5	0.5	LOQ*	1.0	0.4	0.16
Bromodichloromethane	75-27-4	0.6	0.06	LOQ†	1.0	0.4	0.17
Bromoform	75-25-2	4.4	0.44	LOQ*	1.0	1.0	0.458
Bromomethane	74-83-9	10	1	LOQ*	2.0	0.8	0.21
Carbon disulfide	75-15-0	1,000	200	100	2.0	0.8	0.167
Carbon tetrachloride	56-23-5	5	0.5	LOQ*	2.0	0.4	0.19
Chlorobenzene	108-90-7	100	20	10	1.0	0.4	0.17
Chlorodibromomethane	124-48-1	60	6	3.0	1.0	0.4	0.17
Chloroethane	75-00-3	400	80	40	2.0	1.6	0.41
Chloroform	67-66-3	6	0.6	LOQ*	1.0	0.4	0.16
Chloromethane	74-87-3	30	3	2.0	2.0	0.8	0.30
cis-1,2-Dichloroethene	156-59-2	70	7	3.0	1.0	0.4	0.15
cis-1,3-Dichloropropene[5]	10061-01-5	0.4	0.04	LOQ†	1.0	0.4	0.16
Dichlorodifluoromethane	75-71-8	1,000	200	100	2.0	0.8	0.31
Ethyl ether	60-29-7	1,000	100	50	2.0	0.8	0.26
Ethylbenzene	100-41-4	700	140	70	1.0	0.4	0.16

Analyte	CAS Number	WDNR Enforcement Standard[1] (µg/L)	WDNR Preventative Action Limit[2] (µg/L)	Project Quantitation Limit Goal[3] (µg/L)	Laboratory-Specific		
					Limit of Quantitation[7] (µg/L)	Limit of Detection (µg/L)	Detection Limit (µg/L)
Hexane	110-54-3	600	120	60	2.0	0.8	0.163
Methyl tert-butyl ether	1634-04-4	60	12	6.0	5.0	0.8	0.25
Methylene Chloride	75-09-2	5	0.5	LOQ*	5.0	2.0	0.938
m-Xylene & p-Xylene[6]	179601-23-1	2	0.4	LOQ*	2.0	0.8	0.153
o-Xylene[6]	95-47-6	2	0.4	LOQ†	1.0	0.4	0.19
Styrene	100-42-5	100	10	5.0	1.0	0.8	0.356
t-Butyl alcohol	75-65-0	12	1.2	LOQ†	50	12.8	3.60
Tetrachloroethene	127-18-4	5	0.5	LOQ*	1.0	0.4	0.20
Tetrahydrofuran	109-99-9	50	10	7.0	7.0	6.4	2.03
Toluene	108-88-3	800	160	80	1.0	0.4	0.17
trans-1,2-Dichloroethene	156-60-5	100	20	10	1.0	0.4	0.15
trans-1,3-Dichloropropene[5]	10061-02-6	0.4	0.04	LOQ†	1.0	0.4	0.19
Trichloroethene	79-01-6	5	0.5	LOQ*	1.0	0.4	0.16
Trichlorofluoromethane	75-69-4	3,490	698	349	2.0	0.8	0.29
Vinyl chloride	75-01-4	0.2	0.02	LOQ†	1.5	0.2	0.10

Notes:
[1] WDNR Chapter NR 140 Table 1, Public Health Groundwater Quality Standards Enforcement Standard
[2] WDNR Chapter NR 140 Table 1, Public Health Groundwater Quality Standards Preventative Action Limit
[3] Whenever possible, the Project Quantitation Limit Goal is equal to or less than one half of the WDNR Preventative Action Limit. When analytical sensitivity is not sufficient, the goal is the laboratory's statistically derived limit of quantitation in undiluted samples.
[4] The WDNR Enforcement Standard and Preventative Action Limit are for the combined total of 1,2,4- and 1,3,5-trimethylbenzene.
[5] The WDNR Enforcement Standard and Preventative Action Limit are for the combined total of cis- and trans-1,3-dichloropropene.
[6] The WDNR Enforcement Standard and Preventative Action Limit are for the combined total of meta-, ortho-, and para-xylene
[7] Where the Limit Of Quantification (LOQ) is greater than the WDNR Preventive Action Limit, the LOQ will be the Project Screening Level.
*The laboratory's limit of quantitation for this analyte is between the WDNR Enforcement Standard and the Preventative Action Limit. The Project Quantitation Limit Goal is the laboratory's statistically derived limit of quantitation.
†The laboratory's limit of quantitation for this analyte exceeds both the WDNR Enforcement Standard and the Preventative Action Limit. The Project Quantitation Limit Goal is the laboratory's statistically derived limit of quantitation.
µg/L – micrograms per liter
CAS – chemical abstract service
SW-846 8260D – Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry
USEPA – United States Environmental Protection Agency
WDNR – Wisconsin Department of Natural Resources

QAPP WORKSHEET #15C: PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Matrix: Groundwater
Analytical Group: Polycyclic Aromatic Hydrocarbons
Analytical Method: USEPA SW-846 8270 SIM
Laboratory: Eurofins Environment Testing America in Denver, Colorado

Analyte	CAS Number	WDNR Enforcement Standard[1] (µg/L)	WDNR Preventative Action Limit[2] (µg/L)	Project Quantitation Limit Goal[3] (µg/L)	Laboratory-Specific		
					Limit of Quantitation[4] (µg/L)	Limit of Detection (µg/L)	Detection Limit (µg/L)
1-Methylnaphthalene	90-12-0	NL	NL	LOQ	0.100	0.0120	0.00566
2-Methylnaphthalene	91-57-6	NL	NL	LOQ	0.100	0.0120	0.00515
Acenaphthene	83-32-9	NL	NL	LOQ	0.100	0.0300	0.0108
Acenaphthylene	208-96-8	NL	NL	LOQ	0.100	0.0120	0.00996
Anthracene	120-12-7	3,000	600	300	0.100	0.0400	0.0142
Benzo(a)anthracene	56-55-3	NL	NL	LOQ	0.100	0.0400	0.0127
Benzo(a)pyrene	50-32-8	0.2	0.02	LOQ*	0.100	0.0120	0.00514
Benzo(b)fluoranthene	205-99-2	0.2	0.02	LOQ*	0.100	0.0400	0.0143
Benzo(g,h,i)perylene	191-24-2	NL	NL	LOQ	0.100	0.0200	0.00814
Benzo(k)fluoranthene	207-08-9	NL	NL	LOQ	0.100	0.0400	0.0110
Chrysene	218-01-9	0.2	0.02	LOQ*	0.100	0.0400	0.0122
Dibenz(a,h)anthracene	53-70-3	NL	NL	LOQ	0.100	0.0120	0.00482
Fluoranthene	206-44-0	400	80	40	0.100	0.0400	0.0347
Fluorene	86-73-7	400	80	40	0.100	0.0400	0.0188
Indeno(1,2,3-cd)pyrene	193-39-5	NL	NL	LOQ	0.100	0.0200	0.0147
Naphthalene	91-20-3	100	10	5	0.100	0.0120	0.00533
Phenanthrene	85-01-8	NL	NL	LOQ	0.100	0.0200	0.00975
Pyrene	129-00-0	250	50	25	0.100	0.0200	0.00808

Notes:
[1] WDNR Chapter NR 140 Table 1, Public Health Groundwater Quality Standards Enforcement Standard
[2] WDNR Chapter NR140 Table 1, Public Health Groundwater Quality Standards Preventative Action Limit
[3] Whenever possible, the Project Quantitation Limit Goal is equal to or less than one half of the lowest applicable regulatory limit. When analytical sensitivity is not sufficient, the goal is the laboratory's statistically derived limit of quantitation in undiluted samples. If a target does not have WDNR limits, the Project Quantitation Limit Goal is the limit of quantitation achieved in the sample. When a target analyte does not have a regulatory limit, the Project Quantitation Limit Goal is the LOQ achieved in the sample.
[4] Where the Limit Of Quantification (LOQ) is greater than the WDNR Preventive Action Limit, the LOQ will be the Project Screening Level.
*The laboratory's limit of quantitation for this analyte is between the WDNR Enforcement Standard and the Preventative Action Limit. The Project Quantitation Limit Goal is the laboratory's statistically derived limit of quantitation.
µg/L – micrograms per liter
CAS – chemical abstract service
LOQ – limit of quantitation
NL – not listed
SW-846 8270 SIM – Semi-volatile Organic Compounds by Gas Chromatography/Mass Spectrometry with Select Ion Monitoring
USEPA – United States Environmental Protection Agency

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QAPP WORKSHEET #15D: PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Matrix: Soil
Analytical Group: Volatile Organic Compounds
Analytical Method: USEPA SW-846 8260D
Laboratory: Eurofins Environment Testing America in Denver, Colorado

Analyte	CAS Number	WDNR Soil-Groundwater RCL[1] (µg/kg)	WDNR Direct Contact Non-Industrial[2] (µg/kg)	Project Quantitation Limit Goal[3] (µg/kg)	Laboratory-Specific		
					Limit of Quantitation[5] (µg/kg)	Limit of Detection (µg/kg)	Detection Limit (µg/kg)
1,1,1,2-Tetrachloroethane	630-20-6	53.4	2,780,000	25	5.0	5.0	2.22
1,1,1-Trichloroethane	71-55-6	140.2	640,000	70	5.0	5.0	1.98
1,1,2,2-Tetrachloroethane	79-34-5	0.2	810	LOQ*	5.0	0.8	0.285
1,1,2-Trichloroethane	79-00-5	3.2	1,590	LOQ*	5.0	3.2	0.88
1,1-Dichloroethane	75-34-3	483	5,060	240	5.0	0.8	0.21
1,1-Dichloroethene	75-35-4	5	120,000	5.0	5.0	1.6	0.59
1,2,3-Trichloropropane	96-18-4	51.9	5	5.0	5.0	0.8	0.218
1,2,4-Trichlorobenzene	120-82-1	408	24,000	200	5.0	1.6	0.73
1,2,4-Trimethylbenzene[4]	95-63-6	1,378	NL	650	5.0	5.0	2.31
1,2-Dibromo-3-Chloropropane	96-12-8	0.2	8	LOQ†	10	10	3.66
1,2-Dibromoethane (EDB)	106-93-4	0.028	50	LOQ*	5.0	1.6	0.52
1,2-Dichlorobenzene	95-50-1	1,168	376,000	550	5.0	5.0	1.87
1,2-Dichloroethane	107-06-2	2.8	NL	LOQ†	5.0	1.6	0.70
1,2-Dichloropropane	78-87-5	3.3	3,400	LOQ*	5.0	1.6	0.55
1,3,5-Trimethylbenzene[4]	108-67-8	1,378	NL	650	5.0	5.0	2.42
1,3-Dichlorobenzene	541-73-1	1,152	297,000	550	5.0	1.6	0.48
1,4-Dichlorobenzene	106-46-7	144	3,740	70	5.0	0.8	0.245
1,4-Dioxane	123-91-1	1.2	5,720	LOQ*	500	128	56.1
2-Butanone (MEK)	78-93-3	1,666	28,400,000	800	20	12.8	3.89
4-Methyl-2-pentanone (MIBK)	108-10-1	225	2,450,000	100	20	12.8	4.36
Acetone	67-64-1	3,676	63,400,000	1,500	72	72	35.6
Benzene	71-43-2	5.1	1,600	5.0	5.0	0.4	0.151
Bromodichloromethane	75-27-4	0.3	418	LOQ*	5.0	5.0	2.13
Bromoform	75-25-2	2.3	25,400	LOQ*	5.1	5.1	2.55
Bromomethane	74-83-9	5.1	9,600	LOQ*	10	3.2	1.35
Carbon disulfide	75-15-0	591	738,000	250	5.0	5.0	1.66
Carbon tetrachloride	56-23-5	3.9	916	LOQ*	5.0	5.0	2.01
Chlorobenzene	108-90-7	NL	370,000	1,000	5.0	5.0	2.06
Chlorodibromomethane	124-48-1	NL	NL	LOQ	5.0	5.0	2.27
Chloroethane	75-00-3	226	NL	100	10	6.4	1.99
Chloroform	67-66-3	3.3	454	LOQ*	10	0.8	0.29
Chloromethane	74-87-3	15.5	159,000	10	10	1.6	0.77
cis-1,2-Dichloroethene	156-59-2	41.2	156,000	20	5.0	0.8	0.201
cis-1,3-Dichloropropene[5]	10061-01-5	0.3	1,210,000	LOQ*	5.0	0.4	0.100
Dichlorodifluoromethane	75-71-8	NL	126,000	1,000	10	6.4	2.74
Ethyl ether	60-29-7	447	10,100,000	200	10	6.4	1.87
Ethylbenzene	100-41-4	1,570	8,020	750	5.0	0.8	0.305
Hexane	110-54-3	8,465	141,000	1,000	5.0	0.4	0.136

Analyte	CAS Number	WDNR Soil-Groundwater RCL[1] (µg/kg)	WDNR Direct Contact Non-Industrial[2] (µg/kg)	Project Quantitation Limit Goal[3] (µg/kg)	Laboratory-Specific		
					Limit of Quantitation[5] (µg/kg)	Limit of Detection (µg/kg)	Detection Limit (µg/kg)
Methyl tert-butyl ether	1634-04-4	27	63,800	20	20	6.4	2.11
Methylene Chloride	75-09-2	2.6	61,800	LOQ*	5.0	3.2	1.60
m-Xylene & p-Xylene[6]	179601-23-1	3,960	260,000	1,500	3.2	3.2	1.04
o-Xylene[6]	95-47-6	3,960	260,000	1,500	5.0	0.8	0.266
Styrene	100-42-5	220	867,000	100	5.0	0.8	0.280
t-Butyl alcohol	75-65-0	4.9	NL	LOQ†	200	12.8	3.27
Tetrachloroethene	127-18-4	4.5	33,000	LOQ*	5.0	5.0	1.91
Tetrahydrofuran	109-99-9	22.2	23,300,000	20	20	6.4	3.19
Toluene	108-88-3	1,107	818,000	550	5.0	0.8	0.227
trans-1,2-Dichloroethene	156-60-5	62.6	1,560,000	30	5.0	0.8	0.39
trans-1,3-Dichloropropene[5]	10061-02-6	0.3	1,510,000	LOQ*	5.0	0.2	0.0830
Trichloroethene	79-01-6	3.6	1,300	LOQ*	5.0	4.0	1.91
Trichlorofluoromethane	75-69-4	NL	1,230,000	1,000	10	10	3.20
Vinyl chloride	75-01-4	0.1	67	LOQ*	5.0	3.2	1.34

Notes:
[1] WDNR Chapter NR 720, Soil Residual Contaminant Level Worksheet, Soil-to-Groundwater Pathway (Dilution Factor = 2), December 2018
[2] WDNR Chapter NR720, Soil Residual Contaminant Level Worksheet, Direct Contact Non-Industrial Land Usage, December 2018
[3] Whenever possible, the Project Quantitation Limit Goal is equal to or less than one half of the lowest applicable regulatory standard. When analytical sensitivity is not sufficient, the goal is the laboratory's statistically derived limit of quantitation for non-dilute samples. When a target analyte does not have a regulatory limit, the Project Quantitation Limit Goal is the actual limit of quantitation achieved in the sample.
[4] The WDNR Soil Residual Contaminant Level for Direct-Contact Non-Industrial Land Use are for the combined total of 1,2,4- and 1,3,5-trimethylbenzene.
[5] The WDNR Soil Residual Contaminant Level for Direct-Contact Non-Industrial Land Use are for the combined total of cis- and trans-1,3-dichloropropene.
[6] The WDNR Soil Residual Contaminant Levels for Soil-to-Groundwater Pathway and Direct-Contact Non-Industrial Land Use are for the combined total of meta-, ortho-, and para-xylene
[5] Where the Limit of Quantification (LOQ) is greater than the lowest RCL, the LOQ will be the Project Screening Limit
*The laboratory's limit of quantitation for this analyte is between the WDNR Soil-Groundwater RCL and the WDNR Direct Contact Non-Industrial Limit. The Project Quantitation Limit Goal is the laboratory's statistically derived limit of quantitation.
†The laboratory's limit of quantitation for this analyte exceeds both regulatory limits. The Project Quantitation Limit Goal is the laboratory's statistically derived limit of quantitation.
µg/kg – micrograms per kilogram
CAS – chemical abstract service
NL – Not listed
USEPA – United States Environmental Protection Agency
WDNR – Wisconsin Department of Natural Resources

QAPP WORKSHEET #15E: PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Matrix: Soil
Analytical Group: Polycyclic Aromatic Hydrocarbons
Analytical Method: USEPA SW-846 8270 SIM
Laboratory: Eurofins Environment Testing America in Denver, Colorado

Analyte	CAS Number	WDNR Soil-Groundwater RCL[1] (µg/kg)	WDNR Direct Contact Non-Industrial[2] (µg/kg)	Project Quantitation Limit Goal[3] (µg/kg)	Laboratory-Specific		
					Limit of Quantitation[4] (µg/kg)	Limit of Detection (µg/kg)	Detection Limit (µg/kg)
1-Methylnaphthalene	90-12-0	NL	17,600	1,000	10.0	2.00	0.520
2-Methylnaphthalene	91-57-6	NL	239,000	1,000	10.0	2.00	0.618
Acenaphthene	83-32-9	NL	3,590,000	1,000	10.0	1.07	0.320
Acenaphthylene	208-96-8	NL	NL	LOQ	10.0	1.07	0.340
Anthracene	120-12-7	196,949	17,900	1,000	10.0	4.33	1.44
Benzo(a)anthracene	56-55-3	NL	1,140	1,000	10.0	4.33	1.80
Benzo(a)pyrene	50-32-8	470	42	20	10.0	4.33	1.48
Benzo(b)fluoranthene	205-99-2	478	1,150	200	10.0	6.67	2.40
Benzo(g,h,i)perylene	191-24-2	NL	NL	LOQ	10.0	6.67	2.20
Benzo(k)fluoranthene	207-08-9	NL	11,500	1,000	10.0	4.33	2.00
Chrysene	218-01-9	144	115,000	70	10.0	4.33	2.00
Dibenz(a,h)anthracene	53-70-3	NL	115	50	10.0	6.67	2.60
Fluoranthene	206-44-0	88,877	2,390,000	1,000	10.0	4.33	2.00
Fluorene	86-73-7	14,829	2,390,000	1,000	10.0	2.67	0.940
Indeno(1,2,3-cd)pyrene	193-39-5	NL	1,150	550	10.0	6.67	2.20
Naphthalene	91-20-3	658	5,520	300	10.0	2.00	0.652
Phenanthrene	85-01-8	NL	NL	LOQ	10.0	6.67	2.20
Pyrene	129-00-0	54,545	1,790,000	1,000	10.0	6.67	2.20

Notes:
[1] WDNR Chapter NR 720, Soil Residual Contaminant Level Worksheet, Soil-to-Groundwater Pathway (Dilution Factor = 2), December 2018
[2] WDNR Chapter NR720, Soil Residual Contaminant Level Worksheet, Direct Contact Non-Industrial Land Usage, December, 2018
[3] Whenever possible, the Project Quantitation Limit Goal is equal to or less than one half of the lower regulatory limit. When a target analyte does not have a regulatory limit, the Project Quantitation Limit Goal is the actual limit of quantitation achieved in the sample.
[4] Where the Limit of Quantification (LOQ) is greater than the lowest RCL, the LOQ will be the Project Screening Limit
µg/kg – micrograms per kilogram
CAS – chemical abstract service
LOQ – limit of quantitation
NL – not listed
SW-846 8270 SIM – Semi-volatile Organic Compounds by Gas Chromatography/Mass Spectrometry with Select Ion Monitoring
USEPA – United States Environmental Protection Agency

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QAPP WORKSHEET #15F: PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

Matrix: Soil Vapor
Analytical Group: Volatile Organic Compounds
Analytical Method: USEPA Compendium Method TO-15
Concentration: Low
Laboratory: Eurofins Air Toxics in Folsom, California

Analyte	CAS Number	WDNR VRSL[1] (µg/m^3)	WDNR VRSL[2] (µg/m^3)	Project Quantitation Limit Goal[3] (µg/m^3)	Laboratory-Specific		
					Limit of Quantitation[5] (µg/ m^3)	Limit of Detection (µg/ m^3)	Detection Limit[4] (µg/ m^3)
1,1,1-Trichloroethane	71-55-6	170,000	730,000	1,000	2.7	1.52	0.213
1,1,2,2-Tetrachloroethane	79-34-5	16.1	70.5	8	3.4	2.54	0.364
1,1,2-Trichloroethane	79-00-5	6.95	29.2	3	2.7	2.01	0.360
1,1-Dichloroethane	75-34-3	590	2,600	290	2	1.13	0.210
1,1-Dichloroethene	75-35-4	7,000	29,000	1,000	2	1.46	0.289
1,2,4-Trichlorobenzene	120-82-1	NL	NL	LOQ	15	12.6	3.058
1,2,4-Trimethylbenzene	95-63-6	2,090	8,760	1,000	2.4	1.81	0.359
1,2-Dibromoethane	106-93-4	1.56	6.81	LOQ*	3.8	2.84	0.330
1,2-Dichlorobenzene	95-50-1	6,950	29,200	1,000	3	2.22	0.481
1,2-Dichloroethane	107-06-2	36	160	18	2	1.49	0.291
1,2-Dichloropropane	78-87-5	139	584	60	2.3	1.70	0.314
1,3,5-Trimethylbenzene	108-67-8	2,090	8,760	1,000	2.4	1.81	0.516
1,3-Butadiene	106-99-0	31.2	136	15	1.1	0.99	0.201
1,3-Dichlorobenzene	541-73-1	NL	NL	LOQ	3	2.22	0.415
1,4-Dichlorobenzene	106-46-7	85.1	372	40	3	2.22	0.439
1,4-Dioxane	123-91-1	187	818	90	7.2	2.70	0.515
2,2,4-Trimethylpentane	540-84-1	NL	NL	LOQ	2.3	1.72	0.313
2-Butanone	78-93-3	174,000	730,000	1,000	5.9	5.01	0.516
2-Hexanone	591-78-6	1,040	4,380	500	8.2	6.96	0.246
2-Propanol	67-63-0	NL	NL	LOQ	4.9	3.19	0.838
3-Chloropropene	107-05-1	NL	NL	LOQ	6.3	2.34	0.736
4-Ethyltoluene	622-96-8	NL	NL	LOQ	2.4	1.81	0.359
4-Methyl-2-pentanone	108-10-1	104,000	438,000	1,000	2	1.51	0.315
Acetone	67-64-1	NL	NL	LOQ	12	4.03	0.439
alpha-chlorotoluene	100-44-7	NL	NL	LOQ	2.6	1.91	0.295
Benzene	71-43-2	120	520	60	1.6	1.18	0.125
Bromodichloromethane	75-27-4	25.3	110	10	3.4	1.87	0.389
Bromoform	75-25-2	NL	NL	LOQ	5.2	3.82	0.362
Bromomethane	74-83-9	174	730	80	19	6.60	0.753
Carbon Disulfide	75-15-0	24,300	102,000	1,000	6.2	5.29	0.585
Carbon Tetrachloride	56-23-5	160	680	80	3.1	2.32	0.283
Chlorobenzene	108-90-7	1,740	7,300	850	2.3	1.28	0.161
Chloroethane	75-00-3	139,000	584,000	1,000	5.3	4.48	0.391
Chloroform	67-66-3	41	180	20	2.4	1.36	0.132
Chloromethane	74-87-3	3,100	13,000	1,000	10	3.51	0.335
cis-1,2-Dichloroethene	156-59-2	--	--	LOQ	2	1.46	0.325

Analyte	CAS Number	WDNR VRSL[1] (µg/m^3)	WDNR VRSL[2] (µg/m^3)	Project Quantitation Limit Goal[3] (µg/m^3)	Laboratory-Specific		
					Limit of Quantitation[5] (µg/ m^3)	Limit of Detection (µg/ m^3)	Detection Limit[4] (µg/ m^3)
cis-1,3-Dichloropropene	10061-01-5	234	1,020	100	2.3	1.67	0.425
Cumene	98-82-8	13,900	58,400	1,000	2.4	1.81	0.123
Cyclohexane	110-82-7	209,000	876,000	1,000	1.7	1.27	0.207
Dibromochloromethane	124-48-1	NL	NL	LOQ	4.2	3.15	0.417
Ethanol	64-17-5	NL	NL	LOQ	3.8	3.20	1.922
Ethyl Benzene	100-41-4	370	1,600	180	2.2	1.60	0.252
Freon 11	75-69-4	NL	NL	LOQ	2.8	2.07	0.382
Freon 113	76-13-1	174,000	730,000	1,000	3.8	2.83	0.261
Freon 114	76-14-2	NL	NL	LOQ	3.5	2.58	0.496
Freon 12	75-71-8	3,500	15,000	1,000	2.5	1.82	0.331
Heptane	142-82-5	13,900	58,400	1,000	2	1.51	0.197
Hexachlorobutadiene	87-68-3	42.5	186	21	21	18.1	2.357
Hexane	110-54-3	24,300	102,000	1,000	1.8	1.30	0.201
m, p-Xylene	108-38-3	3,500	15,000	1,000	2.2	1.60	0.122
Methyl tert-butyl ether	1634-04-4	3,600	15,700	1,000	7.2	2.70	0.256
Methylene Chloride	75-09-2	21,000	88,000	1,000	17	5.90	0.271
o-Xylene	95-47-6	3,500	15,000	1,000	2.2	1.60	0.230
Propylbenzene	103-65-1	NL	NL	LOQ	2.4	1.81	0.167
Styrene	100-42-5	34,800	146,000	1,000	2.1	1.57	0.153
Tetrachloroethene	127-18-4	1,400	5,800	700	3.4	2.50	0.373
Tetrahydrofuran	109-99-9	69,500	292,000	1,000	1.5	1.09	0.224
Toluene	108-88-3	174,000	730,000	1,000	1.9	1.39	0.132
trans-1,2-Dichloroethene	156-60-5	1,400	5,800	700	2	1.46	0.468
trans-1,3-Dichloropropene	10061-02-6	NL	NL	LOQ	2.3	1.67	0.163
Trichloroethene	79-01-6	70	290	35	2.7	1.98	0.392
Vinyl Chloride	75-01-4	56	930	25	1.3	0.94	0.233

Notes:
[1] WDNR Vapor Risk Screening Level – Residential Buildings
[2] WDNR Vapor Risk Screening Level – Small Commercial Buildings
[3] Whenever possible, the Project Quantitation Limit Goal is equal to or less than one half of the lowest applicable regulatory limit. When analytical sensitivity is not sufficient, the goal is the laboratory's statistically derived limit of quantitation. When a target analyte does not have a regulatory limit, the Project Quantitation Limit Goal is the actual limit of quantitation achieved in the sample.
[4] Detection limit values are instrument-specific and are generated at regular intervals. Actual values may vary slightly at the time of analysis.
[5] Where the Limit of Quantification (LOQ) is greater than the lowest VRSL, the LOQ will be the Project Screening Limit
*The laboratory's limit of quantitation for this analyte is the WDNR VRSLs. The Project Quantitation Limit Goal is the laboratory's statistically derived limit of quantitation in undiluted samples.
µg/m^3 – micrograms per cubic meter
CAS – chemical abstract service
Compendium TO-15 – Determination of Volatile Organic Compounds in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/ Mass Spectrometry
NL – not listed in either VRSL source
USEPA – United States Environmental Protection Agency
VRSL – Vapor Risk Screening Level; from WDNR Pub RR0136 or determined from USEPA Vapor Intrusion Screening Levels (VISL) Calculator using Wisconsin default value
WDNR – Wisconsin Department of Natural Resources

QAPP WORKSHEET #17: SAMPLING DESIGN AND RATIONALE

Based on criteria outlined in the USEPA Guidance on Choosing a Sampling Design for Environmental Data Collection (USEPA, 2002), judgmental sampling was selected for this SI. Four site areas have been identified with observable soil impacts during UST removals. The boundaries of each of these areas are presented on **Figure 2.** Because the objective of this SI is to define the nature and extent within the study boundary of COPCs within soil, groundwater, and soil vapor, the extent of the SI activities will be limited to the horizontal and vertical limits of proposed sampling areas. The sample design and rationale for each site area is described below. Note that if proposed sample locations are obstructed by utilities or other infrastructure, sample locations will be offset to the nearest location possible from the originally proposed location.

CTU006 (UST 212)

Soil samples will be collected from five boring locations at CTU006, including one boring within the former UST footprint and four additional borings located to the north, south, east, and west of the former UST (**Figure 3**). Soil sample locations were selected by considering potential source areas documented in historical reports and possible migration pathways from the potential source area. Soil borings will be advanced to a maximum depth of 20 feet bgs using DPT. Two discrete soil samples will be collected at each boring location and will be biased toward intervals that exhibit impacted soil (soil staining, petroleum smell, elevated PID measurement). If soil boring intervals do not indicate the presence of impacts, samples will be collected at depths of the former USTs (approximately 10 ft bgs for CTU006) and the 1-ft interval just above the saturated zone.

Three of the five soil borings advanced at CTU006 will be converted to monitoring wells, as illustrated on **Figure 3**. These three wells will facilitate characterization of groundwater below the former UST footprint as well as upgradient and downgradient. Monitoring wells will be installed at a depth of approximately 20 ft bgs to characterize the upper surficial aquifer and evaluate whether residual impacts from the former USTs are present in groundwater. Based on AECOM's recent groundwater monitoring experience on other projects at the airport, groundwater depth is expected to be less than 20 ft bgs. In the unlikely event that groundwater is not encountered within 20 ft, AECOM will advance the boring an additional 5 feet. If groundwater is still not encountered, AECOM will confer with the AFCEC PM to determine the course of action.

Three soil vapor points will also be installed at CTU006 to characterize soil vapor and evaluate risks associated with potential COPC concentrations in soil vapor. The locations were placed within the former UST footprint and adjacent to Building 209 and 212 (**Figure 3**).

Site CTU007 (UST 215)

Soil samples will be collected from eight boring locations at CTU007, including three borings within the three former UST footprints and five additional borings located immediately adjacent to the former USTs (**Figure 4**). Soil sample locations were selected by considering potential source areas documented in historical reports and possible migration pathways from the potential source area. Soil borings will be advanced to a maximum depth of 20 feet bgs using DPT. Two discrete soil samples will be collected at each boring location and will be biased toward intervals that exhibit impacted soil (soil staining, petroleum smell, elevated PID measurement). If soil

boring intervals do not indicate the presence of impacts, samples will be collected at depths of the former USTs (approximately 10 ft bgs for CTU007) and the 1-ft interval just above the saturated zone.

Four of the eight soil borings advanced at CTU007 will be converted to monitoring wells, as illustrated on **Figure 4**. These three wells will facilitate characterization of groundwater below the former UST footprints as well as upgradient and downgradient. Monitoring wells will be installed at a depth of approximately 20 ft bgs to characterize the upper surficial aquifer and evaluate whether residual impacts from the former USTs are present in groundwater.

CTU008 (UST219)

Soil samples will be collected from six boring locations at CTU008, including one boring within the former UST footprint and five additional borings located to the north, south, east, and west of the former UST (**Figure 5**). Soil sample locations were selected by considering potential source areas documented in historical reports and possible migration pathways from the potential source area. Soil borings will be advanced to a maximum depth of 20 feet bgs using DPT. Two discrete soil samples will be collected at each boring location and will be biased toward intervals that exhibit impacted soil (soil staining, petroleum smell, elevated PID measurement). If soil boring intervals do not indicate the presence of impacts, samples will be collected at depths of the former USTs (approximately 11 ft bgs for CTU008) and the 1-ft interval just above the saturated zone.

Three of the five soil borings advanced at CTU008 will be converted to monitoring wells, as illustrated on **Figure 5**. These three wells will facilitate characterization of groundwater below the former UST footprint as well as upgradient and downgradient. Monitoring wells will be installed at a depth of approximately 20 ft bgs to characterize the upper surficial aquifer and evaluate whether residual impacts from the former USTs are present in groundwater.

Three soil vapor points will also be installed at CTU006 to characterize soil vapor and evaluate risks associated with potential COPC concentrations in soil vapor. The locations were placed within the former UST footprint and adjacent to Building 219 and 220 (**Figure 5**).

Site CTU011, CTU012, and CTU013 (UST 8002)

Soil samples will be collected from nine boring locations at CTU011, CTU012, and CTU013; including four borings within the former UST footprints, two borings along the fuel line conveyance, one boring at the former fuel island, and two borings north of the former USTs (**Figure 6**). Soil sample locations were selected by considering potential source areas documented in historical reports and possible migration pathways from the potential source area. Soil borings will be advanced to a maximum depth of 20 feet bgs using DPT. Two discrete soil samples will be collected at each boring location and will be biased toward intervals that exhibit impacted soil (soil staining, petroleum smell, elevated PID measurement). If soil boring intervals do not indicate the presence of impacts, samples will be collected at depths of the former USTs (approximately 10 ft bgs) and the 1-ft interval just above the saturated zone.

Four of the nine soil borings advanced at the site will be converted to monitoring wells, as illustrated on **Figure 6**. These three wells will facilitate characterization of groundwater below the former UST footprints,

below the former fuel island and downgradient. Monitoring wells will be installed at a depth of approximately 20 ft bgs to characterize the upper surficial aquifer and evaluate whether residual impacts from the former USTs are present in groundwater.

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QAPP WORKSHEET #18: SAMPLING LOCATIONS AND METHODS

This worksheet identifies planned samples at sites CTU006 (UST 212), CTU007 (UST 215), CTU008 (UST 219), and CTU011, CTU012 and CTU013 (UST 8002). Samples are either groundwater, soil, or soil vapor matrix.

Location ID	Sample ID	Sample Matrix	Depth (ft bgs)[1,2]	Sample Type	Analytical Group	SOP Reference
CTU006 (UST212)						
CTU006-SB01-01	CTU006-SB01-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU006-SB01-02	CTU006-SB01-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU006-SB02-01	CTU006-SB02-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU006-SB02-02	CTU006-SB02-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU006-SB03-01	CTU006-SB03-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU006-SB03-02	CTU006-SB03-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU006-SB04-01	CTU006-SB04-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU006-SB04-02	CTU006-SB04-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU006-SB05-01	CTU006-SB05-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU006-SB05-02	CTU006-SB05-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU006-SB05-02	CTU006-SB05-02-*FD*	Soil	9 - 10	Field Duplicate	VOCs, PAHs	SOP 3
CTU006-MW01	CTU006-MW01-*mmddyy*	Groundwater	10 - 20	Regular	VOCs, PAHs	SOP 4
CTU006-MW02	CTU006-MW02-*mmddyy*	Groundwater	10 - 20	Regular	VOCs, PAHs	SOP 4
CTU006-MW03	CTU006-MW03-*mmddyy*	Groundwater	10 - 20	Regular	VOCs, PAHs	SOP 4
CTU006-VP01	CTU006-VP01	Soil Vapor	10	Regular	VOCs	NA
CTU006-VP01	CTU006-VP01-FD	Soil Vapor	10	Field Duplicate	VOCs	NA
CTU006-VP02	CTU006-VP02	Soil Vapor	10	Regular	VOCs	NA
CTU006-VP03	CTU006-VP03	Soil Vapor	10	Regular	VOCs	NA
CTU007 (UST 215)						
CTU007-SB01-01	CTU007-SB01-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU007-SB01-02	CTU007-SB01-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU007-SB02-01	CTU007-SB02-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU007-SB02-02	CTU007-SB02-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU007-SB03-01	CTU007-SB03-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3

Location ID	Sample ID	Sample Matrix	Depth (ft bgs)[1,2]	Sample Type	Analytical Group	SOP Reference
CTU007-SB03-02	CTU007-SB03-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU007-SB04-01	CTU007-SB04-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU007-SB04-01	CTU007-SB04-01-MS	Soil	0 - 1	Matrix Spike	VOCs, PAHs	SOP 3
CTU007-SB04-01	CTU007-SB04-01-MSD	Soil	0 - 1	Matrix Spike Duplicate	VOCs, PAHs	SOP 3
CTU007-SB04-02	CTU007-SB04-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU007-SB05-01	CTU007-SB05-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU007-SB05-02	CTU007-SB05-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU007-SB05-02	CTU007-SB05-02-*FD*	Soil	9 - 10	Field Duplicate	VOCs, PAHs	SOP 3
CTU007-SB06-01	CTU007-SB06-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU007-SB06-02	CTU007-SB06-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU007-SB07-01	CTU007-SB07-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU007-SB07-02	CTU007-SB07-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU007-SB08-01	CTU007-SB08-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU007-SB08-02	CTU007-SB08-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU007-MW01	CTU007-MW01	Groundwater	10 - 20	Regular	VOCs, PAHs	SOP 4
CTU007-MW02	CTU007-MW02	Groundwater	10 - 20	Regular	VOCs, PAHs	SOP 4
CTU007-MW03	CTU007-MW03	Groundwater	10 - 20	Regular	VOCs, PAHs	SOP 4
CTU007-MW04	CTU007-MW04	Groundwater	10 - 20	Regular	VOCs, PAHs	SOP 4
CTU008 (UST 219)						
CTU008-SB01-01	CTU008-SB01-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU008-SB01-02	CTU008-SB01-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU008-SB02-01	CTU008-SB02-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU008-SB02-01	CTU008-SB02-01-FD	Soil	0 - 1	Field Duplicate	VOCs, PAHs	SOP 3
CTU008-SB02-02	CTU008-SB02-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU008-SB03-01	CTU008-SB03-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU008-SB03-02	CTU008-SB03-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU008-SB04-01	CTU008-SB04-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU008-SB04-02	CTU008-SB04-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU008-SB05-01	CTU008-SB05-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3

Location ID	Sample ID	Sample Matrix	Depth (ft bgs)[1,2]	Sample Type	Analytical Group	SOP Reference
CTU008-SB05-02	CTU008-SB05-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU008-SB06-01	CTU008-SB06-01	Soil	0 - 1	Regular	VOCs, PAHs	SOP 3
CTU008-SB06-01	CTU008-SB06-01-FD	Soil	0 - 1	Field Duplicate	VOCs, PAHs	SOP 3
CTU008-SB06-02	CTU008-SB06-02	Soil	9 - 10	Regular	VOCs, PAHs	SOP 3
CTU008-MW01	CTU008-MW01-*mmddyy*	Groundwater	10 - 20	Regular	VOCs, PAHs	SOP 4
CTU008-MW02	CTU008-MW02-*mmddyy*	Groundwater	10 - 20	Regular	VOCs, PAHs	SOP 4
CTU008-MW03	CTU008-MW03-*mmddyy*	Groundwater	10 - 20	Regular	VOCs, PAHs	SOP 4
CTU008-MW03	CTU008-MW03-*mmddyy-FD*	Groundwater	10 - 20	Field Duplicate	VOCs, PAHs	SOP 4
CTU008-VP01	CTU008-VP01	Soil Vapor	10	Regular	VOCs	NA
CTU008-VP02	CTU008-VP02	Soil Vapor	10	Regular	VOCs	NA
CTU008-VP02	CTU008-VP02-MS	Soil Vapor	10	Matrix Spike	VOCs	NA
CTU008-VP02	CTU008-VP02-MSD	Soil Vapor	10	Matrix Spike Duplicate	VOCs	NA
CTU008-VP03	CTU008-VP03	Soil Vapor	10	Regular	VOCs	NA
CTU011/012/013 (UST 8002)						
CTU011-SB01-01	CTU011-SB01-01	Soil	0 - 1	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB01-01	CTU011-SB01-01-FD	Soil	0 - 1	Field Duplicate	VOCs, PAHs, Lead	SOP 3
CTU011-SB01-02	CTU011-SB01-02	Soil	9 - 10	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB01-02	CTU011-SB01-02-MS	Soil	9 - 10	Matrix Spike	VOCs, PAHs, Lead	SOP 3
CTU011-SB01-02	CTU011-SB01-02-MSD	Soil	9 - 10	Matrix Spike Duplicate	VOCs, PAHs, Lead	SOP 3
CTU011-SB02-01	CTU011-SB02-01	Soil	0 - 1	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB02-02	CTU011-SB02-02	Soil	9 - 10	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB03-01	CTU011-SB03-01	Soil	0 - 1	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB03-02	CTU011-SB03-02	Soil	9 - 10	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB04-01	CTU011-SB04-01	Soil	0 - 1	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB04-02	CTU011-SB04-02	Soil	9 - 10	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB05-01	CTU011-SB05-01	Soil	0 - 1	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB05-02	CTU011-SB05-02	Soil	9 - 10	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB06-01	CTU011-SB06-01	Soil	0 - 1	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB06-02	CTU011-SB06-02	Soil	9 - 10	Regular	VOCs, PAHs, Lead	SOP 3

Location ID	Sample ID	Sample Matrix	Depth (ft bgs)[1,2]	Sample Type	Analytical Group	SOP Reference
CTU011-SB06-02	CTU011-SB06-02-FD	Soil	9 - 10	Field Duplicate	VOCs, PAHs, Lead	SOP 3
CTU011-SB07-01	CTU011-SB07-01	Soil	0 - 1	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB07-02	CTU011-SB07-02	Soil	9 - 10	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB08-01	CTU011-SB08-01	Soil	0 - 1	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB08-01	CTU011-SB08-01-MS	Soil	0 - 1	Matrix Spike	VOCs, PAHs, Lead	SOP 3
CTU011-SB08-01	CTU011-SB08-01-MSD	Soil	0 - 1	Matrix Spike Duplicate	VOCs, PAHs, Lead	SOP 3
CTU011-SB08-02	CTU011-SB08-02	Soil	9 - 10	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB09-01	CTU011-SB09-01	Soil	0 - 1	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB09-02	CTU011-SB09-02	Soil	9 - 10	Regular	VOCs, PAHs, Lead	SOP 3
CTU011-SB09-02	CTU011-SB09-02-FD	Soil	9 - 10	Field Duplicate	VOCs, PAHs, Lead	SOP 3
CTU011-MW01	CTU011-MW01-*mmddyy*	Groundwater	10 - 20	Regular	VOCs, PAHs, Lead	SOP 4
CTU011-MW01	CTU011-MW01-*mmddyy-FD*	Groundwater	10 - 20	Field Duplicate	VOCs, PAHs, Lead	SOP 4
CTU011-MW02	CTU011-MW02-*mmddyy*	Groundwater	10 - 20	Regular	VOCs, PAHs, Lead	SOP 4
CTU011-MW03	CTU011-MW03-*mmddyy*	Groundwater	10 - 20	Regular	VOCs, PAHs, Lead	SOP 4
CTU011-MW04	CTU011-MW04-*mmddyy*	Groundwater	10 - 20	Regular	VOCs, PAHs, Lead	SOP 4
CTU011-MW04	CTU011-MW04-*mmddyy-MS*	Groundwater	10 - 20	Matrix Spike	VOCs, PAHs, Lead	SOP 4
CTU011-MW04	CTU011-MW04-*mmddyy-MSD*	Groundwater	10 - 20	Matrix Spike Duplicate	VOCs, PAHs, Lead	SOP 4

Notes:
CUES – CTI-URS Environmental Services, LLC
ft bgs – feet below ground surface
ID – Identification
mmddyy – two-digit *month*, two-digit *day*, two-digit *year*
MS/MSD – matrix spike and matrix spike duplicate
PAHs – polynuclear aromatic hydrocarbons
SOP – standard operating procedure
VOCs – volatile organic compounds

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

Laboratory: Eurofins Environment Testing America, 4955 Yarrow Street, Arvada, Colorado 80002
Accreditation: United States Department of Defense Environmental Laboratory Accreditation Program (DoD-ELAP) Accreditation Certificate Number 2907.01
Accreditation Expiration: October 31, 2023
Sample Delivery Method: Shipment (FedEx Overnight)
Data Package Turnaround: 15 Business Days

Analyte Group	Matrix	Method	SOP	Containers per Sample	Preservation	Analytical Holding Time
Metals	Groundwater	Analysis: USEPA SW-846 6020B Preparation: USEPA SW-846 3005A	Analysis: DV-MT-0022 Preparation: DV-IP-0014	1 x 250 mL HDPE	HNO_3 pH<2	180 days
Metals	Soil	Analysis: USEPA SW-846 6020B Preparation: USEPA SW-846 3050	Analysis: DV-MT-0022 Preparation: DV-IP-0015	1 x 4 oz glass jar with Teflon®-lined lid	4 °C, not to exceed 6 °C	180 days
VOCs	Groundwater	Analysis: USEPA SW-846 8260D	DV-MS-0010	3 x 40 mL glass vials	HCl pH<2 4 °C, not to exceed 6 °C	14 days (7 days if pH>2)
VOCs	Soil	Analysis: USEPA SW-846 8260D	DV-MS-0010	3 x 5g Terra Cores, (1 per tared 40 mL glass vial)	5 mL methanol 4 °C, not to exceed 6 °C	14 days
				1 x 4 oz glass jar for percent solids	4 °C, not to exceed 6 °C	none – used for dry weight corrections
PAHs	Groundwater	Analysis: USEPA SW-846 8270 SIM Preparation: USEPA SW-846 3510C	Analysis: DV-MS-0002 Preparation: DV-OP-0006, DV-OP-0007	2 x 1 L amber glass or 1 x 250 mL amber (reduced volume extraction)	4 °C, not to exceed 6 °C	7 days from collection to extraction; 40 days from extraction to analysis
PAHs	Soil	Analysis: USEPA SW-846 8270 SIM Preparation: USEPA SW-846 3550	Analysis: DV-MS-0002 Preparation: DV-OP-0016	1 x 4 oz glass jar	4 °C, not to exceed 6 °C	14 days from collection to extraction; 40 days from extraction to analysis

Notes:
°C – degrees Celsius
< – less than
> – greater than
g – grams
HCl – hydrochloric acid
HDPE – high density polyethylene
HNO_3 – nitric acid
L – liter
mL – milliliter
oz – ounce
PAH – polycyclic aromatic hydrocarbon
pH – potential hydrogen
USEPA – United States Environmental Protection Agency
SOP – standard operating procedure
SW-846 3005 – Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by [flame atomic absorption] FLAA or [inductively coupled plasma] ICP Spectroscopy
SW-846 3050 – Acid Digestion of Sediments, Sludges, and Soils
SW-846 3510C – Separatory Funnel Liquid-Liquid Extraction
SW-846 3550 – Ultrasonic Extraction
SW-846 6020B – Inductively Coupled Plasma – Mass Spectrometry
SW-846 8260D – Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry
SW-846 8270 SIM – Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry with Select Ion Monitoring
VOC – volatile organic compound

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

Laboratory: Eurofins Air Toxics, 180 Blue Ravine Road, Folsom, California 95630

Accreditation: DoD-ELAP Accreditation Certificate Number ADE-1451

Accreditation Expiration: April 27, 2022

Sample Delivery Method: Shipment (FedEx Overnight)

Data Package Turnaround: 21 Days

Analyte Group	Matrix	Method	Laboratory SOP	Containers per Sample	Preservation	Analytical Holding Time
VOCs	Soil Vapor	USEPA Compendium Method TO-15	SOP #6	1 L Summa Canister	None, Ambient Temperature	30 Days

Notes:

°C – degrees Celsius

Compendium Method TO-15 – Determination of Volatile Organic Compounds in Air Collected in Specially-Prepared Canisters and Analyzed by Gas Chromatography/ Mass Spectrometry

L - liter

SOP – standard operating procedure

USEPA – United States Environmental Protection Agency

VOC – volatile organic compound

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QAPP WORKSHEET #20: FIELD QC SUMMARY

This worksheet provides a summary of the types of samples to be collected and analyzed for the project. Worksheet #18 lists sampling locations and sample identifications individually, by site. Worksheet #17 details the minimum required frequency for field Quality Control (QC) and defines the analyte groups. A field QC summary table is also included in Worksheet #18.

Matrix	Analyte	Field Samples	Field Duplicates	Matrix Spike	Matrix Spike Duplicates	Field Blanks	Equipment Blanks	Trips Blanks	Total # Analyses
Soil	VOCs, PAHs, Lead	56	6	3	3	1	6	10	85
Groundwater	VOCs, PAHs, Lead	14	2	1	1	1	3	2	24
Soil Vapor	VOCs	6	1	1	1	0	0	1	10

Notes:

[1] Equipment blanks will be collected daily when non-dedicated sampling is used. The target analyte list for equipment blanks should match the analyte lists of the associated samples (those collected the same day, using the non-dedicated equipment)

[2] Trip blanks will be included in each cooler containing VOC aliquots.

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QAPP WORKSHEET #21: FIELD STANDARD OPERATING PROCEDURES (SOPS)

The standard operating procedures listed in the table below have not been modified for project work.

SOP Number	Title, Revision, Date	Organization	SOP Option or Equipment Type	Comments
SOP 2	Equipment Calibration and Maintenance, 2/23/21	CUES	NA	NA
SOP 3	Soil Sampling, 2/23/21	CUES	NA	NA
SOP 4	Groundwater Sampling, 2/23/21	CUES	Low-Flow Methodology	NA
SOP 6	Groundwater Level Measurement, 2/23/21	CUES	NA	NA
SOP 7	Monitoring Well Installation, 2/23/21	CUES	NA	NA
SOP 8	Well Development, 2/23/21	CUES	NA	NA
SOP 10	Drilling, 2/23/21	CUES	NA	NA
SOP 11	Borehole Abandonment, 2/23/21	CUES	NA	NA
SOP 14	Equipment Decontamination, 2/23/21	CUES	NA	NA
SOP 15	Field Equipment Decontamination, 2/23/21	CUES	NA	NA
SOP 16	IDW Management, 2/23/21	CUES	NA	NA
SOP 17	Sample Handling and Custody, 2/23/21	CUES	NA	NA
SOP 18	Field Documentation, 2/23/21	CUES	NA	NA
NA	Soil Gas Sampling, 4/18/01	USEPA	Summa Cannisters	NA

Notes:
CUES – CTI-URS Environmental Services, LLC
NA – not applicable
SOP – standard operating procedure

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

Field Equipment	Activity	SOP Reference	Title or Position of Responsible Person	Frequency	Acceptance Criteria	Corrective Action
multimeter (pH, temperature, conductivity, DO, ORP)	Calibration, Testing, and Inspection	SOP 2[1], Manufacturer's User Guide	Field Technician	Daily before use and as recommended by the manufacturer.	Manufacturer criteria. Generally, ±20% of expected/certified value	Re-calibrate. If instrument calibration continues to fail, remove the equipment from service and alert the deputy project manager.
turbidimeter	Calibration, Testing, and Inspection	SOP 2[1], Manufacturer's User Guide	Field Technician	Daily before use and as recommended by the manufacturer.	Manufacturer criteria. Generally, ±20% of expected/certified value	Re-calibrate. If instrument calibration continues to fail, remove the equipment from service and alert the deputy project manager.
water level meter	Testing and Inspection	SOP 2[1], Manufacturer's User Guide	Field Technician	Daily before use and as recommended by the manufacturer.	Manufacturer criteria.	Replace batteries. If instrument continues to fail, remove the equipment from service and alert the deputy project manager.
photoionization detector	Calibration, Testing, and Inspection	SOP 2[1], Manufacturer's User Guide	Field Technician	Daily before use and as recommended by manufacturer.	Manufacturer criteria. Generally, ±20% of expected/certified value	Re-calibrate. If instrument calibration continues to fail, remove the equipment from service and alert the deputy project manager.

Notes:
[1] CTI & Associates, Inc. Standard Operating Procedure 2. See Appendix B
% – percent
± – plus or minus
DO – dissolved oxygen
ORP – oxidation reduction potential
pH – potential hydrogen
SOP – standard operating procedure

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QAPP WORKSHEET #23: ANALYTICAL SOPS

Eurofins owns all referenced standard operating procedures (SOPs). The SOPs referenced below are specific to each Eurofins location performing analysis. Full analytical SOPs are included in **Appendix C**.

Eurofins Location	Laboratory SOP	Title, Date, Revision Number	Definitive or Screening Data	Matrix, Analytical Group	Equipment Type	Modified for Project? Y/N
ETA Denver	DV-IP-0014	Acid Digestion of Aqueous Samples for Analysis by ICP-MS [(US)EPA 200.8 and SW-846 3005A, 3020A, and 3050B], 2/18/2022, Revision 15	Preparatory	Groundwater, Metals (Digestion)	Bench equipment: temperature-adjustable digestion block, centrifuge	No
ETA Denver	DV-IP-0015	Acid Digestion of Solids [Method (US)EPA 3050B], 6/11/2021, Revision 16	Preparatory	Soil, Metals (Digestion)	Bench equipment: top-loading balance, temperature-adjustable digestion block	No
ETA Denver	DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW[-]846 3510C and [US]EPA 600 Series, 6/11/2021, Revision 21	Preparatory	Groundwater, PAHs (Extraction)	Bench equipment: separatory funnel, balance, graduated cylinder, pipettes.	No
ETA Denver	DV-OP-0007	Concentration and Clean-Up of Organic Extracts (SW-846 3510C, 3540C, 3546, 3550B, 3550C, 3620C, 3630C, 3660B, 3665A, ASTM Method D7065-11, and [US]EPA 600 Series Methods), 12/23/2021, Revision 16	Preparatory	Groundwater and Soil, PAHs (Concentration of Extracts)	Bench equipment: evaporator with thermostat-controlled bath, glassware.	No
ETA Denver	DV-OP-0016	Ultrasonic Extraction of Solid Samples (SW-846 3550B & 3550C), 06/11/2021, Revision 15	Preparatory	Soil, PAHs (Extraction)	Bench equipment: Sonicator, balance, glassware, vacuum pump	No

Eurofins Location	Laboratory SOP	Title, Date, Revision Number	Definitive or Screening Data	Matrix, Analytical Group	Equipment Type	Modified for Project? Y/N
ETA Denver	DV-MS-0002	Polynuclear Aromatic Hydrocarbons by GC/MS Selected Ion Monitoring (SIM) [SW-846 Method 8270C/D/E], 5/21/2021, Revision 16	Definitive	Groundwater and Soil, PAHs	GC/MS	No
ETA Denver	DV-MS-0010	Determination of Volatile Organics by GC/MS (8260B/C/D and 624/624.1), 9/17/2021, Revision 28	Definitive	Groundwater and Soil, VOCs	GC/MS	No
ETA Denver	DV-MT-0022	Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020A/B	Definitive	Groundwater and Soil, Metals	ICP/MS	No
Air Toxics	SOP #6	Method: [US]EPA Method TO-14A/TO-15 Volatile Organic Compounds (Standard or Quad), 4/12/2021, Revision 44	Definitive	Air, VOCs	GC/MS	No

Notes:
Air Toxics – Eurofins Air Toxics in Folsom, California
ASTM – American Society for Testing and Materials
ETA Denver – Eurofins Environment Testing America in Denver, Colorado
GC/MS – gas chromatography/mass spectrometry
ICP/MS – inductively-coupled plasma/mass spectrometry
PAH – polycyclic aromatic hydrocarbon
SIM – select ion monitoring
USEPA – United States of America Environmental Protection Agency
VOC – volatile organic compound

QAPP WORKSHEET #24A: ANALYTICAL INSTRUMENT CALIBRATION

Inductively Coupled Plasma/Mass Spectrometry (ICP/MS) in Soil and Groundwater

Instrument	Calibration Procedure	Calibration Frequency	Acceptance Criteria	CA	Person(s) responsible for CA	SOP Reference
ICP/MS	Instrument Detection Limit Study	At initial set-up, after significant change in instrument type, personnel, test method, or sample matrix.	Calculated instrument detection limits < LOD	Not Applicable	Lab Manager/ Analyst	DV-MT-0022
ICP/MS	Linear dynamic range or high-level check standard	Every 6 months	Within ±10% of the true value for all target analytes.	Dilute samples within the calibration range or re-establish/verify the linear dynamic range.	Lab Manager/ Analyst	DV-MT-0022
ICP/MS	Tuning	Prior to initial calibration	Mass calibration ≤ 0.1 amu from true value; Resolution < 0.9 amu full width at 10% peak height.	Correct problem then retune instrument and verify. No Samples shall be analyzed without a valid tune.	Lab Manager/ Analyst	DV-MT-0022
ICP/MS	ICAL per manufacturer's instructions, with a minimum of one standard and a calibration blank	Daily Initial calibration prior to sample analysis	Correlation coefficient >0.99 (if more than one point).	The validity of the calibration is determined by the subsequent calibration verifications. If invalid, identify and correct problem, then repeat ICAL.	Lab Manager/ Analyst	DV-MT-0022
ICP/MS	Second-source ICV, prepared at the calibration midpoint	Once after each ICAL, prior to beginning analytical run	Within ±10% of the true value for all target analytes.	Evaluate standards and instrument response. If standard issue, repeat or remake then repeat standard as appropriate. If still fails, repeat initial calibration.	Lab Manager/ Analyst	DV-MT-0022
ICP/MS	Initial Calibration Blank	Immediately after each ICV	Absolute values of all analytes must be < ½ LOQ or <1/10 the amount measured in any sample. Non-detects associated with positive blank infractions may be reported. Sample results >10 times LOQ associated with negative blanks may be reported.	Reanalyze once. If acceptable, continue. If unacceptable, terminate analysis; correct the problem, recalibrate the instrument, verify calibration and rerun all samples since the last acceptable CCB.	Lab Manager/ Analyst	DV-MT-0022
ICP/MS	Low concentration standard at or near the reporting limit	Daily, after one point calibration	Within ±20% of the true value for all target analytes.	Evaluate standard and instrument response. If problem with instrument (autosampler failure, response poor, etc.) or standards, correct as appropriate, then repeat. If still fails, repeat initial calibration.	Lab Manager/ Analyst	DV-MT-0022
ICP/MS	CCV, same source as Initial Calibration	Following initial calibration, after every 10 samples and the end of the sequence	Within ±10% of the true value for all target analytes.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Lab Manager/ Analyst	DV-MT-0022

Instrument	Calibration Procedure	Calibration Frequency	Acceptance Criteria	CA	Person(s) responsible for CA	SOP Reference
ICP/MS	CCB	Immediately following each CCV	Absolute values of all analytes must be < ½ RL or <1/10 the amount measured in any sample. Non-detects associated with positive blank infractions may be reported. Sample results >10x LOQ associated with negative blanks may be reported.	Reanalyze once. If acceptable, continue. If unacceptable, terminate analysis; correct the problem, recalibrate the instrument, verify calibration and rerun all samples since the last acceptable CCB.	Lab Manager/ Analyst	DV-MT-0022

Notes:
< – less than
≤ – less than or equal to
% – percent
± – plus or minus
amu – atomic mass units
CCB – continuing calibration blank
CCV – continuing calibration verification
ICAL – initial calibration
ICP/MS – inductively coupled plasma/mass spectrometry
ICV – initial calibration verification
LOD – limit of detection
LOQ – limit of quantitation

QAPP WORKSHEET #24B: ANALYTICAL INSTRUMENT CALIBRATION

Volatile Organic Compounds (VOCs) and Polycyclic Aromatic Hydrocarbons (PAHs) in Soil and Groundwater

Instrument	Calibration Procedure	Calibration Frequency	Acceptance Criteria	CA	Person(s) responsible for CA	SOP Reference
GC/MS	Tune Check, mass spectral ion intensities using BFB (VOCs) or DFTPP (PAHs).	Prior to ICAL and prior to each 12-hour period of sample analysis	Method-specified ion abundance criteria.	Retune instrument and verify	Lab Manager/ Analyst	DV-MS-0010, DV-MS-0002
GC/MS (PAHs only)	Performance Check – PAHs only.	Prior to sample analysis, at the beginning of each 12-hour period.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol present at normal responses. Tailing factor for each <2.	Correct problem (inspect/change liner, clip front end of column, or other maintenance as indicated), then repeat the performance check.	Lab Manager/ Analyst	DV-MS-0002
GC/MS	ICAL minimum five-point linear or six-point quadratic initial calibration for all analytes and surrogates. Lowest concentration standard at or below limit of quantitation.	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	Each analyte must meet one of the three options: Option 1: RSD for each analyte ≤ 15% Option 2 & 3: linear least squares regression or non-linear least squares regression (quadratic) for each analyte: r2≥0.99	Verify standard solutions are valid, perform instrument maintenance if needed, then repeat ICAL.	Lab Manager/ Analyst	DV-MS-0010, DV-MS-0002
GC/MS	Retention Time window position establishment and evaluation	Once per ICAL and at the beginning of the analytical sequence for each analyte and surrogate.	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.	Not Applicable.	Analyst	DV-MS-0010, DV-MS-0002
GC/MS	Evaluation of RRT	With each sample	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst	DV-MS-0010, DV-MS-0002
GC/MS	ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of the true value	Correct problem and verify second source standard. Rerun ICV. If that fails, repeat ICAL.	Lab Manager/ Analyst	DV-MS-0010, DV-MS-0002
GC/MS	CCV	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes (except pentachlorophenol) and surrogates within ±20% of the true value. If pentachlorophenol is a target analyte, it must be within ±50% of true value. All reported analytes and surrogates ≤50% for end of analytical batch CCV.	Immediately analyze two consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) and recalibrate. Then reanalyze all associated samples since last acceptable CCV.	Lab Manager/ Analyst	DV-MS-0010, DV-MS-0002
GC/MS	Internal Standards	During acquisition of calibration standard.	Retention time within ± 10 seconds from retention time of midpoint standard in ICAL. EICP area within -50% to +100% of ICAL midpoint standard.	Inspect instrument for malfunctions. Reanalysis of samples analyzed during system malfunction is mandatory.	Lab Manager/ Analyst	DV-MS-0010, DV-MS-0002

Notes:

≥ – greater than or equal to
≤ – less than or equal to
% – percent
± – plus or minus
µg/L – micrograms per liter
BFB – bromofluorobenzene
CA – corrective action
CCV – continuing calibration verification
DDT – dichlorodiphenyltrichloroethane
DFTPP – decafluorotriphenylphosphine
GC/MS – gas chromatography/mass spectrometry
EICP – extracted ion current profile
ICV – initial calibration verification
ICAL – initial calibration
NA – not applicable
PAH – polycyclic aromatic hyrdocarbon
r^2 – regression factor
RRT – relative retention time
RSD – relative standard deviation
SOP – standard operating procedure
VOC – volatile organic compound

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QAPP WORKSHEET #24C: ANALYTICAL INSTRUMENT CALIBRATION

Volatile Organic Compounds (VOCs) in Soil Vapor

Instrument	Calibration Procedure	Calibration Frequency	Acceptance Criteria	CA	Person(s) Responsible for CA	Laboratory SOP Reference
GC/MS (VOCs only)	Tune Check, mass spectral ion intensities using BFB.	Prior to ICAL and every 24-hours during sample analysis	Method-specified ion abundance criteria.	Retune instrument and verify	Bench Analyst/ Scientist	SOP #6
GC/MS	ICAL: Minimum five-point linear or six-point quadratic initial calibration for all targets. Lowest concentration standard at or below limit of quantitation. One standard must be the same concentration as the daily CCV.	Initial calibration prior to sample analysis.	Each analyte must meet one of the three options: Option 1: RSD for each analyte < 30% Option 2 & 3: linear least squares regression or non-linear least squares regression (quadratic) for each analyte: r2≥0.99	Correct the problem, then repeat initial calibration.	Bench Analyst/ Scientist	SOP #6
GC/MS	Retention Time window position establishment and evaluation	Once per ICAL and at the beginning of the analytical batch.	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.	Not Applicable.	Bench Analyst/ Scientist	SOP #6
GC/MS	Evaluation of RRT	With each sample	RRT of each reported analyte within ± 0.06 RRT units of the mean RRT of the calibration standards. RRTs may be updated daily based on the daily CCV.	Correct problem. Rerun ICV. If that fails, rerun ICAL.	Bench Analyst/ Scientist	SOP #6
GC/MS	Internal Standards	Every field sample, standard, blank, and QC sample.	Within the ICAL, the area response for each IS within ± 40% of the mean area response of all calibration standards.	Inspect mass spectrometer and GC for malfunctions and correct problem.	Bench Analyst/ Scientist	SOP #6
GC/MS	ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 30% of the true value	Correct problem and repeat ICV. If that fails, repeat ICAL.	Bench Analyst/ Scientist	SOP #6
GC/MS	CCV	Daily after tune check and before sample analysis; after every 24 hours of analysis time; and at the end of the analytical batch.	The CCV concentration must be at or below the concentration of the mid-point calibration standard. All reported analytes and surrogates within ±30% of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, perform corrective action(s) until a passing CCV is obtained, and then analyze all samples since the previous acceptable CCV. Alternately, perform ICAL and reanalyze all associated samples since last acceptable CCV.	Bench Analyst/ Scientist	SOP #6

Notes:
≥ – greater than or equal to
≤ – less than or equal to
% – percent
± – plus or minus
µg/L – micrograms per liter
BFB – bromofluorobenzene
CA – corrective action
CCV – continuing calibration verification
DDT – dichlorodiphenyltrichloroethane
ICV – initial calibration verification
ICAL – initial calibration
IS – internal standard
NA – not applicable
r^2 – regression factor
RRT – relative retention time
RSD – relative standard deviation
SOP – standard operating procedure
GC – gas chromatograph
GC/MS – gas chromatography/mass spectrometry

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

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Person(s) Responsible for CA	SOP Reference
ICPMS	Change pump tubing, clean torch, check/clean nebulizer	None	Preventative Maintenance	Weekly	None	None	Eurofins Denver Chemist	DV-MT-0022
ICPMS	Clean sample and skimmer cones, clean/flush nebulizer	Tuning Check	Instrument tuning performance check	Daily	SOP Section 10.6	Check pump tubing, clean lenses as needed.	Eurofins Denver Analyst	DV-MT-0022
ICPMS	Replace pump tubing	Tuning Check	Instrument performance and sensitivity	Weekly or as needed	SOP Section 10.6	Check valves, clean lenses as needed.	Eurofins Denver Analyst	DV-MT-0022
ICPMS	Clean lens, torch, injector	Tuning Check	Instrument performance and sensitivity	Monthly or as needed	SOP Section 10.6	Check valves, clean lenses as needed.	Eurofins Denver Analyst	DV-MT-0022
ICPMS	Check and clean air filters, verify coolant level	None	Preventative Maintenance	Monthly	None	None	Eurofins Denver Analyst	DV-MT-0022
ICPMS	Change vacuum pump oil	Verify Vacuum Pressure	Preventative Maintenance	Semi-annually or as needed	<100 Pa	None	Eurofins Denver Analyst	DV-MT-0022
ICPMS	Manufacturer Preventive Maintenance	None	Preventative Maintenance	Bi-annually or annually, per service agreement	None	None	Manufacturer	DV-MT-0022

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Person(s) Responsible for CA	SOP Reference
GC/MS	Clean sources,maintain vacuum pumps	Tuning	Instrument performance and sensitivity	Service vacuum pumps twice per year, other maintanenace as needed	Tune and CCV pass method-specified criteria.	Recalibrate instrument	Chemist	DV-MS-0010, DV-MS-0002
GC/MS	Change septum, clean injection port, change or clip column, install new liner, change trap	Response factors and chromatogram review	Instrument performance and sensitivity	As needed	Tune and CCV pass method-specified criteria.	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Chemist	DV-MS-0010, DV-MS-0002
GC/MS	Preventative maintenance	Instrument performance checks	Ion source, injector liner, column, column flow, purge lines, trap	Varies, from daily to every 6 months and as needed	Instrument performance meets Worksheet #24b criteria	Correct the proeblem and repeat calibration or calibration verification	Bench Analyst/ Scientist/ Engineer	SOP #6, SOP #10

Notes:
CA – corrective action
CCV – continuing calibration verification
GC/MS – gas chromatograph/mass spectrometer
Pa - pascal
SOP – standard operating procedure

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

Sampling Organization: CTI-URS Environmental Services, LLC

Laboratory: Eurofins ETA Denver, LLC

Method of sample delivery: Shipper - FedEx Overnight Delivery

Number of days from reporting until sample disposal: 21 Days

Activity	Organization and title of responsible party	SOP Reference
Sample labeling	CUES, Field Manager	SOP 17
Chain of custody form completion		
Sample packaging		
Shipping coordination		
Sample receipt, inspection, & log-in	Eurofins ETA Denver, Sample receipt personnel	DV-QA-0003
Sample custody and storage	Eurofins ETA Denver, Sample receipt personnel	DV-QA-0003
Sample disposal	Eurofins ETA Denver, Waste management personnel	DV-HS-001P

Notes:
CUES – LATA and CTI Environmental Services, LLC
Eurofins ETA Denver – Eurofins Environmental Testing America in Denver, Colorado
SOP – standard operating procedure
Standard operating procedures are included in Appendix B
Examples of chain of custody form, sample labels, and sample receipt forms are included in Appendix A.

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QAPP WORKSHEET #28A: ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION

Matrices: Groundwater and Soil
Analytical Group: Lead
Analytical Method/SOP: United States Environmental Protection Agency SW-846 6020B/DV-MT-0022

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	CA	Person(s) Responsible for CA	Project-Specific MPC
Internal Standards	Each field and QC sample	IS intensity within 30-120% of the IS in the ICAL	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. Reanalyze sample at 5-fold dilutions until criteria is met. For failed QC samples, correct problem and rerun all associated failed field samples.	Lab Manager/ Analyst	Accuracy/ Bias
Method Blank	One per digestion batch	No analytes detected > ½ LOQ, or >1/10th the amount measured in any sample, or 1/10th the regulatory limit, whichever is greater.	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory, correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM 5.3 requirements.	Lab Manager/ Analyst	Bias Contamination
ICS	At the beginning of the analytical run, and every 12 hours.	ICS-A: Absolute value of concentration for all non-spiked project analytes <1/2 LOQ (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis, correct problem, then reanalyze ICS and all affected samples in accordance with DoD QSM requirements	Lab Manager/ Analyst	Accuracy
LCS	One per preparatory batch	QC acceptance criteria as specified by DoD QSM 5.3 table C-5 and C-6: Lead in soil (table C-5): 84 – 118% Lead in water (table C-6): 88 – 115%	Evaluate LCS data and reanalyze if bias appears instrument related. If bias appears preparation related, determine if trend requires correction prior to repreparation and reanalysis of the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Lab Manager/ Analyst	Accuracy/ Bias
MS and MSD	One MS and MSD pair per preparatory batch	QC acceptance criteria as specified by DoD QSM 5.3 table C-5 and C-6: Lead in soil (table C-5): 84 – 118% Lead in water (table C-6): 88 – 115%	Examine the project specific DQOs. Evaluate the data and re-prepare/reanalyze the native sample and MS/MSD pair as indicated.	Lab Manager/ Analyst	Accuracy/ Bias, Precision
Dilution Test	One per batch of 20 samples or less	Five-fold dilution must agree within ±10% of the original determination in accordance with DoD QSM 5.3 requirements. Only applicable to samples with concentrations > 50 times the LOQ prior to dilution.	Perform post-digestion spike addition in accordance with DoD QSM requirements.	Lab Manager/ Analyst	Precision
Post Digestion Spike	Perform when MS/MSD fails or analyte concentration of all samples is < 50 times the LOQ	Recovery within 80% to 120% of expected results in accordance with DoD QSM 5.3 requirements Only applicable to samples with concentrations < 50 times the LOQ prior to dilution.	Flag in accordance with DoD QSM 5.3 requirements.	Lab Manager/ Analyst	Accuracy

Notes:
< - less than
> – greater than
% – percent
± – plus or minus
CA – corrective action
DoD QSM 5.3 – Department of Defense Quality Systems Manual Version 5.3
ICAL – initial calibration
ICS – interference check standard
IS – internal standard
LCS – laboratory control sample
LOQ – limit of quantitation
MS – matrix spike
MSD – matrix spike duplicate
QC – quality control

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QAPP WORKSHEET #28B: ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION

Matrices: Groundwater and Soil
Analytical Group: Volatile Analytical Compounds (VOCs)
Analytical Method/SOP: United States Environmental Protection Agency SW-846 8260D/DV-MS-0010

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	CA	Person(s) Responsible for CA	Project-Specific MPC
Internal Standards	Each calibration standard, sample, and QC sample	Retention times within ±10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of the ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning in accordance with DoD QSM 5.3 requirements. If field samples are still outside of criteria, qualify data and explain in case narrative.	Analyst/ Section Supervisor	Accuracy/ Bias
Method Blank	One per preparatory batch (maximum 20 samples)	No analytes detected > ½ LOQ, or >1/10th the amount measured in any sample, or 1/10th the regulatory limit, whichever is greater. For common laboratory contaminants, no analytes detected > LOQ. Data may not be reported without a valid Method Blank.	If sufficient sample is available, re-prepare and reanalyze samples. Qualify data as needed.	Analyst/ Section Supervisor	Bias Contamination
LCS	One per preparatory batch (maximum 20 samples)	Recovery limits from DoD QSM 5.3 Table C-23 for soil samples and Table C-24 for groundwater samples. For target analytes not listed in DoD QSM 5.3 tables C-23 and C-24, recovery within laboratory-established acceptance criteria.	Reanalyze the LCS once. If acceptable, report. Otherwise: evaluate, re-prepare, and reanalyze the LCS and all samples in the associated preparatory batch for the failed analytes (if sufficient sample remains).	Analyst/ Section Supervisor	Accuracy/ Bias
MS and MSD	One MS and MSD pair per preparatory batch (maximum 20 samples)	Recovery limits from DoD QSM 5.3 Table C-23 for soil samples and Table C-24 for groundwater samples. For target analytes not listed in DoD QSM 5.3 tables C-23 and C-24, recovery within laboratory-established acceptance criteria. RPD (between MS and MSD) for all reported targets ≤20%	Determine the root cause, flag MS/MSD data, and discuss in the case narrative.	Analyst/ Section Supervisor	Accuracy/ Bias, Precision
Surrogate Compounds	Every field and QC sample.	Recovery limits from DoD QSM 5.3 Table C-23 for soil samples and Table C-24 for groundwater samples. For surrogate compounds not listed in DoD QSM 5.3 tables C-23 and C-24, recovery within laboratory-established acceptance criteria.	Correct the problem, then reprepare and reanalyze all failed samples for all surrogates if sufficient sample remains. If samples will not be reanalyzed due to obvious chromatographic interference, notify the Project Chemist and Project Manager per Worksheet #6 prior to reporting. All surrogate compound recovery failures must be discussed in the Case Narrative	Analyst/ Section Supervisor	Accuracy/ Bias

Notes:
< - less than
≤ – less than or equal to
> – greater than
% – percent
± – plus or minus
CA – corrective action
CCV – continuing calibration verification
DoD QSM 5.3 – Department of Defense Quality Systems Manual Version 5.3
EICP – extracted ion current profile
GC – gas chromatograph
GC/MS – gas chromatography/ mass spectrometry
ICAL – initial calibration
LCS – laboratory control sample
LOQ – limit of quantitation
MPC – measurement performance criteria
MS – matrix spike
MSD – matrix spike duplicate
QC – quality control
RPD – relative percent difference
SOP – standard operating procedure
SW-846 8260D – Volatile Organic Compounds by GC/MS

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QAPP WORKSHEET #28C: ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION

Matrix: Groundwater and Soil
Analytical Group: Polycyclic Aromatic Hydrocarbons
Analytical Method/SOP: United States Environmental Protection Agency SW-846 8270 SIM/DV-MS-0002

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	CA	Person(s) Responsible for CA	Project-Specific MPC
Internal Standards	Each calibration standard, sample, and QC sample	Retention times within ±10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of the ICAL midpoint standard. On days when ICAL is not performed, the daily initial CCV can be used.	Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning in accordance with DoD QSM 5.3 requirements. If field samples are still outside of criteria, qualify data and explain in case narrative.	Analyst/ Section Supervisor	Accuracy/ Bias
Method Blank	One per preparatory batch (maximum 20 samples)	No analytes detected > ½ LOQ, or >1/10th the amount measured in any sample, or 1/10th the regulatory limit, whichever is greater. For common laboratory contaminants, no analytes detected > LOQ. Data may not be reported without a valid Method Blank.	If sufficient sample is available, re-prepare and reanalyze samples. Qualify data as needed.	Analyst/ Section Supervisor	Bias Contamination
LCS	One per preparatory batch (maximum 20 samples)	Recovery limits from DoD QSM 5.3 Table C-25 for soil samples and Table C-26 for groundwater samples. For target analytes not listed in DoD QSM 5.3 tables C-25 and C-26, recovery within laboratory-established acceptance criteria.	Reanalyze the LCS once. If acceptable, report. Otherwise: evaluate, re-prepare, and reanalyze the LCS and all samples in the associated preparatory batch for the failed analytes (if sufficient sample remains).	Analyst/ Section Supervisor	Accuracy/ Bias
MS and MSD	One MS and MSD pair per preparatory batch (maximum 20 samples)	Recovery limits from DoD QSM 5.3 Table C-27 for soil samples and Table C-28 for groundwater samples. For target analytes not listed in DoD QSM 5.3 tables C-27 and C-28, recovery within laboratory-established acceptance criteria. RPD (between MS and MSD) for all reported targets ≤20%	Determine the root cause, flag MS/MSD data, and discuss in the case narrative.	Analyst/ Section Supervisor	Accuracy/ Bias, Precision
Surrogate Compounds	Every field and QC sample.	Recovery limits from DoD QSM 5.3 Table C-27 for soil samples and Table C-28 for groundwater samples. For target analytes not listed in DoD QSM 5.3 tables C-27 and C-28, recovery within laboratory-established acceptance criteria.	Correct the problem, then reprepare and reanalyze all failed samples for all surrogates if sufficient sample remains. If samples will not be reanalyzed due to obvious chromatographic interference, notify the Project Chemist and Project Manager per Worksheet #6 prior to reporting. All surrogate compound recovery failures must be discussed in the Case Narrative	Analyst/ Section Supervisor	Accuracy/ Bias

Notes:
< - less than
≤ – less than or equal to
> – greater than
% – percent
± – plus or minus
CA – corrective action
CCV – continuing calibration verification
DoD QSM 5.3 – Department of Defense Quality Systems Manual Version 5.3
EICP – extracted ion current profile
GC – gas chromatograph
GC/MS – gas chromatography/ mass spectrometry
ICAL – initial calibration
LCS – laboratory control sample
LOQ – limit of quantitation
MPC – measurement performance criteria
MS – matrix spike
MSD – matrix spike duplicate
QC – quality control
RPD – relative percent difference
SOP – standard operating procedure
SW-846 8270 SIM – Semivolatile Organic Compounds by GC/MS with Select Ion Monitoring

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QAPP WORKSHEET #28D: ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION

Matrix: Air
Analytical Group: Volatile Organic Compounds
Analytical Method/SOP: United States Environmental Protection Agency Compendium Method TO-15/SOP #6

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	CA	Person(s) Responsible for CA	Project-Specific MPC
Internal Standards	Each calibration standard, sample, and QC sample	Field samples, blanks, and QC: Retention times within ±0.33 minutes from retention time of the most recent valid calibration, either ICAL midpoint standard or most recent CCV, as appropriate. Area response for each IS within ±40% of the mean area response of the ICAL midpoint or CCV (whichever is most recent) and retention time shift for each IS within 20 seconds of the mean retention time.	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.	Bench Analyst/ Scientist	Accuracy/ Bias
Method Blank	One per analytical batch, after the first CCV and prior to analysis of field samples.	No analytes detected > ½ LOQ, or >1/10th the amount measured in any sample, or 1/10th the regulatory limit, whichever is greater. For common laboratory contaminants, no analytes detected > LOQ. Data may not be reported without a valid Method Blank.	Inspect the system and re-analyze the blank and all samples if analysis was performed with the contaminated blank. Qualify data as needed.	Bench Analyst/ Scientist	Bias Contamination
LCS/ LCSD	One LCS and LCSD pair per analytical batch	Recovery limits from DoD QSM 5.3 Table C-43. RPD (between LCS and LCSD) for all reported targets ≤30% Initial and closing CCV can serve as LCS and LCSD.	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. Reanalyze samples if analysis was performed with a failed LCS.	Bench Analyst/ Scientist	Accuracy/ Bias, Precision
Surrogate Compounds	All standards, blanks, and samples	Recovery within 70 to 130%.	Blanks: Inspect the system and reanalyze the blank. Samples: Re-analyze the sample unless obvious matrix interference is documented. If the reanalysis recovery is within control limits, report the second analysis. If recovery is outside of limits a second time, narrate the results.	Bench Analyst/ Scientist	Accuracy/ Bias

Notes:
≤ – less than or equal to
> – greater than
% – percent
± - plus or minus
CA – corrective action
CCV – continuing calibration verification
DoD QSM 5.3 – Department of Defense Quality Systems Manual Version 5.3
ICAL – initial calibration
IS – internal standard
LCS – laboratory control sample
LCSD – laboratory control sample duplicate
LOQ – limit of quantitation
MB – method blank
MPC – measurement performance criteria
QC – quality control
RPD – relative percent difference
SOP – standard operating procedure

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QAPP WORKSHEET #29: PROJECT DOCUMENTS AND RECORDS

Sample Collection and Field Records

Record	Generation	Verification	Storage Location/Archival
Field logbooks	Field Team	Field Manager	Project files
Sample location coordinates	Field Team	Field Manager	Project Files, ERPIMS
Chain of Custody forms	Field Team	Field Manager	Copy in project files
Shipping records	Field Team	Field Manager	Project files
Deviations	Field Team	Deputy Project Manager	Project files
Field Corrective Action reports	Field Manager	Deputy Project Manager	Project files
Correspondence	Project Team	Deputy Project Manager	Project files

Notes:
ERPIMS – Environmental Resources Program Information Management System

Locations recorded in field documentation records will be stored in project files. Project chemist will input verified sample location coordinates into Air Force Civil Engineering Center Environmental Resources Program Information Management System database.

Laboratory Analysis Documents and Records

Record	Generation	Verification	Storage Location/Archival
Analytical data package[1]	Laboratory	Project Chemist	Project files
Electronic Data Deliverables	Laboratory	Project Chemist	ERPIMS
Laboratory Corrective Action Reports	Laboratory	Project Chemist	Project files
Analytical laboratory data packages	Laboratory	Project Chemist	Project files

Notes:
ERPIMS – Environmental Resources Program Information Management System

Analytical data packages shall include all elements required for Stage 4 validation as defined in the Department of Defense Quality Systems Manual Version 5.3 Appendix A Section 7.0. Elements include chain of custody forms, sample receipt documentation, standard traceability logs, instrument calibration logs, sample preparation worksheets/logs, sample analysis worksheets and run logs, sample results forms, QC sample results, chromatograms and instrument printouts, and other raw data as relevant.

Data Assessments and Records

Record	Generation	Verification	Storage Location/Archival
Data Validation Report	Data Validator/ Project Chemist	Project Chemist/ CUES QA Manager	Project files
Validation Qualifiers	Data Validator/ Project Chemist	Project Chemist/ CUES QA Manager	ERPIMS
Validation Corrective Actions	Project Chemist	Project Chemist/ CUES QA Manager	Project files
Data Usability Assessment	Project Chemist	Project Team	Project files

Notes:
CUES – LATA and CTI Environmental Services, LLC
ERPIMS – Environmental Resources Program Information Management System
QA – quality assurance

QAPP WORKSHEET #31, 32, & 33: ASSESSMENTS AND CORRECTIVE ACTIONS

Field, laboratory, and data review performance will be assessed following SI fieldwork activities. A description of this process follows.

Field Performance Assessment

Field performance will be assessed for compliance with the following:

- Field samples were collected at the correct locations.
- Field quality control (QC) samples were collected at the required frequencies.
- Chain of Custody documentation reflects proper procedures.
- Correct analytical methods were requested on chain of custody forms.
- Field sampling forms and logbooks contain sufficient information to support data quality objectives.

Compliance will be assessed during data validation and verification after each quarterly event and annually during preparation of the LUC Inspection and Groundwater Monitoring Report. Nonconformances will be addressed when they are discovered and documented in these reports as applicable.

Data Performance Assessments

Data validation described in Worksheet #36 include the necessary elements of a data performance audit. Validation will be completed within three (3) weeks of receipt of the final laboratory report and ERPIMS electronic data deliverable (EDD), and will include:

- Verifying samples were analyzed for the correct analytes by the methods requested on the chain of custody forms.
- Verifying the correct units e.g., micrograms per liter, were reported.
- Verifying that non-detect results are reported at the concentration of the limit of detection.

Submission of validated EDDs to the Environmental Resources Program Information Management System will occur within 90 days of sample collection, unless an extension is granted by written waiver. The process includes the following performance checks on laboratory EDDs:

- Screening EDDs for accuracy by checking at least 10 percent of reported results against the hard-copy deliverables.
- Screening EDDs to ensure the same sample identifications, analytical methods, and specific analytes match hard-copy deliverables and requirements in Worksheet #18.

If non-compliance is found during data performance assessments, corrective actions will be communicated as indicated on Worksheet #6. The laboratory will document the issue on a laboratory nonconformance form, determine the cause and document corrective actions in accordance with their approved Quality Assurance Manual (however named). The laboratory will supply CTI with a Nonconformance Corrective Action Report, which will be retained in the project file.

Data Validation Assessments

Data validation reports will be reviewed for clarity and content by the Deputy Project Manager after completion. Data validation reports will be included in the Annual Groundwater Monitoring and LUC Inspection report for Independent Technical Review.

QAPP WORKSHEET #34: DATA VERIFICATION AND VALIDATION INPUTS

Data verification is a completeness check, ensuring all planned sampling collection and analysis have been completed with satisfactory documentation. Data validation evaluates the conformance of analytical data to the specifications within the approved project Uniform Federal Policy-Quality Assurance Project Plan (UFP-QAPP).

Item	Description	Verification	Validation
Planning and Guidance Documents			
1	Approved UFP-QAPP	X	X
2	Contract	X	
3	Field SOPs	X	
5	Laboratory SOPs	X	X
6	DoD Quality Systems Manual Version 5.3		X
7	DoD General Data Validation Guidelines		X
8	Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by GC/MS (DoD, 2020)		X
Field Records			
9	Field logbooks	X	X
10	Field Worksheets	X	X
11	Chain of Custody forms	X	X
12	Relevant correspondence	X	X
13	Field corrective action reports	X	X
14	Change orders and deviations	X	X
Analytical Data Package			
15	Cover sheet (laboratory identifying information)	X	X
16	Case narrative	X	X
17	Inter-laboratory custody documentation	X	X
18	Sample receipt records	X	X
19	Relevant correspondence record	X	X
20	LOD/LOQ verification	X	X
21	Reported DL, LOD, and LOQ	X	X

Item	Description	Verification	Validation
22	Standards traceability logs	X	X
23	Instrument calibration records	X	X
24	Sample preparation and analysis worksheets, logs, bench sheets	X	X
25	Sample chronology (dates and times of receipt, preparation, analysis)	X	X
26	Sample and quality control results forms	X	X
27	Chromatograms, mass spectra, and instrument printouts	X	X
28	Raw data not otherwise described	X	X
29	Electronic data deliverable	X	X
30	Laboratory corrective action reports	X	X

Notes:
DL – detection limit
DoD – department of defense
GC/MS – gas chromatography/mass spectrometry
LOD – limit of detection
LOQ – limit of quantitation
SOP – standard operating procedure

QAPP WORKSHEET #35: DATA VERIFICATION PROCEDURES

Verification Input	Documents Required for Review	Process Description	Responsible Party
Field logbooks and worksheets	UFP-QAPP, CUES SOP 18	Verify completion and legibility. Verify documents indicate locations of field duplicate samples and samples collected with non-dedicated equipment.	Field Manager (daily), Deputy Project Manager (conclusion of field activities)
Chain of custody forms	UFP-QAPP, CUES SOP 17	Verify completion and consistency with field logbooks and worksheets. Verify appropriate sample containers were used, correct analytical methods were selected, and sufficient sample volume for requested QC e.g., MS/MSDs. Verify required signatures and dates are present. Check for transcription errors.	Field Manager (daily), Deputy Project Manager (conclusion of field activities)
Laboratory analytical report	UFP-QAPP	Verify the report contains all elements specified in the UFP-QAPP. Verify submitted samples were received intact, properly preserved, and with sufficient volume to perform requested analyses. Verify the report contains results for all samples and analyses requested on the chain of custody form. Verify the correct target analyte lists are reported. Verify QC exceedances are documented in the case narrative. Verify that the report is finalized and signed by appropriate laboratory personnel.	Laboratory Quality Assurance Manager (before release), Project Chemist (upon release)
Laboratory EDD	UFP-QAPP	Verify the EDD format is consistent with ERPIMS, contains all reported results for all submitted samples.	Laboratory Quality Assurance Manager (before release), Project Chemist (upon release)
Corrective action reports	UFP-QAPP	Verify that corrective actions were implemented and documented according to UFP-QAPP requirements.	Project Quality Assurance Manager

Notes:
CUES – LATA and CTI Environmental Services, LLC
EDD – electronic data deliverable
ERPIMS – Environmental Resources Program Information Management System
MS/MSD – matrix spike and matrix spike duplicate
QC – quality control
SOP – standard operating procedure
UFP-QAPP – Uniform Federal Policy-Quality Assurance Project Plan

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QAPP WORKSHEET #36: DATA VALIDATION PROCEDURES

Field sample analytical data packages will be validated after SI fieldwork activities at 100 (%) Stage 2B, as defined in the United States Department of Defense (DoD) General Data Validation Guidelines (DoD, 2019b). Investigation Derived Waste analytical data will not be validated.

Data Validator: CTI & Associates, Inc.

Analytical Group:	Metals	VOCs	PAHs	VOCs in Soil Vapor
Analytical Method:	USEPA SW-846 6020B	USEPA SW-846 8260D	USEPA SW-846 8270 with Select Ion Monitoring	USEPA Compendium Method TO-15
Data Deliverable Requirements	Level IV Data Package with ERPIMS EDD	Level IV Data Package with ERPIMS EDD	Level IV Data Package with ERPIMS EDD	Level IV Data Package with ERPIMS EDD
Analytical Specifications	Worksheet #12a	Worksheet #12b	Worksheet #12c	Worksheet #12d
Percentage of data package validated:	100%	100%	100%	100%
Percentage of raw data reviewed:	0%	0%	0%	0%
Percentage recalculated:	0%	0%	0%	0%
Validation Code[2]	S2BVM 100%	S2BVM 100%	S2BVM 100%	S2BVM 100%

Notes:
[2] Validation codes are defined in the DoD General Data Validation Guidelines (DoD, 2019b)
% – percent
EDD – electronic data deliverable
ERPIMS – Environmental Resources Program Information Management System
PAH – polycyclic aromatic hydrocarbon
S2BVM – Manual Stage 2B Validation
USEPA – United States Environmental Protection Agency
VOCs –volatile organic compounds

The following qualifiers may be applied during data validation:

Qualifier	Definition
U	The analyte was not detected and is reported as less than the limit of detection.
UJ	The analyte was not detected and is reported as less than the limit of detection; however, the associated numerical value is approximate
J	The reported result is a quantitative estimate with potential imprecision and/or an unknown bias.
J+	The reported result is a quantitative estimate, and the result may be biased high.
J-	The reported result is a quantitative estimate, and the result may be biased low.
B	The result has been qualified due to suspected contamination in an associated blank.
Q	The result has been both J qualified as a quantitative estimate, and B qualified as being potentially impacted by contamination.
NJ	The analyte is a quantitative and qualitative estimate. Analyte identification is presumptive, rather than definitive.
X	The sample result is affected by a serious quality control deficiency, and the presence or absence of the target analyte cannot be substantiated. Exclusion of the data is recommended by the data validator.

Data validation will be performed in accordance with the United States Department of Defense (DoD) General Data Validation Guidelines (DoD, 2019b), DoD Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by [gas chromatography/mass spectrometry] GC/MS (DoD, 2020), the DoD Quality Systems Manual Version 5.3 (DoD, 2019a), and quality control (QC) criteria specified in this QAPP. The DoD has not released validation modules for metals by inductively-coupled plasma mass spectrometry or volatile organic compounds in soil vapor; for these methods, validation will rely on the DoD Quality Systems Manual, General Data Validation Guidelines, and this Uniform Federal Policy-Quality Assurance Project Plan (UFP-QAPP).

Stage 2B validation includes review of analytical results, field sample QC results, laboratory QC results, and instrument QC summary forms and preparation logs. Data are compared to criteria in UPF-QAPP Worksheets #12a through 12d, Worksheets #15a through 15f, and Worksheet #19 & 30. Instrument QC summary forms are reviewed against analytical instrument calibration elements listed in Worksheet #24.

Validation qualifiers will be assigned with descriptive reason codes, which will be defined within each validation report. Qualifiers and reason codes will be added to the Environmental Resources Program Information Management System (ERPIMS) electronic data deliverable before final data submission to the ERPIMS database in accordance with the schedule from Worksheet #14/16.

QAPP WORKSHEET #37: DATA USABILITY ASSESSMENT

After data validation is completed, sample results (including any qualifiers assigned during data validation) will be assessed for usability. The usability assessment requires participation from a broad project team, who evaluate whether data are of sufficient precision, accuracy, representativeness, completeness, comparability, and sensitivity to meet data quality objectives using the process outlined in this Worksheet.

Personnel listed below may participate in the data usability assessment:

Project Manager
Deputy Project Manager
Project Quality Assurance Manager
Project Engineer
Project Chemist
Field Manager

The assessment will be performed in the five-step process outlined below. Not all personnel will participate in every step. For example, the Field Manager may only be required to give input in Step 2, by describing site conditions preventing sample collection at a planned location.

Step 1	**Review the project's objectives and sampling design.** This step provides context for interpreting data in subsequent steps. Review project DQOs and MPCs (Worksheets #11 and #12a through 12d), for continued relevance and applicability to the project. Review the sampling design (Worksheets #17 and #18) for consistency with DQOs and MPCs.
Step 2	**Review data verification and validation outputs.** Review data verification findings to determine the extent of gaps in data collection and consider the implication of any incomplete components. Review the data validation report and data qualifiers to identify potentially impactful issues e.g., potential low analytical bias in results that will be used to demonstrate the concentration of lead in soil is below the Wisconsin Department of Natural Resources action limit listed in Worksheet #15a. The project team should consider the impacts of validation qualifiers within the context of the broader data set using graphs, charts, and maps as appropriate to determine whether the data are consistent with historical site usage and contemporaneous results from similar locations at the site.
Step 3	**Verify the assumptions of the conceptual site model.** The team should review assumptions from the site conceptual model, with a focus on statistical elements such as the distributional form of the data, independence of the data, dispersion characteristics, homogeneity, and assumed hydrogeological models.
Step 4	**Implement the statistical method.** This project involves hypothesis testing. The primary hypothesis is "concentrations of project COPCs are below the project action level (Worksheets #15a through #15f)." The team should consider the consequences of supporting or nullifying each hypothesis with data that have been qualified as quantitatively or qualitatively uncertain.

Step 5	**Document data usability and draw conclusions.** Determine if the data can be used as intended, considering the implications of verification and validation findings. Discuss data quality indicators and their implications for hypothesis testing. Identify limitations on data usage. Review the conceptual site model to determine whether it should be updated. Prepare the data usability summary, which can be in the form of text or a table and may be included as a section in the PA/SI Report.

Notes:
% – percent
COC – contaminant of concern
DQO – data quality objective
MCL – maximum contaminant level
MPC – measurement performance criteria
PAL – project action limit
PA/SI – Preliminary Assessment/Site Investigations

FIGURES

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LEGEND

FORMER GENERAL MITCHELL ARS PROPERTY LINE

NOTES:

1. THE FORMER GENERAL MITCHELL ARS PROPERTY LINE IS APPROXIMATE AND IS OBTAINED FROM HISTORICAL DOCUMENTATION.

N

0 1500 3000 Feet

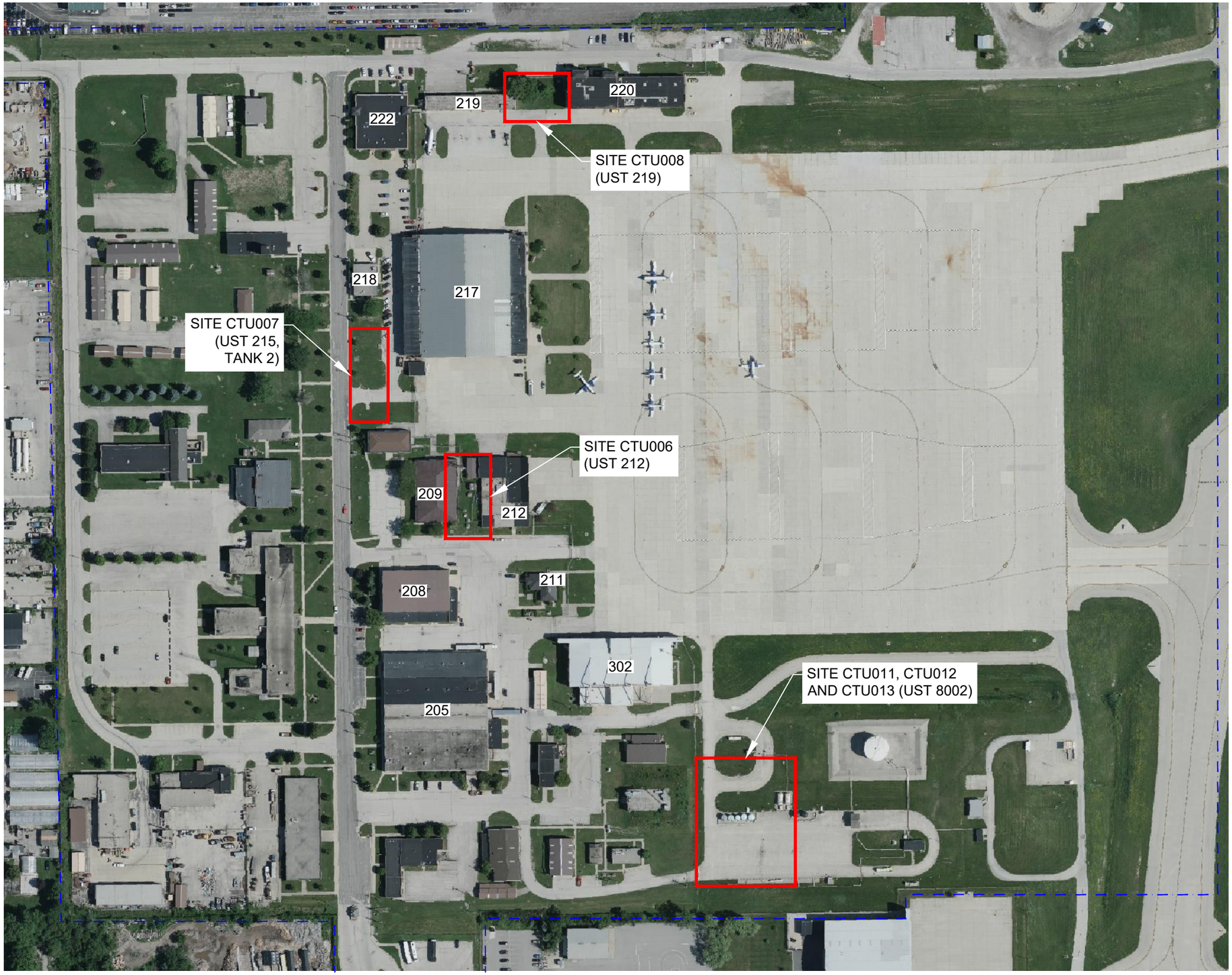
CTI-URS
Environmental Services, LLC

INSTALLATION LOCATION MAP

PRELIMINARY ASSESSMENT AND SITE INSPECTION
FORMER GENERAL MITCHELL AIR RESERVE STATION
MILWAUKEE, WISCONSIN

Drawn By:	Date:	
RAB	MARCH 2022	FIGURE 01
Reviewed By:	Project No.:	
JB	1215010024	

Z:\Shared\proj\Projects\Federal\AFCEC\FA8903-16-D-0053 - AE13ES\Task Orders\TO FA8903-21-F-1088 - Gen Mitchell PA-SI\7.0 Drawings&Specifications\900-CAD-GIS\910-CAD\15-SHEETS\Figures\UFP-QAPP Figures\Figure 1 - Installation Location Map.dwg User:rbrill Mar 08, 2022 - 5:52pm



LEGEND

- FORMER GENERAL MITCHELL ARS PROPERTY LINE
- SITE BOUNDARY
- 217 BUILDING IDENTIFIER

NOTES:

1. THE FORMER GENERAL MITCHELL ARS PROPERTY LINE IS APPROXIMATE AND IS OBTAINED FROM HISTORICAL DOCUMENTATION.

N

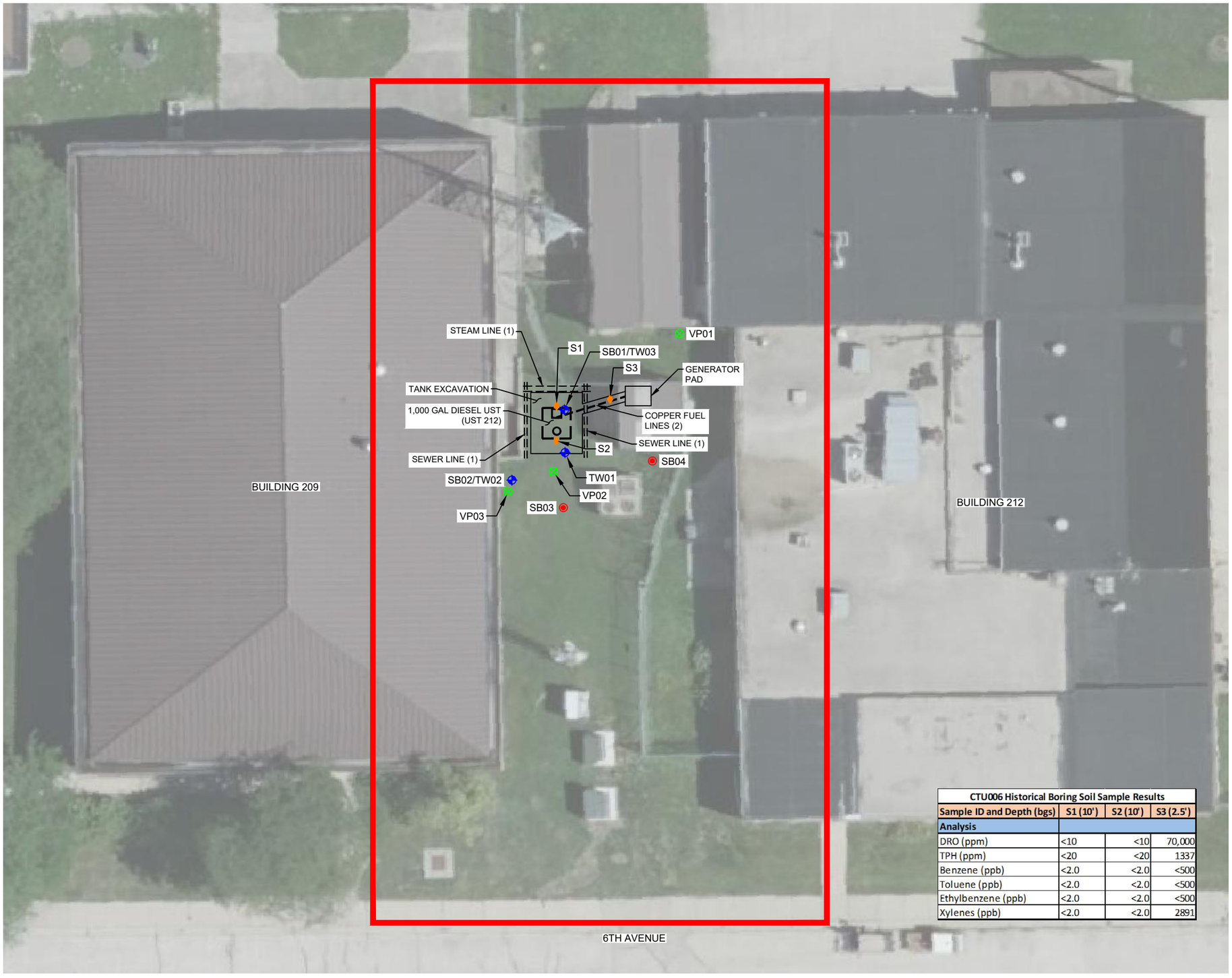
0 200 400 Feet

CTI-URS
Environmental Services, LLC

OVERALL SITE MAP

PRELIMINARY ASSESSMENT AND SITE INSPECTION
FORMER GENERAL MITCHELL AIR RESERVE STATION
MILWAUKEE, WISCONSIN

Drawn By:	Date:	
RAB	MARCH 2022	FIGURE 02
Reviewed By:	Project No.:	
JB	1215010024	



CTU006 Historical Boring Soil Sample Results			
Sample ID and Depth (bgs)	S1 (10')	S2 (10')	S3 (2.5')
Analysis			
DRO (ppm)	<10	<10	70,000
TPH (ppm)	<20	<20	1337
Benzene (ppb)	<2.0	<2.0	<500
Toluene (ppb)	<2.0	<2.0	<500
Ethylbenzene (ppb)	<2.0	<2.0	<500
Xylenes (ppb)	<2.0	<2.0	2891

LEGEND

- SOIL BORING/TEMPORARY MONITORING WELL
- SOIL BORING LOCATION
- HISTORICAL BORING LOCATION
- TEMPORARY VAPOR WELL
- APPROXIMATE REMOVED UST LOCATION
- APPROXIMATE SITE BOUNDARY

NOTES:

1. UST, ASSOCIATED PIPING, UTILITIES, HISTORICAL BORINGS, AND FORMER BUILDING LOCATIONS ARE APPROXIMATE AND BASED ON HISTORICAL DOCUMENTATION.

N

0 20 40 Feet

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SITE MAP - CTU006 (UST 212)

PRELIMINARY ASSESSMENT AND SITE INSPECTION
FORMER GENERAL MITCHELL AIR RESERVE STATION
MILWAUKEE, WISCONSIN

Drawn By:	Date:	
RAB	JUNE 2022	FIGURE 03
Reviewed By:	Project No.:	
JB	1215010024	

CTU007 Historical Boring Soil Sample Results

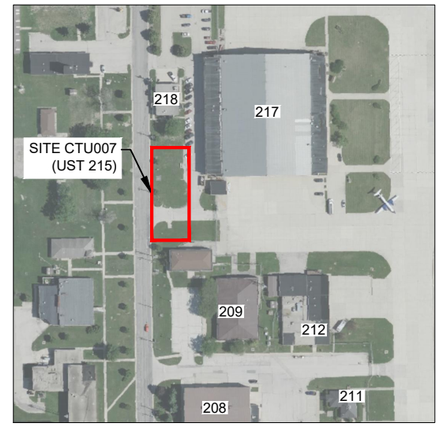
Sample ID and Depth (bgs)	3W (5')	3E (5')	3N (5')	1N (5')	1S (5')	1E (5')	1W (5')
Analysis							
DRO (ppm)	<4.2	4.6	<4.0	<4.2	<4.3	<4.2	<4.0
Benzene (ppb)	<25	<25	<25	<25	<25	<25	<25
Toluene (ppb)	<25	<25	<25	<25	<25	<25	<25
Ethylbenzene (ppb)	<25	<25	<25	<25	<25	<25	<25
Xylenes (ppb)	<25	<25	<25	<25	<25	<25	<25

LEGEND

- SOIL BORING/TEMPORARY MONITORING WELL
- SOIL BORING LOCATION
- HISTORICAL BORING LOCATION
- APPROXIMATE REMOVED UST LOCATION
- APPROXIMATE SITE BOUNDARY

NOTES:

1. UST, ASSOCIATED PIPING, HISTORICAL BORINGS, UTILITIES, AND FORMER BUILDING LOCATIONS ARE APPROXIMATE AND BASED ON HISTORICAL DOCUMENTATION.



N

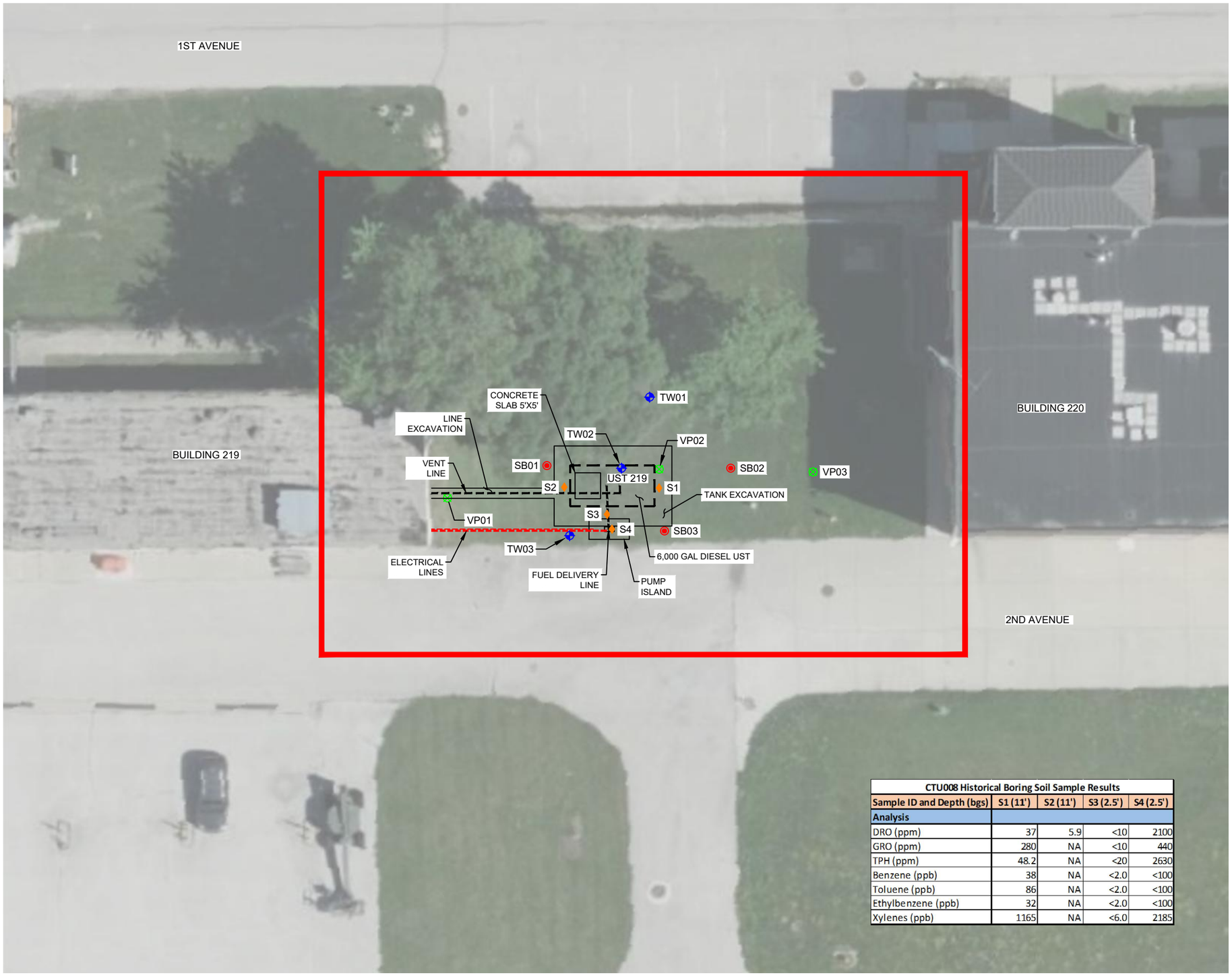
0 20 40 Feet

CTI-URS
Environmental Services, LLC

SITE MAP - CTU007 (UST 215)

PRELIMINARY ASSESSMENT AND SITE INSPECTION
FORMER GENERAL MITCHELL AIR RESERVE STATION
MILWAUKEE, WISCONSIN

Drawn By:	Date:	
RAB	JUNE 2022	FIGURE 04
Reviewed By:	Project No.:	
JB	1215010024	

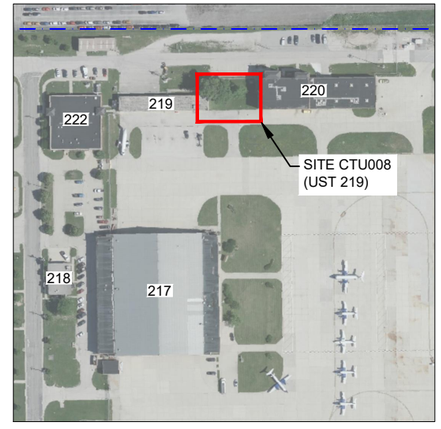


LEGEND

- SOIL BORING/TEMPORARY MONITORING WELL
- SOIL BORING LOCATION
- HISTORICAL BORING LOCATION
- TEMPORARY VAPOR WELL
- APPROXIMATE REMOVED UST LOCATION
- FORMER GENERAL MITCHELL ARS PROPERTY LINE
- APPROXIMATE SITE BOUNDARY

NOTES:

1. UST, ASSOCIATED PIPING, UTILITIES, HISTORICAL BORINGS, AND FORMER BUILDING LOCATIONS ARE APPROXIMATE AND BASED ON HISTORICAL DOCUMENTATION.



N

0 20 40 Feet

CTU008 Historical Boring Soil Sample Results

Sample ID and Depth (bgs)	S1 (11')	S2 (11')	S3 (2.5')	S4 (2.5')
Analysis				
DRO (ppm)	37	5.9	<10	2100
GRO (ppm)	280	NA	<10	440
TPH (ppm)	48.2	NA	<20	2630
Benzene (ppb)	38	NA	<2.0	<100
Toluene (ppb)	86	NA	<2.0	<100
Ethylbenzene (ppb)	32	NA	<2.0	<100
Xylenes (ppb)	1165	NA	<6.0	2185

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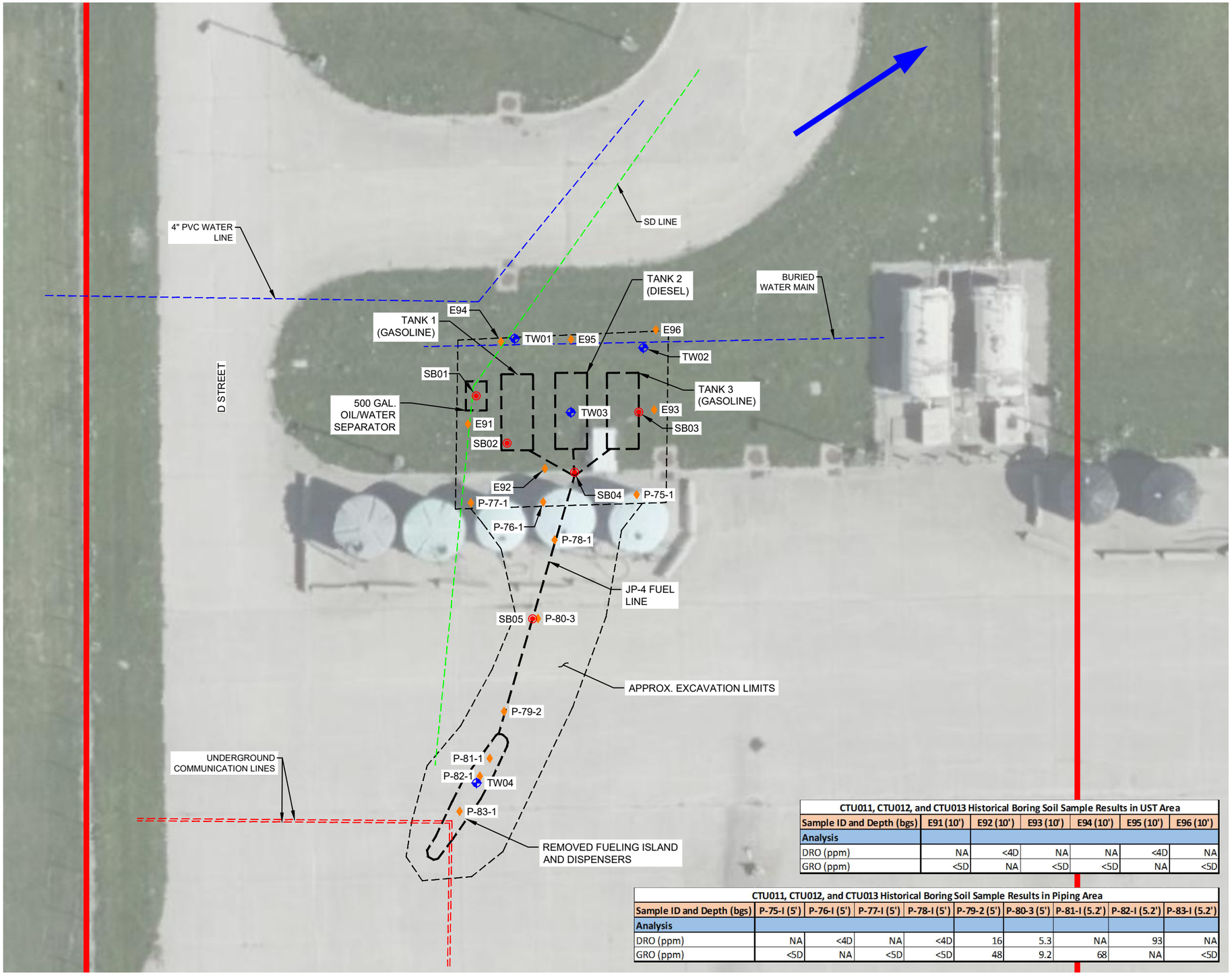
SITE MAP - CTU008 (UST 219)

PRELIMINARY ASSESSMENT AND SITE INSPECTION
FORMER GENERAL MITCHELL AIR RESERVE STATION
MILWAUKEE, WISCONSIN

Drawn By:	Date:	
RAB	JUNE 2022	FIGURE 05
Reviewed By:	Project No.:	
JB	1215010024	

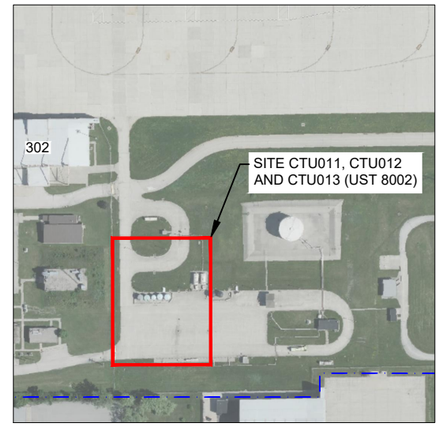


LEGEND

- SOIL BORING/TEMPORARY MONITORING WELL
- SOIL BORING LOCATION
- HISTORICAL BORING LOCATION
- APPROXIMATE REMOVED UST AND PIPING LOCATIONS
- FORMER GENERAL MITCHELL ARS PROPERTY LINE
- APPROXIMATE SITE BOUNDARY
- ESTIMATED GROUNDWATER FLOW DIRECTION

NOTES:

1. UST, ASSOCIATED PIPING, HISTORICAL BORINGS, AND UTILITIES ARE APPROXIMATE AND BASED ON HISTORICAL DOCUMENTATION.



N

0 20 40 Feet

CTU011, CTU012, and CTU013 Historical Boring Soil Sample Results in UST Area

Sample ID and Depth (bgs)	E91 (10')	E92 (10')	E93 (10')	E94 (10')	E95 (10')	E96 (10')
Analysis						
DRO (ppm)	NA	<4D	NA	NA	<4D	NA
GRO (ppm)	<5D	NA	<5D	<5D	NA	<5D

CTU011, CTU012, and CTU013 Historical Boring Soil Sample Results in Piping Area

Sample ID and Depth (bgs)	P-75-I (5')	P-76-I (5')	P-77-I (5')	P-78-I (5')	P-79-2 (5')	P-80-3 (5')	P-81-I (5.2')	P-82-I (5.2')	P-83-I (5.2')
Analysis									
DRO (ppm)	NA	<4D	NA	<4D	16	5.3	NA	93	NA
GRO (ppm)	<5D	NA	<5D	<5D	48	9.2	68	NA	<5D

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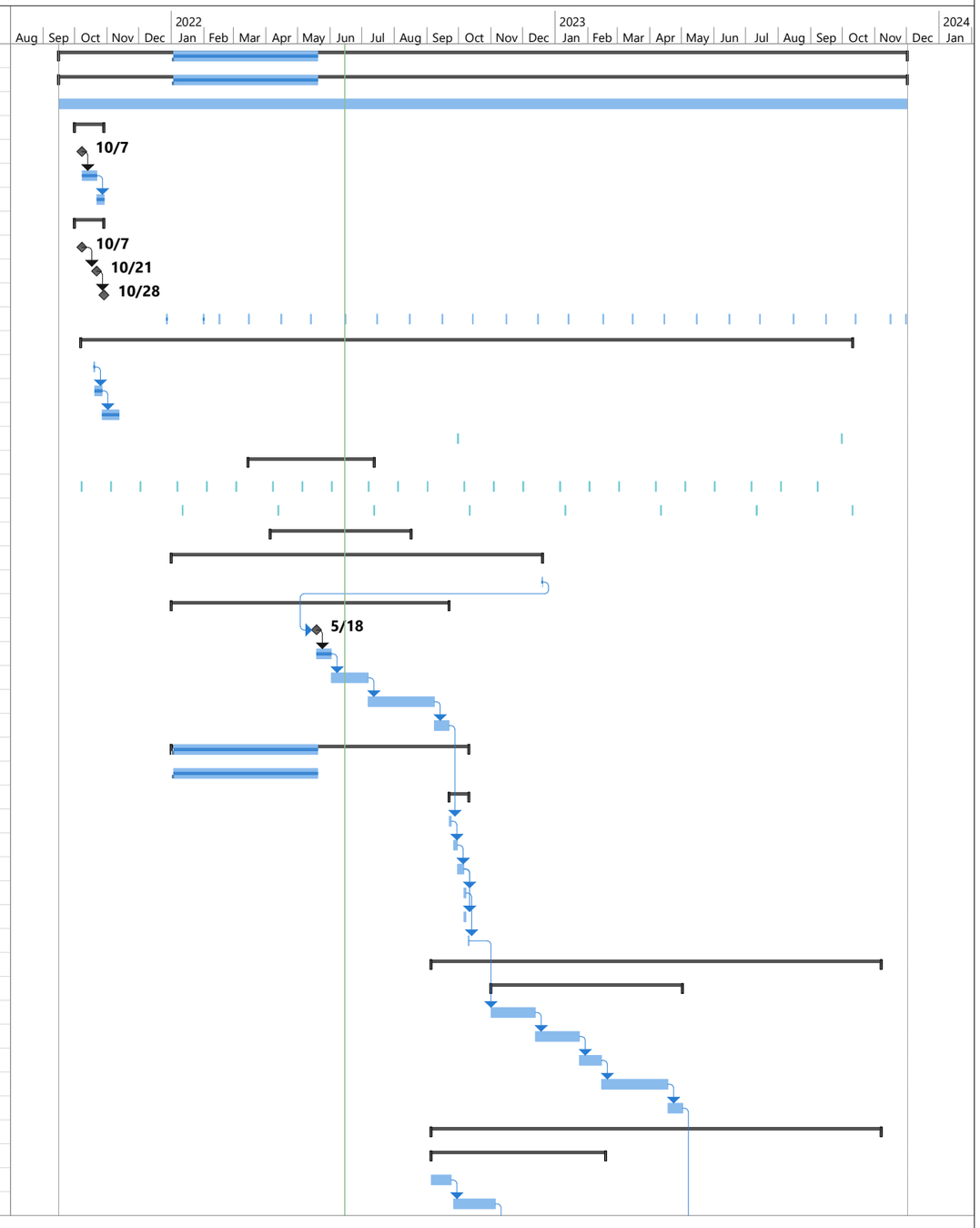
SITE MAP - CTU011, CTU012 AND CTU013 (UST 8002)

PRELIMINARY ASSESSMENT AND SITE INSPECTION
FORMER GENERAL MITCHELL AIR RESERVE STATION
MILWAUKEE, WISCONSIN

Drawn By:	Date:	
RAB	JUNE 2022	FIGURE 06
Reviewed By:	Project No.:	
JB	1215010024	

Figure 7 - General Mitchell UST Site Inspections and Closure Reports

ID	Task Mode	Task Name	Duration	Start	Finish	% Complete
1		**Task Order Award**	**577 days**	**9/16/21**	**12/1/23**	**8%**
2		**Task 1: Project Management**	**577 days**	**9/16/21**	**12/1/23**	**8%**
3		Task 1A: Project Website	577 days	9/16/21	12/1/23	0%
4		**Task 1B: Workbreakdown Structure**	**20 days**	**10/1/21**	**10/28/21**	**100%**
5		Prepare and Submit Draft Workbreakdown Structure (WBS)	0 days	10/1/21	10/7/21	100%
6		AFCEC Review of Draft WBS	10 days	10/8/21	10/21/21	100%
7		Prepare and Submit Final WBS	5 days	10/22/21	10/28/21	100%
8		**Task 1C: Project Planning Chart**	**20 days**	**10/1/21**	**10/28/21**	**100%**
9		Prepare and Submit Draft Project Planning Chart (PPC)	0 days	10/1/21	10/7/21	100%
10		AFCEC Review of Draft PPC	0 days	10/21/21	10/21/21	100%
11		Prepare and Submit Final PPC	0 days	10/28/21	10/28/21	100%
12		**Task 1D: Contractor's Progress, Status, and Management Report (CPSMR)**	**504 days**	**12/27/21**	**11/30/23**	**8%**
38		**Task 1E: Meetings**	**524 days**	**10/7/21**	**10/10/23**	**32%**
39		Task Order Kickoff Meeting	1 day	10/19/21	10/19/21	100%
40		Draft Meeting Minutes	5 days	10/20/21	10/26/21	100%
41		Final Meeting Minutes	12 days	10/27/21	11/11/21	100%
42		**Project Management Review (PMR) Meetings**	**261 days**	**9/30/22**	**9/30/23**	**0%**
45		**On-site Technical Review Meetings**	**86 days**	**3/15/22**	**7/12/22**	**0%**
48		**Technical Teleconferences**	**501 days**	**10/7/21**	**9/7/23**	**0%**
73		**BCT Teleconferences**	**456 days**	**1/11/22**	**10/10/23**	**0%**
82		**On-Site BCT Meetings**	**96 days**	**4/5/22**	**8/16/22**	**0%**
85		**Task 2: Task Order Planning**	**251 days**	**1/1/22**	**12/19/22**	**12%**
86		Task 2A: Scoping Planning Visit	1 day	12/17/22	12/19/22	100%
87		**Task 2B: Quality Program Plan (QPP)**	**188 days**	**1/1/22**	**9/21/22**	**11%**
88		Prepare and Submit Draft QPP (includes WP, FSP, SAP, and HSP)	0 days	1/1/22	5/18/22	100%
89		AFCEC Review of Draft QPP	10 days	5/18/22	6/1/22	100%
90		Prepare and Submit Draft-Final QPP, including AFCEC backcheck	25 days	6/1/22	7/6/22	0%
91		Wisconsin DNR Review of Draft-Final QPP	45 days	7/7/22	9/7/22	0%
92		Prepare and Submit Final QPP, including AFCEC backcheck	10 days	9/8/22	9/21/22	0%
93		**Task 3: Preliminary Assessment/Site Inspection**	**201 days**	**1/1/22**	**10/10/22**	**87%**
94		Task 3A: Preliminary Assessment	99 days	1/1/22	5/19/22	100%
95		**Task 3B: Site Inspection**	**13 days**	**9/22/22**	**10/10/22**	**0%**
96		Mobilization	2 days	9/22/22	9/23/22	0%
97		Soil Sampling	4 days	9/26/22	9/29/22	0%
98		Monitoring Well Installation	4 days	9/30/22	10/5/22	0%
99		Monitoring Well Development and Sampling	2 days	10/6/22	10/7/22	0%
100		VI Sampling	2 days	10/6/22	10/7/22	0%
101		Demobilization	1 day	10/10/22	10/10/22	0%
102		**Task 4: Reporting**	**306 days**	**9/5/22**	**11/6/23**	**0%**
103		**Task 4A: PA/SI Report**	**130 days**	**11/1/22**	**5/1/23**	**0%**
104		Prepare and Submit Draft PA/SI Report	30 days	11/1/22	12/12/22	0%
105		AFCEC Review of Draft PA/SI Report	30 days	12/13/22	1/23/23	0%
106		Prepare and Submit Draft-Final PA/SI Report, including AFCEC backcheck	15 days	1/24/23	2/13/23	0%
107		Wisconsin DNR Review of Draft-Final PA/SI Report	45 days	2/14/23	4/17/23	0%
108		Prepare and Submit Final PA/SI Report, including AFCEC backcheck	10 days	4/18/23	5/1/23	0%
109		**Task 4B: Site Closure Letters**	**306 days**	**9/5/22**	**11/6/23**	**0%**
110		**CTU001 (OWS 104)**	**120 days**	**9/5/22**	**2/17/23**	**0%**
111		Prepare and Submit Draft Site Closure Letter	15 days	9/5/22	9/23/22	0%
112		AFCEC Review of Draft Site Closure Letter	30 days	9/26/22	11/4/22	0%

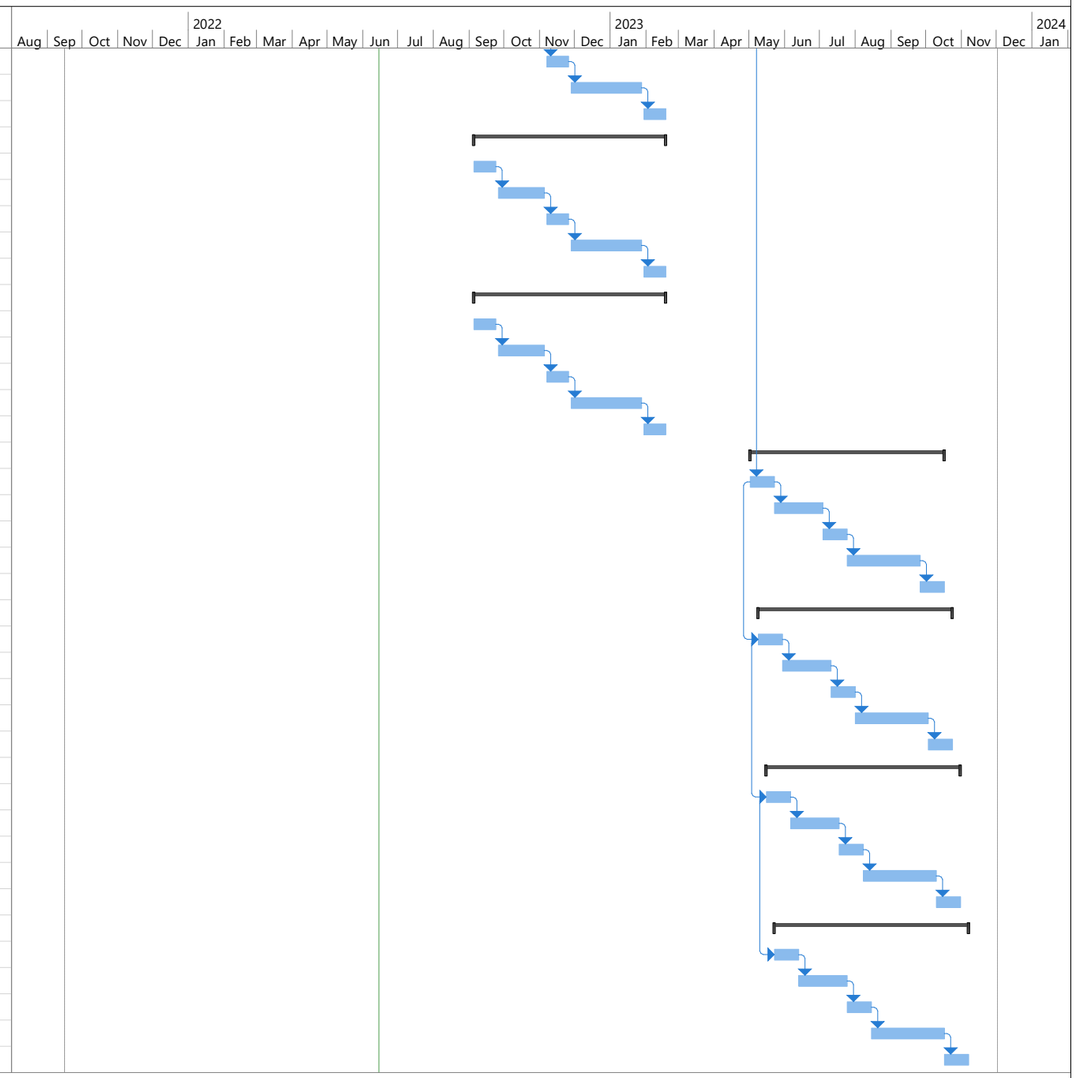


Project: UST PA/SI
Date: 6/15/22

Task | Split | Milestone | Summary | Project Summary | Inactive Task | Inactive Milestone | Inactive Summary | Manual Task | Duration-only | Manual Summary Rollup | Manual Summary | Start-only | Finish-only | External Tasks | External Milestone | Deadline | Progress | Manual Progress

Figure 7 - General Mitchell UST Site Inspections and Closure Reports

ID	Task Mode	Task Name	Duration	Start	Finish	% Complete
113		Prepare and Submit Draft-Final Site Closure Letter, including AFCEC backcheck	15 days	11/7/22	11/25/22	0%
114		Wisconsin DNR Review of Draft-Final Site Closure Letter	45 days	11/28/22	1/27/23	0%
115		Prepare and Submit Final Site Closure Letter, including AFCEC backcheck	15 days	1/30/23	2/17/23	0%
116		**CTU014 (OWS 308)**	**120 days**	**9/5/22**	**2/17/23**	**0%**
117		Prepare and Submit Draft Site Closure Letter	15 days	9/5/22	9/23/22	0%
118		AFCEC Review of Draft Site Closure Letter	30 days	9/26/22	11/4/22	0%
119		Prepare and Submit Draft-Final Site Closure Letter, including AFCEC backcheck	15 days	11/7/22	11/25/22	0%
120		Wisconsin DNR Review of Draft-Final Site Closure Letter	45 days	11/28/22	1/27/23	0%
121		Prepare and Submit Final Site Closure Letter, including AFCEC backcheck	15 days	1/30/23	2/17/23	0%
122		**CTU015 (West Ditch)**	**120 days**	**9/5/22**	**2/17/23**	**0%**
123		Prepare and Submit Draft Site Closure Letter	15 days	9/5/22	9/23/22	0%
124		AFCEC Review of Draft Site Closure Letter	30 days	9/26/22	11/4/22	0%
125		Prepare and Submit Draft-Final Site Closure Letter, including AFCEC backcheck	15 days	11/7/22	11/25/22	0%
126		Wisconsin DNR Review of Draft-Final Site Closure Letter	45 days	11/28/22	1/27/23	0%
127		Prepare and Submit Final Site Closure Letter, including AFCEC backcheck	15 days	1/30/23	2/17/23	0%
128		**CTU006 (UST 212)**	**120 days**	**5/2/23**	**10/16/23**	**0%**
129		Prepare and Submit Draft Site Closure Letter	15 days	5/2/23	5/22/23	0%
130		AFCEC Review of Draft Site Closure Letter	30 days	5/23/23	7/3/23	0%
131		Prepare and Submit Draft-Final Site Closure Letter, including AFCEC backcheck	15 days	7/4/23	7/24/23	0%
132		Wisconsin DNR Review of Draft-Final Site Closure Letter	45 days	7/25/23	9/25/23	0%
133		Prepare and Submit Final Site Closure Letter, including AFCEC backcheck	15 days	9/26/23	10/16/23	0%
134		**CTU007 (UST 215 [Tank 2])**	**120 days**	**5/9/23**	**10/23/23**	**0%**
135		Prepare and Submit Draft Site Closure Letter	15 days	5/9/23	5/29/23	0%
136		AFCEC Review of Draft Site Closure Letter	30 days	5/30/23	7/10/23	0%
137		Prepare and Submit Draft-Final Site Closure Letter, including AFCEC backcheck	15 days	7/11/23	7/31/23	0%
138		Wisconsin DNR Review of Draft-Final Site Closure Letter	45 days	8/1/23	10/2/23	0%
139		Prepare and Submit Final Site Closure Letter, including AFCEC backcheck	15 days	10/3/23	10/23/23	0%
140		**CTU008 (UST 219)**	**120 days**	**5/16/23**	**10/30/23**	**0%**
141		Prepare and Submit Draft Site Closure Letter	15 days	5/16/23	6/5/23	0%
142		AFCEC Review of Draft Site Closure Letter	30 days	6/6/23	7/17/23	0%
143		Prepare and Submit Draft-Final Site Closure Letter, including AFCEC backcheck	15 days	7/18/23	8/7/23	0%
144		Wisconsin DNR Review of Draft-Final Site Closure Letter	45 days	8/8/23	10/9/23	0%
145		Prepare and Submit Final Site Closure Letter, including AFCEC backcheck	15 days	10/10/23	10/30/23	0%
146		**CTU011, CTU012, and CTU013 (UST 8002)**	**120 days**	**5/23/23**	**11/6/23**	**0%**
147		Prepare and Submit Draft Site Closure Letter	15 days	5/23/23	6/12/23	0%
148		AFCEC Review of Draft Site Closure Letter	30 days	6/13/23	7/24/23	0%
149		Prepare and Submit Draft-Final Site Closure Letter, including AFCEC backcheck	15 days	7/25/23	8/14/23	0%
150		Wisconsin DNR Review of Draft-Final Site Closure Letter	45 days	8/15/23	10/16/23	0%
151		Prepare and Submit Final Site Closure Letter, including AFCEC backcheck	15 days	10/17/23	11/6/23	0%



Project: UST PA/SI
Date: 6/15/22

Task
Split
Milestone
Summary
Project Summary
Inactive Task
Inactive Milestone
Inactive Summary
Manual Task
Duration-only
Manual Summary Rollup
Manual Summary
Start-only
Finish-only
External Tasks
External Milestone
Deadline
Progress
Manual Progress

APPENDIX A
FIELD FORMS

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GROUNDWATER LEVEL MEASUREMENT SHEET

Installation/Facility: _____ **Site ID(s):** _____

Location: _____ **Personnel:** _____

Weather Conditions: _____ **Measuring Device:** Water Level Indicator / Inter-phase probe

Remarks: _____

Well Number	Date	Time	Total Well Depth (feet)*	Water Level Indicator Reading (feet)*	Free Product (feet)*	Comments (condition of well)

* All measurements to the nearest 0.01 foot

GROUNDWATER SAMPLING RECORD

Project Name: Project No.

Installation:		Initial Depth to Water:	
Well ID:		Depth to Water After Sampling:	
Sample ID:		Total Depth of Well:	
Duplicate ID:		Well Diameter (inches):	
Sample Depth:		1 Casing Volume (gal):	
Date:		3 Casing Volumes (gal):	
Sample Collection Time:		Total Volumes Removed:	
Sample Container Type:		Sample Technician:	
Preservative:		Method of Purging:	
Analysis/Method:		Pump Intake Depth (feet):	
Method of Sampling:		Measuring Point (toc, tor, etc.):	

Time	Water Level (feet)	Flow Rate (ml/min)	Cum. Vol. (gal.)	Temp. (°C)	pH (units)	Specific Electrical Conductance (mS/cm)	DO (mg/L)	ORP (mV)	Turbidity (NTU)	Comments (color, sediment, etc.)
Stabilization Criteria				±0.5°C	±0.1	±3%	±10%	±10%	±10% and <10 NTU	

Instruments (Manufacturer, Model, and Serial No.):

Calculations:
1" diameter = 0.041 gal/ft
2" diameter = 0.163 gal/ft
4" diameter = 0.653 gal/ft
6" diameter = 1.47 gal/ft

Notes:

Sample Technician Name(s) and Signature(s):

QA/QC'd by: **Date:** **Page 1 of** ___

GROUNDWATER SAMPLING RECORD (CONTINUED)

Project Name: Project No.

Installation: Well ID:

Time	Water Level (feet)	Flow Rate (ml/min)	Cum. Vol. (gal.)	Temp. (°C)	pH (units)	Specific Electrical Conductance (mS/cm)	DO (mg/L)	ORP (mV)	Turbidity (NTU)	Comments (color, sediment, etc.)

Notes:

Sample Technician Name(s) and Signature(s):

QA/QC'd by: **Date:** **Page ___ of ___**

		EQUIPMENT CALIBRATION WORKSHEET		Dates:	
		Project Name:		Weather:	
Instrument I. D.		Project Number:		Scientist/ Engineer:	

Multi - Parameter Instrument Calibration

Date / Time	pH			ORP	Specific Conductivity	DO		Barometric Pressure	Comments
	4	7	10	mV	(µs/cm²)	100%	mg/L	mmHg	
Lot No.									

Geotech Turbidimeter Calibration (If applicable)

Date	<0.1	15	100	750

DAILY QUALITY CONTROL REPORT

Site:	**Project Manager:**	**Quality Control:**	**Page No.: ____ of ____**

Date:	**Week No.:**	**Hours on Site:** **Hours Off Site:**	**Delivery Order:**

Written By:	**Reviewed By:**
Weather/Temperature:	
Location of Work:	

Project Personnel:	**Equipment:**	**Visitors/Affiliation:**
• **Field Team Leader:**		
• **CQC Manager:**		
• **SSHO:**		
• **Others:**		

Work Performed by CTI:

Work to be Performed Tomorrow:

Safety Observations/Violations/Comments:

Calibration of Field Equipment (See Calibration Logs in File):

Certification:

I certify that the above report is complete and correct and that I, or my authorized representative, have inspected all work performed this day and have determined that all materials, equipment, and workmanship are in strict compliance with the plans and specification, except as may be noted above.

Signature: _____

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APPENDIX B

STANDARD OPERATING PROCEDURES

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EQUIPMENT CALIBRATION AND MAINTENANCE STANDARD OPERATING PROCEDURE

1.0 Purpose

This Standard Operating Procedure (SOP) establishes the methods and responsibilities associated with the calibration, control, and maintenance of field measurement and test equipment. It applies to all tools, gauges, instruments, and other test equipment where the manufacturer requires or recommends equipment accuracy to be checked periodically. In the case of commercial devices such as rulers, tape measures, and levels calibration controls are not required.

2.0 References

2.1 None

3.0 Responsibilities

3.1 The *Field Task Manager* or his/her designee is responsible for monitoring the effective implementation of this SOP and/or the equipment manufacturer's recommendations.

3.2 The *Project Manager* and *Field Task Manager* are responsible for the selection of equipment to be used in the field activity and to assure it is of the proper type, range, accuracy and tolerance required to meet project objectives. Additionally, the *Field Task Manager* is responsible for storage and protection of equipment.

3.3 The *Field Technician* performing tests are responsible for assuring that all equipment is properly calibrated prior to and during use, and for documenting the calibration or deficiencies of equipment.

4.0 Definitions

4.1 Calibration

The comparison of a measurement standard or instrument of a known accuracy with another standard or instrument to detect, correlate, report, or eliminate by adjustment, any variation in the accuracy of the items being compared within allowable deviations.

4.2 Reference Standard

An item of known and verifiable value which is used to check or establish the basis for tests or inspections.

5.0 Procedure

5.1 Equipment Identification and Control

Equipment that requires calibration will be uniquely identified by the manufacturer's serial number, or other suitable assigned number. If this should prove to be impractical, an identification label will be affixed using materials and methods which provide a clear

and legible identification and do not detrimentally affect the function or service life of the equipment. This identification will be replaced as needed to provide clear identification of the equipment.

5.2 Calibration

All equipment and reference standards should be stored between uses in a manner that will minimize damage or deterioration (e.g., in a clean location, away from sunlight and temperature extremes to the extent possible).

Written procedures will be used for calibration of equipment. Calibration procedures that have been previously established and approved by the equipment manufacturer or a nationally recognized authority (i.e., the American Society for Testing and Materials, U.S. Environmental Protection Agency) will be used when available. If no preexisting procedure is available, procedures will be developed by qualified personnel familiar with the equipment and approved by the *Project Manager*. Development of procedures will take into consideration the intended use and objective of the resulting data, equipment characteristics, required accuracy and precision of data, location of field testing, and effects of climate or any other parameter which would adversely influence the calibration. The procedures will include, as applicable:

- Name/type of equipment to be calibrated;
- Reference standards to be used;
- Calibration method and sequential actions;
- Acceptance criteria;
- Frequency of calibrations/checks;
- Data recording form/format
- Data processing methodology;
- Any special instructions; and
- Operator training and qualification requirements.

Field equipment will be calibrated prior to use. Calibrations of equipment will be performed by trained and qualified personnel, approved external agencies or by the equipment manufacturer. The following types of calibrations and checks will be performed by qualified personnel:

Periodic calibrations are performed at prescribed intervals established for the equipment to assure that the equipment is operating within its designed range and accuracy. These are usually performed by outside agencies or the equipment manufacturer.

Specific calibrations are performed for specific measurements or tests and vary from instrument to instrument. Specific calibrations are performed prior to start of each work shift.

5.3 Calibration Frequency

Specific calibration of photoionization detectors (PIDs) will occur at the beginning of each field workday in which the equipment is to be used. The frequency of periodic

calibrations will be based on manufacturer's recommendations, national standards of practice, equipment type and characteristics, and past experience, and will be carried out by the equipment rental company (i.e., typically not in the field).

Scheduled calibrations of equipment do not relieve the user of the responsibility for selecting the appropriate and properly functioning equipment. In the event that the calibration has expired, the equipment will be removed from service and tagged as "out-of-service" to prevent inadvertent use until it has been appropriately recalibrated.

5.4 Reference Standards and Equipment

Calibration reference standards and equipment will have known relationships to the National Institute of Standards and Technology or other nationally recognized standards. If a national standard does not exist, the basis for calibration will be fully documented by the *Project Manager* and approved by the client's *Project Manager*.

Physical and chemical standards will have certifications traceable to NIST, EPA or other recognized agencies. It is the responsibility of the user to select, verify and use the correct standard in accordance with an approved procedure or established practice.

5.5 Calibration Failure

Each individual user of equipment is responsible for checking the calibration status of equipment to be used and confirming the acceptable calibration status prior to use. Equipment for which the periodic calibration period has expired, equipment that fails calibration, or equipment that becomes inoperable during use will be removed from service and tagged as out-of-service.

Out-of-service equipment will be repaired and/or recalibrated by the appropriate vendor or manufacturer. Any equipment consistently found to be out-of-calibration will be replaced.

Results of activities performed using equipment that has failed recalibration will be evaluated by the *Project Manager*. If the activity results are adversely affected, the results of the evaluation will be documented as a nonconformance for the client.

5.6 Calibration Documentation

Specific calibration records will be prepared and documented for each piece of calibrated equipment used. Calibration records will be maintained and available for review with the *Field Task Manager*. Calibration data will be recorded on a Calibration Log Form or in the bound field logbook. The *Field Task Manager* will be responsible for reviewing the calibration data for appropriateness, accuracy, readability, and completeness.

Calibration records will include, as applicable, the following information:

- Name of individual performing calibration;
- Equipment identification number and type (manufacturer and model if applicable);
- Date/time of calibration;

- Identification of reference standard(s) used (e.g., type, manufacturer, lot number);
- Pre-calibration readings if applicable; and
- Post-calibration readings or calibration result (pass/fail).

5.7 Preventive Maintenance

Preventive maintenance of equipment will be performed by the equipment rental company in accordance with manufacturer's recommendation to maintain proper equipment performance, minimize equipment failure and to increase measurement reliability.

6.0 Required Forms

Equipment Calibration Log Forms
Bound Field Logbook

SOP NO. 3
REVISION NO. 0
DATE: 2/23/21

SOIL SAMPLING
STANDARD OPERATING PROCEDURE

1.0 Purpose

This Standard Operating Procedure (SOP) establishes guidelines and procedures for use by field personnel in the collection and documentation of surface and subsurface soil samples for chemical analysis. Proper collection procedures are necessary to assure the quality and integrity of all surface and subsurface soil samples. Additional specific procedures and requirements will be provided in the project specific work plan.

2.0 References

ASTM D4700-15, Standard Guide for Soil Sampling from the Vadose Zone, ASTM International, West Conshohocken, PA, 2015, www.astm.org

ASTM D6169 / D6169M-13, Standard Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations, ASTM International, West Conshohocken, PA, 2013, www.astm.org

ASTM D6282 / D6282M-14, Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations, ASTM International, West Conshohocken, PA, 2014, www.astm.org

3.0 Responsibilities

3.1 The *Field Task Manager* is responsible for ensuring that all sample collection activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

3.2 The *Project Chemist* is responsible for periodic review of field generated documentation associated with this SOP. The *Project Chemist* is also responsible for implementation of corrective action (i.e., retraining personnel, additional review of specific work plan and SOPs, variances to QC sampling requirements, issuing nonconformances, etc.) if problems occur.

3.3 The *Field Technician is* responsible for completing their tasks according to specifications outlined in this SOP and other appropriate procedures. All staff are responsible for reporting deviations from procedures to the *Field Task Manager* or *Project Chemist* as appropriate.

4.0 Definitions/Materials

4.1 Direct Push Technology (DPT)

A direct push machine "pushes" tools and sensors into the ground without the use of drilling to remove soil to make a path for the tool.

4.2 Surface Soil Sample
Soil collected from the surface to a depth of 1 foot.

4.3 Subsurface Soil Sample
Soil collected at any depth interval greater than 1 foot.

4.4 Disturbed Soil Sample
A soil sample whose in situ physical structure and fabric has been disturbed as the direct result of sample collection.

4.5 Undisturbed Soil Sample
A soil sample where in situ physical structure and fabric has not been disturbed as the result of sample collection.

4.6 Grab Samples
Representative disturbed soil sample that is collected by using such devices as a shovel, stainless steel spoon, etc.

5.0 Procedure
This section contains both the responsibilities and procedures involved with surface and subsurface soil sampling. Proper surface and subsurface soil sampling procedures are necessary to insure the quality and integrity of the samples. The details within this SOP should be used in conjunction with project specific work plan. The project specific work plan will generally provide the following information:

- Sample collection objectives;
- Locations and depths of soil samples to be collected;
- Numbers and volumes of soil samples to be collected;
- Types of analyses to be conducted for the samples;
- Specific quality control (QC) procedures and sampling required;
- Any additional surface or subsurface soil sampling requirements or procedures beyond those covered in this SOP, as necessary.

At a minimum, the procedures outlined below for surface and subsurface soil sampling will be followed.

5.1 Surface Soil Sampling Equipment
A number of devices are available for the collection of surface soil samples. These include, but are not limited to: core samplers, hand augers, spoons, scoops, trowels, shovels, etc. These devices are constructed of a number of materials including, but not limited to, stainless steel, brass, glass, teflon, etc. The sampling and analytical requirements, as well as site characteristics, must be taken into account when determining the proper surface soil sampling equipment to use.

The chosen method for the collection of surface samples and subsurface samples, is DPT. A direct push machine "pushes" rods into the ground without the use of drilling to remove soil to make a path for the tool. The direct push machines rely on a relatively small amount of static (vehicle) weight combined with percussion as the energy for advancing a tool string (rods and sampler). Direct push tools do not remove cuttings from the probe hole but depend on compression of soil or rearrangement of soil particles to permit advancement for the tool string. Direct push tools are advanced as far as possible using only the static weight of the carrier vehicle. Percussion is applied as required when probing through sands, gravels, hard pans, high friction clays, tills, fill materials, and surface frost.

The sample containers to be used will be specified in the project specific work plan.

5.2 Surface Soil Sample Collection

5.2.1 Prior to sampling and between sampling locations, decontaminate the sample equipment according to SOP 15 and procedures outlined in the project specific work plan.

5.2.2 Ensure that all surface and subsurface soil sampling locations have been appropriately cleared of all underground utilities and buried objects per the project specific work plan. Review all forms and diagrams documenting the location of the cleared sampling locations, as well as that of any underground utilities or lines, or other buried objects.

5.2.3 As required, calibrate any health and safety monitoring equipment according to the instrument manufacturer's specifications. Calibration results will be recorded on the appropriate form(s), as specified in the project specific work plan. Instruments that cannot be calibrated according to the manufacturer's specifications will be removed from service and tagged.

5.2.4 Don appropriate personal protection equipment as specified in the project Health and Safety Plans.

5.2.5 Clear the area to be sampled of surface debris and vegetation using equipment that will not be used for sample collection.

5.2.6 If using the solid barrel, piston-sealed, direct push device for collecting discrete interval samples of unconsolidated materials at depth (sampler is approximately 30 inches long and has a 1.5-inch outside diameter), the sampler is capable of recovering a discrete sample core 22 inches long by 1.0 inch diameter contained inside a removable liner. Sample volume measures up to 283 mL.

The liner is a removable / replaceable thin-walled tube that fits inside the sample tube. Liners facilitate retrieval of the sample and may be used for storage when applicable.

5.2.7 Retrieve the device; check to see that soil recovery is adequate in the sampler. If there is sufficient recovery, mark or note the leading end of the sampler.

5.2.8 If using a different sample collection device, use the other device to scoop or collect soil and directly transfer the soil into the sample container (e.g., glass jar, brass sample sleeve, etc.). Fill the sample container such that little to no head-space exists.

5.2.9 If using sample sleeves, place teflon squares over each end of the sleeve and seal each end with plastic end caps. With a permanent marker, write a "T" for top on the trailing end and a "B" for bottom on the leading end. Place custody tape over each end cap so that any attempt to remove the cap will cause the tape to be broken. When VOCs are collected, seal over base of cap with Teflon tape. If using glass jars, cap or seal the jars appropriately and place custody tape over the cap so that any attempt to remove the cap will cause the tape to be broken.

5.2.10 Appropriately label and number the sample containers per the project specific work plan. The label will be filled out with waterproof ink and will contain, at a minimum, the following information:

- Project number;
- Sample number;
- Sample location;
- Sample depth;
- Sample type;
- Date and time of collection;
- Parameters for analysis; and
- Sampler's initials.

5.2.11 Document the sampling event on the Sample Collection Log or an equivalent form as specified in the project specific work plan. Note any pertinent field observations, conditions or problems on the Field Activity Daily Log. Any encountered problems or unusual conditions should also be immediately brought to the attention of the *Field Task Manager*.

5.2.12 Appropriately preserve, handle, package, and ship the samples per SOP 17 and the project specific work plan. The samples shall also be maintained under custody per SOP 17.

5.2.13 Fill and abandon the sample hole using a tremie pipe, per state requirements abandonment guidance. The tremie pipe grout method is performed by inserting PVC tremie pipe into the open borehole. Grout is then pumped through the tremie pipe to allow for jetting the tremie pipe to total depth. Once total depth has been reached, the tremie pipe is withdrawn from the borehole as grout is injected.

5.3 Subsurface Soil Sampling

5.3.1 The method to collect subsurface soil samples is to use a DPT equipment to bore to the desired sampling depth and then retrieve the sample with a sampler, e.g., 30 inch long and 1.5 inch outside diameter solid barrel, piston-sealed soil sampler.

The borehole stability should be maintained to prevent the recovery of slough in the samples. If sloughing cannot be controlled, then another sampling methodology may have to be considered.

5.3.2 As with surface soil samples, subsurface soil sampling follows the same sample collection procedures specified in Sections 5.2.1 through 5.2.13.

6.0 Required Forms

Bound Field Logbook
Chain of Custody

SOP NO. 4
REVISION NO. 0
DATE: 2/23/21

GROUNDWATER SAMPLING
STANDARD OPERATING PROCEDURE

1.0 Purpose

This Standard Operating Procedure (SOP) establishes guidelines and procedures for use by field personnel in the collection and documentation of groundwater samples for chemical analysis. Proper collection procedures are necessary to assure the quality and integrity of all groundwater samples.

2.0 References

2.1 EPA Region I, 19 January 2010. Low Stress (low flow) Purging and Sampling Procedure for Collection of Groundwater Samples from Monitoring Wells. EQASOP-GW 001

2.2 EPA, April 1996. Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures, EPA/540/S-95/504.

2.3 EPA, September 1987, Compendium of Superfund Field Operations Methods, EPA 540/P-87/001a, OSWER 9355.0-14.

2.4 EPA, August 1988, EPA Guidance for Conducting Remedial Investigation and Feasibility Studies Under CERCLA, Interim Final OSWER Directive 9355.3-01.

2.5 ASTM, 1988, Standards Technology Training Program - Groundwater and Vadose Zone Monitoring, Nielsen, et al.

3.0 Responsibilities

The *Field Manager* is responsible for ensuring that all sample collection activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QC/QC). The *Field* Manager is also responsible for periodic review of field generated documentation associated with this SOP, and for implementation of corrective action (i.e., retraining personnel, additional review of work plans and SOPs, writing variances to sampling requirements, etc.) if problems occur.

4.0 Definitions/Materials

4.1 Bladder Pump

A bladder pump is an enclosed cylindrical tube containing a flexible membrane bladder. Well water enters the bladder through a one-way check-valve at the bottom. Gas is forced into the annular space (positive displacement) surrounding the bladder through a gas supply line. The gas displaces the well water through a one-way check-value at the top. The water is brought to the surface through a water discharge line. Gas (typically air, nitrogen, or carbon dioxide) is provided by a compressor or cylinders.

4.2 Peristaltic Pump

A peristaltic pump is a self-priming, low volume pump consisting of a rotor and ball bearing rollers. Tubing placed around the rotors is squeezed by the rotors as they rotate. The squeezing produces a wavelike contraction movement which causes water to be drawn through the tubing. The peristaltic pump is limited to sampling at a head of less than 30 feet.

4.3 Electric Submersible Pump

An electric submersible pump is an enclosed cylindrical tube containing a motor with rotary attachments. Electrical power to the motor causes rotors or impellers to turn and displace the groundwater. Well water typically exits the top of the pump through a one-way check valve.

4.4 Bailer

A bailer is an enclosed cylindrical tube containing a floating ball check-valve at the bottom. Lowering the bailer into water causes the ball to float allowing water to enter the cylinder. Raising the bailer through the water causes the ball to settle, creating a seal to trap the water so that it can be brought to the surface.

4.5 Dedicated Groundwater Monitoring Equipment

Dedicated groundwater monitoring equipment is used to purge and sample only one well. The equipment is installed and remains in the well for the duration of the monitoring program. Dedicated equipment does not need to be decontaminated between sampling events.

4.6 Non-Dedicated Groundwater Monitoring Equipment

Non-dedicated groundwater monitoring equipment is decontaminated between wells and is used to purge and sample more than one well.

4.7 Materials:

- Clean rope or wire line of sufficient length for conditions.
- Appropriate sample containers with labels and preservatives, as required.
- Cooler with cold packs (or ice) for samples.
- Water quality multimeter (i.e., temperature, pH, conductivity, turbidity, etc.) meter.
- Multimeter calibration standards.
- Electronic water level indicator.
- Photoionization detector (PID).
- Lidded buckets or other containers for purge water.
- Decontamination supplies, as required by SOP 15.
- Personal protective clothing and equipment as required by the Accident Prevention Plan (APP).
- Field logbook and monitoring well purge forms.

5.0 Procedure

If non-dedicated equipment is to be used and the contaminant histories of the wells are known, it is advisable to establish a sampling order starting with the least contaminated well and progressing to the most contaminated well.

5.1 Low-Flow Methodology

The purpose of well purging is to remove stagnant water from the well and obtain a representative water sample while minimizing disturbance of the water column during sample collection. Using the low-flow purging methodology, the well will be purged until field parameters (e.g., pH, temperature, conductivity, turbidity, dissolved oxygen [DO], and oxidation-reduction potential [ORP]) have stabilized. Field parameter readings will be taken and recorded periodically, at intervals at least 3 minutes apart. Low-flow purging rates on the order of 0.1 – 0.5 L/min will be used. The maximum allowable drawdown during low-flow purging is 2.00 feet (ft). If the maximum allowable drawdown limit of 2.00 ft is exceeded, then the well will be purged and sampled according to Section 3.3.4.

Purge water will be managed as outlined in SOP 16. Necessary precautions will be taken to prevent spilling of potentially contaminated water. The water will be containerized, analyzed, and appropriately disposed based on the laboratory analytical results.

5.1.1 Low-Flow Purging

For standard low-flow well purging, the following steps will be performed at each well:

- The condition of the well completion (well lock, outer well casing, concrete well pad, protective bollards, well label, well cap, and well casing) and any unusual conditions of the area around the well will be noted on the Well Purge Form. The well may also be photographed as needed. Any deficiencies encountered will be repaired or reported to the *Project Manager* so repairs can be made.
- Set up and establish the exclusion zone around the work area if needed, using traffic cones.
- Don personal protective equipment (PPE) as specified in the APP.
- Measure the static water level in the well from the highest point on the well casing with a water level indicator (to the nearest 0.01 ft). If a high concentration of organic vapors is detected in the well using a photoionization detector (PID) at the wellhead, an oil/water interface probe will be used to determine the presence or absence of immiscible phase light non-aqueous phase liquids (LNAPL) or dense non-aqueous phase liquids (DNAPL).
- Once annually, the total depth of the well will be measured from the same measuring point on the casing and recorded to the neatest 0.01 ft if possible. The distance between the water sensor (zero point) and the end of the water level indicator probe should be measured independently and added to each total depth measurement.
- Slowly lower the pump and/or tubing into the well casing to the midpoint of the well screen. If the water level in the well is determined to be lower than

the top of the well screen, the tubing inlet will be placed approximately halfway between the top of the water column and the bottom of the well screen. Reinsert the water level indicator to monitor water levels during purging.

- Start the pump. As soon as water is discharging, adjust the pump speed to a rate between 0.1 – 0.5 L/min that is suitable to create minimal drawdown. During purging and sampling, the maximum allowable drawdown is 2.00 ft, but drawdown less than 0.50 ft is preferred.
- Using a stopwatch and a graduated cylinder or measuring cup, measure the pump flow rate. Monitor the water level, flow rate, cumulative volume withdrawn, and field parameters (temperature, pH, turbidity, conductivity, and DO) approximately every 3 to 5 minutes.
- Low-flow purging is complete when at least five water quality parameter readings have been taken and all required field parameters have stabilized (e.g., pH, temperature, conductivity, turbidity, DO, and ORP). Stabilization is achieved when three consecutive readings show pH values are within ± 0.1 pH units, temperature is within ± 1.0 degree Celsius, conductivity is within ± 10 percent (%), turbidity is within ± 10% or less than 15 nephelometric turbidity units (NTU), DO is within ± 10% or less than 0.50 milligrams per liter (mg/L), and ORP is within ± 10 millivolts (mV). The *Field Manager* has the responsibility of determining if redevelopment of any monitoring well is necessary and appropriate.
- After low-flow purging is complete, collect the groundwater sample per the steps described in Section 5.1.2.

5.1.2 Low-Flow Sampling

Using low-flow sampling procedures, samples for chemical analysis will be collected immediately following purging. The water quality samples will be taken from within the well screen interval. The following sampling procedure will be used at each well:

- After the monitoring well is considered purged and before filling sample vials, the water quality meter and any other instruments or hardware (e.g., flow meters, couplings, etc.) will be disconnected from the sampling tube.
- Immediately following purging, the pump will be used to collect the groundwater sample. The flow rate should not be altered between purging and sampling, and the pump tubing intake should remain at the same depth in the well.
- Identification labels for sample bottles will be completed and attached to sample bottles for each well prior to collecting samples.
- Sample vials will be filled using a steady, laminar stream of water to the extent practicable. Aeration of the sample will be reduced by slightly tilting the sample bottle so that the stream flows down the side of the vial.
- Containers for volatile organic compound (VOC) analysis will be filled first in a manner such that there is no/minimal headspace (total air bubble less than 2 millimeters [mm] in diameter). Other sample containers will be filled to the neck of the bottle. Samples intended for inorganic analyses will not be filtered in the field prior to filling the sample container.

- Fill containers for other parameter analyses into the neck of the bottles. Samples will be preserved and managed as detailed in the Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP) or project specific work plan.
- After the samples have been collected, they should immediately be placed in an ice-filled cooler until received by the appropriate laboratory for analysis.
- After removing the pump and equipment from the well, replace and lock the well cap.

5.2 Total Well Volume Methodology

If a water level drawdown greater than 2.00 ft occurs at a purge rate of 0.1 L/min or less, or if it is deemed necessary for other reasons, the total well volume purge and sampling methodology will be used. Using the total well volume purging methodology, the well will be purged with a pump or bailer until a minimum of three total well casing volumes have been removed or the well goes dry. Purging may be conducted at discharge rates between 0.25 and 1.5 gallons per minute (gpm) throughout the purge. If necessary, the pump head can be lowered to the bottom of the well during the purge to fully evacuate the well if it is going dry.

When purging by this methodology, if field parameters have not stabilized after three well casing volumes, purging will continue for up to two more well casing volumes. If after five well casing volumes have been purged, and the well still contains sufficient water to collect samples, then purging will cease and samples will be collected.

The volume of water in the well will be calculated based on the length of the saturated casing in the well and the screen diameter (see below for calculation of volumes). The well volume can be calculated in gallons using the following equation:

Well Volume V = H x F

where V = one well volume (measured in liters)
H = the difference between the depth of the well and static depth to water (ft)
F = factor for the volume of 1-ft saturated cylinder of well casing from the table below.

Diameter of Well Casing (inches)	F Factor (liters)
2	0.618
4	2.47
6	5.57

F can also be calculated from the following equation:

$$\mathbf{F = \pi\ (D/2)^2 \times 28.3\ L/ft^3}$$

where D = the inside diameter of the well casing (ft)

The well will be sampled immediately following purging without moving or adjusting the position of the pump, unless a bailer is used for sampling. The well purge water will be managed as outlined in SOP 16. Necessary precautions will be taken to prevent spilling potentially contaminated water. The water will be containerized and appropriately categorized prior to discharge.

If a well is purged to dryness, sample collection may begin when a sufficient volume of water has entered the well to allow collection of the necessary sample volume.

5.2.1 Total Well Volume Purging

For total well volume purging, the following procedures will be performed at each well:

- The condition of the well completion (well lock, outer well casing, concrete well pad, protective bollards, well label, well cap, and well casing) and any unusual conditions of the area around the well will be noted on the Well Purge Form. The well may also be photographed as needed. Any deficiencies encountered will be repaired or reported to the *Project Manager* so repairs can be made.
- Set up and establish the exclusion zone around the work area if necessary, using traffic cones.
- Don PPE as specified in the APP.
- The static water level in the well will be measured from the highest point on the well casing with a water level indicator (to the nearest 0.01 ft). If a high concentration of organic vapors is detected in the well using a PID at the wellhead, an oil/water interface probe will be used to determine the presence or absence of immiscible phase LNAPL or DNAPL.
- Once annually, the total depth of the well will be measured from the same measuring point on the casing and recorded to the neatest 0.01 ft if possible. The distance between the water sensor (zero point) and the end of the water level indicator probe should be measured independently and added to each total depth measurement.
- The following applies if a pump is used to purge the well:
  - Slowly lower the pump or pump tubing into the well casing to halfway between the top and bottom of the well screen. If, during gauging, the water level in the well is determined to be lower than the top of the well screen, then the tubing inlet will be placed approximately halfway between the top of the water column and the bottom of the well screen. Reinsert the water level indicator to monitor water levels during purging.
  - Start the pump. Using a stopwatch and a graduated cylinder, measure the pumping rate. Monitor the water level, pumping rate, cumulative withdrawal, and field parameters every 15-30 minutes and/or per well volume. Field parameters (e.g., pH,

temperature, conductivity, turbidity, DO, and ORP) will be monitored.

- The following applies if a bailer is used to purge the well:
  - Attach a new piece of nylon rope to the bailer and slowly lower until the bailer is completely submerged, being careful not to drop the bailer into the water, causing turbulence and the possible loss of volatile organic contaminants.
  - Pull the bailer out, ensuring the rope either falls onto clean plastic sheeting or never touches the ground.
  - Continue purging the well and perform field parameter measurements after each purged well volume (at a minimum).

- Purging is complete when one of the following three scenarios occurs:
  - Three well casing volumes have been purged and all required field parameters have stabilized. Water parameters will be measured after removal of each volume (at a minimum). Stabilization is achieved when three consecutive readings show pH values are within ± 0.1 pH units, temperature is within ± 1.0 degree Celsius, conductivity is within ± 10%, turbidity is within ± 10% or less than 15 NTU, DO is within ± 10% or less than 0.50 mg/L, and ORP is within ± 10 mV. The *Field Manager* has the responsibility of determining if redevelopment of any monitoring well is necessary and appropriate.
  - Five well casing volumes have been removed.
  - The well is pumped or bailed dry.

- After purging is completed, collect the groundwater sample per the steps described in Section 5.2.2.

5.2.2 Total Well Volume Sampling

Samples for chemical analysis will be collected immediately following purging. For wells that were purged dry, samples will be collected as soon as possible after a sufficient volume of groundwater is available in the well. The following sampling procedure will be used at each well:

- After the monitoring well is considered purged and before filling sample vials, the water quality meter and any other instruments or hardware (e.g., flow meters, couplings, etc.) will be disconnected from the sampling tube.
- A pump or bailer will be used to collect the groundwater sample. If using a pump, prior to sample collection, reduce the pump rate to produce a steady, laminar stream of water to the extent practicable.
- Identification labels for sample bottles will be completed and attached to sample bottles for each well prior to collecting samples.

- Sample vials will be filled using a steady, laminar stream of water to the extent practicable. Aeration of the sample will be reduced by slightly tilting the sample bottle so that the stream flows down the side of the vial.
- Containers for VOC analysis will be filled first in a manner such that there is no/minimal headspace (total air bubble less than 2 mm in diameter). Other sample containers will be filled to the neck of the bottle. Samples intended for inorganic analyses will not be filtered in the field prior to filling the sample container.
- Fill containers for other parameter analyses to the neck of the bottles. Samples will be preserved and managed as detailed in the UFP-QAPP or project specific work plan.
- After the samples have been collected, they should immediately be placed in an ice-filled cooler until received by the appropriate laboratory for analysis.
- After removing the pump and equipment from the well, replace and lock the well cap.

6.0 Required Forms

Well Purge Form
Sample Log Form
Bound field logbook

GROUNDWATER LEVEL MEASUREMENT
STANDARD OPERATING PROCEDURE

1.0 Purpose

This Standard Operating Procedure (SOP) establishes guidelines for personnel to use in determining the depth to water in monitoring wells.

2.0 References

2.1 EPA, 1986, RCRA Groundwater Monitoring Technical Enforcement Guidance Document, OSWER-9950.1, U.S. Government Printing Office, Washington, D.C.

2.2 EPA, 1991, Environmental Compliance Branch, Standard Operating Procedures and Quality Assurance Manual, Region IV, Environmental Services Division, Athens, Georgia, U.S. Government Printing Office, Washington, D.C.

2.3 U.S. Environmental Protection Agency (EPA), 1991, Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells, Environmental Monitoring Systems Laboratory Office of Research and Development U.S. Environmental Protection Agency, Las Vegas, Nevada, EPA160014-891034.

3.0 Responsibilities

3.1 The *Field Manager* is responsible for ensuring that all sample collection activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC). The *Field Manager* is responsible for the periodic review of documentation generated as a result of this SOP and the periodic review and audit of field personnel as they perform the work. If problems arise, the *Field Manager* is also responsible for verifying implementation of corrective action(s) (i.e., retraining personnel, additional review of work plans and SOPs, variances to requirements, and issuing nonconformances) and assuring through monitoring the continued implementation of stated corrective actions.

3.2 The *Field Sampling Team* is responsible for ensuring that monitoring well water level measurements are properly collected and documented.

4.0 Definitions/Materials

A number of devices are available for the determination of water level measurements in monitoring wells. Those most commonly used and covered in this SOP include: steel tapes, electric sounders, and petroleum product probes. The equipment must be capable of recording a measurement to the accuracy required by the project.

5.0 Procedure

Water level measurements are commonly taken in each monitoring well immediately prior to, during, and following well development, and both before and during well purging and sampling. Water level measurements may also be taken where no development or purging is being conducted, strictly to monitor or generate water table or pieziometric surfaces. When such measurements are made to monitor water table or pieziometric surfaces, water levels in all wells at a given site should be measured within a 24-hour maximum period whenever possible. When measuring wells for water for water table or potentiometric surface analysis, and if the contaminant history is known for each of the wells, it is advisable to monitor water levels beginning with the least contaminated wells first and progressing to the most contaminated wells last.

5.1 Equipment Selection

Project data quality objectives and site characteristics must be taken into account when determining the water level measurement equipment to use. The total number of wells to be measured, weather, pumping, and construction can all affect water level measurements.

5.2 Determining Water Level Measurements in Monitoring Wells

The standard procedure for determining depth to water is described below.

Prior to taking a water level measurement at each well, decontaminate the measuring device according to the procedures outlined in SOP 15. During decontamination, all measuring tapes should be inspected for kinks, cracks, or tears and, if present, repaired or replaced with undamaged equipment.

Visually inspect the well to ensure that it is undamaged, properly labeled and secured. Any damage or problems with the well should be noted on the appropriate field form and the *Field Manager* notified for repair or replacement.

Uncap the well and monitor the air space immediately above the open casing per the project-specific Accident Prevention Plan (APP). Observe if any air is flowing into or out of the casing. In the event such conditions are observed, they should be noted on the Water Level Measurement Form, Well Development Record, Well Purge Form, or bound field logbook as appropriate. Lower the electric sounder or equivalent (product probe or steel tape) into the well until the water surface is encountered. If air is observed to be entering flowing out of the casing, the sounder should not be placed inside the well until the air flow stops and pressure equalizes.

Measure the distance from the water surface to the permanent reference point. The reference point is usually a groove cut into the north side of the casing. The point of water level measurement (if not the groove cut into the casing) should then be noted on the appropriate form on which the water level is recorded. Any aboveground completions without permanent reference points or marks should be brought to the attention of the *Field Manager*.

Collect measurements until two consecutive measurements are identical or within the 0.01 ft. Record all appropriate information on the appropriate form or logbook depending upon the task being performed. At a minimum, the following information must be recorded:

- Project name;
- Unique well identification number;
- Date and time of water level measurement;
- Depth to water to the specified tolerance;
- Total depth of monitoring well (collected at least once per calendar year for each well measured);
- Weather conditions; and
- Any problems encountered.

If product or other nonaqueous liquid is encountered, notify the *Field Manager*.

Cap and relock the well.

6.0 Required Forms

Bound Field Logbook
Water Level Measurement Form
Well Development Record
Well Purge Form

SOP NO. 7
REVISION NO. 0
DATE: 2/23/21

MONITORING WELL INSTALLATION STANDARD OPERATING PROCEDURE

1.0 Purpose

This Standard Operating Procedure (SOP) provides procedures and requirements for the installation of monitoring wells using rotary, dual-tube percussion, or hollow-stem auger drilling techniques. Monitoring wells are installed to provide access to groundwater for collecting samples, as well as for obtaining water level and other data. Because monitoring wells are used to collect samples, it is important that construction materials not interfere with sample quality either by contributing contaminants or by sorbing contaminants already present. Further, construction materials must be compatible with (i.e., not degraded by) contaminants present in soils or groundwater.

Monitoring wells are potential vertical contaminant migration routes between aquifers or from the surface to the subsurface. Construction procedures and standards must ensure that neither passive nor active introduction of contaminants can occur. Properly installed hydraulic seals and locking well covers reduce the potential for cross-contamination of monitoring wells.

2.0 References

2.1 U.S. Environmental Protection Agency (EPA), Manual of Water Well Construction Practices, U.S. Environmental Protection Agency, Office of Water Supply, U.S. Government Printing Office, Washington D.C.

2.2 U.S. Environmental Protection Agency (EPA), 1986, Resource Conservation and Recovery Act (RCRA) Ground Monitoring Technical Enforcement Guidance Document, OSWER- 9950.1, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, U.S. Government Printing Office, Washington D.C.

2.3 U.S. Environmental Protection Agency (EPA), 1987, A Compendium of Superfund Field Operations Methods, EPA-500/P-87/001, U.S. Government Printing Office, Washington D.C.

2.4 U.S. Environmental Protection Agency (EPA), 1991, Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells, Environmental Monitoring Systems Laboratory Office of Research and Development U.S. Environmental Protection Agency, Las Vegas, Nevada, EPA160014-891034.

3.0 Responsibilities

3.1 The *Field Manager* is responsible for ensuring that all monitoring well installation activities are conducted and documented in accordance with this and any other appropriate procedures. This will be accomplished through staff training and by quality assurance/quality control (QA/QC) monitoring activities. The *Field Manager* is

responsible for periodic review of well installation activities to assure implementation of this SOP. The *Field Manager* is also responsible for the review and approval of corrective action (i.e., retraining personnel, additional review of work plans and SOPs, variances to monitoring well installation requirements, issuing nonconformances, etc.) identified during the performance of these activities.

3.2 The *Site Geologist* assigned to monitoring well installation activities is responsible for completing the tasks according to specifications outlined in this SOP and other appropriate procedures. All staff are responsible for reporting deviations from the procedures to the *Field Manager*.

4.0 Definitions/Materials

4.1 Cuttings

Pieces of soil, sediment, or rock cut by a bit in the process of drilling borings.

4.2 Borehole

Any hole drilled into the subsurface for the purpose of identifying lithology, collecting soil samples, and/or installing groundwater wells.

4.3 Grout

For the purposes of this SOP, the term "grout" consists of a neat cement grout generally containing three to five percent bentonite powder to water by weight. The grout is emplaced as a slurry, and once properly set and cured, is capable of restricting movement of water.

4.4 Hollow-Stem Auger Drilling

A drilling method using augers with open centers. The augers are advanced with a screwing or rotating motion into the ground. Cuttings are brought to the surface by the rotating action of the augers, thereby clearing the borehole.

4.5 Air Rotary Casing Hammer Drilling

A drilling method using a nonrotating drive casing that is advanced simultaneously with a slightly smaller diameter rotary bit attached to a string of drill pipe. The drive casing is a heavy-walled, threaded pipe that allows for pass-through of the rotary drill bit inside the center of the casing. Air is forced down through the center drill pipe to the bit, and then upward through the space between the drive casing and the drill pipe. The upward return stream removes cuttings from the bottom of the borehole.

4.6 Mud Rotary Drilling

For the purposes of this monitoring well installation SOP, the term "mud rotary drilling" refers to direct circulation (as opposed to reverse circulation) mud rotary drilling. Mud rotary drilling uses a rotating drill bit which is attached to the lower end of a string of drill pipe. Drilling mud is pumped down through the inside of the drill pipe and out through the bit. The mud then flows upward in the annular space between the borehole and the drill pipe, carrying the cuttings in suspension to the surface.

4.7 Dual-tube Percussion Drilling
A drilling method using nonrotating drive casing with a bit on the bottom of the casing string. A smaller diameter tube or drill pipe is positioned inside the drive casing. The drive casing is advanced by the use of a percussion hammer, thereby causing the bit to cut or break up the sediment or soil at the bottom of the boring. Air is forced down the annular space between the drive casing and inner drill pipe and cuttings are forced up the center of the inner drill pipe.

4.8 Monitoring Well
A well that provides for the collection of representative groundwater samples, the detection and collection of representative light and dense nonaqueous phase organic liquids, and the measurement of fluid levels.

4.9 Annular Space
The space between:

- Concentric drill pipes;
- An inner drill pipe and outer drive casing;
- Drill pipe or drive casing and the borehole wall; or
- Well screen or casing and the borehole wall.

4.10 Filter Pack
Granular filter material (sand, gravel, etc.) placed in the annular space between the well screen and the borehole to increase the effective diameter of the well and prevent fine-grained material from entering the well.

4.11 Well Screen
A perforated, wire wound, continuous wrap or slotted casing segment used in a well to maximize the entry of water from the producing zone and to minimize the entrance of sand.

4.12 Tremie
A tubular device or pipe used to place grout, bentonite, or filter pack in the annular space.

5.0 Well Installation Procedures

This section contains the procedures for monitoring well installation activities. The procedures described herein are applicable as requirements for monitoring well installations using mud rotary, air rotary, air rotary casing hammer, dual tube percussion, or hollow-stem auger drilling techniques.

Site-specific factors need to be considered in the selection of well construction and completion materials, specification of well designs, and choosing well drilling methods. The factors to consider include:

- Objectives of the monitoring well;
- Specific location of the well to be installed;
- Zone or depth well is to be installed;
- Drilling method(s) to be used;
- Well construction materials to be used;
- Specification of well design(s) including Well Construction Diagrams; and
- Additional procedures or requirements beyond this SOP.

Before mobilization of a rig to the well site, ensure that the monitoring well location has been appropriately cleared for underground utilities and buried objects per the Accident Prevention Plan (APP). Review all forms and diagrams documenting the location of the cleared monitoring well site and the location of any identified underground utility lines or other buried objects.

Decontaminate all downhole equipment and well construction materials before monitoring well installation, as described in SOP 7. Decontaminate the drilling rig and all drilling equipment before monitoring well installation per SOP 14.

Clear the work site of all brush, tall grass that might contact hot surfaces or exhaust gas from the drill rig or support equipment, and minor obstructions and then mobilize the rig to the monitoring well location. The rig geologist or engineer should then review with the driller the proposed well design and details of the well installation including any anticipated potential drilling or completion problems.

Calibrate health and safety monitoring equipment according to the instrument manufacturer's specifications and SOP 2. Document the calibration results on the appropriate form(s). Instruments that cannot be calibrated according to the manufacturer's specifications will be removed from service and tagged.

Workers will be provided with, and don, the appropriate personal protective equipment as specified by the APP. Typically, the minimum personal protection will include a hard hat, safety glasses, gloves, steel-toed boots, hearing protection, and coveralls.

Commence drilling and advance the borehole while conducting health and safety monitoring in accordance with the APP. Perform readings as often as necessary to ensure the safety of workers. Record all measurements on the appropriate form(s). Record all other pertinent information (date, site, well or boring number, and location) per SOP 18. Also note and record observed field conditions, any unusual circumstances, and weather conditions. Drilling of the borehole should be conducted in conformance with applicable SOPs, as appropriate.

During drilling, collect and describe representative cutting and soil samples. Compile a boring or lithologic log from the cuttings and samples.

At total depth, remove soil cuttings through circulation or rapidly spinning the augers prior to constructing the well. Review logs and notes with the driller for any zones or

depths exhibiting drilling problems which may affect the well installation. Condition the hole or take other actions mutually agreed upon by the Project Geologist, lead technical personnel, and the driller to ensure or aid in the well development.

Remove the drill pipe and bit if using rotary techniques, or remove the center bit boring if using the hollow-stem auger technique. The well construction materials will then be installed inside the open borehole or through the center of the drive casing or augers.

Measure the total depth of the completed boring using a weighted sounding line. The borehole depth is checked to assure that formation material has not heaved to fill the borehole. If heaving has taken place, options for cleaning, redrilling, or installation in the open section of the boring should be discussed with lead technical personnel.

In the event that the hole was overdrilled, grout, bentonite pellets, or bentonite chips may be added to the bottom of the boring to raise the bottom of the hole to the desired depth. The grout should be pumped through a tremie pipe and filled from the bottom of the boring upward. During grouting, the tremie pipe should be submerged below the top of the grout column in the borehole to prevent free-fall and bridging. If bentonite is used, it should be added gradually to prevent bridging. Grout or bentonite addition will stop when its level has reached approximately one foot below the desired base of the well string (casing, screen, end plug or sump, etc.). The bentonite plug will be hydrated for at least one hour before installation of a filter pack.

Calculate volumes of filter pack, bentonite pellets/slurry, and grout required, based on borehole and well casing dimensions. Determine the appropriate filter pack and well screen slot size for the monitoring well.

Place a layer of filter pack (one to two feet) at the bottom of the borehole. The filter pack will be installed through the center of the drive casing/augers. Filter pack will be added slowly while withdrawing the drive casing/augers.

Measure and record the casing, screen, and end plug (sump) lengths. Inspect all well construction materials prior to installation to assure that no damage has occurred during shipment and decontamination activities.

Connect and carefully lower the well string through the open borehole, drive casing, or inside of the augers until the well string is at the desired depth. The well string should be suspended by the installation rig and should not rest on the bottom of the boring. In the event the well string was dropped, lowered abruptly, or for any other reason suspected of being damaged during placement, the string should be removed from the boring and inspected. In certain instances, the well string may rise after being placed in the borehole due to heaving sands. If this occurs, the driller must not place any drilling equipment (drill pipe, hammers, etc.) on the well string to prevent the casing from rising. The amount of rise should be noted by the rig geologist or engineer who should then consult lead technical personnel for an appropriate course of action.

Record the following information on the Well Completion Form and/or other appropriate forms:

- Length of well screen and casing (per section and total);
- Total depth of well boring;
- Depth from ground surface to top of grout or bentonite plug in bottom of borehole (if present);
- Depth to base of well string; and
- Depth to top and bottom of well screen.

When using the mud rotary drilling technique, tremie the filter pack into the annular space around the screen. Clean, potable water may be used to assist with the filter pack tremie operation. For all other drilling techniques, the filter pack may be allowed to free fall or be tremied. If using drive casing or augers, the drive casing or augers should be pulled slowly during filter pack installation in increments no greater than five feet.

Filter pack settlement should be monitored by initially measuring the sand level (before beginning to withdraw the drive casing/augers). In addition, depth soundings using a weighted tape shall be taken repeatedly to continually monitor the level of the sand. The top of the well casing shall also be monitored to detect any movement due to settlement or from drive casing/auger removal. If the top of the well casing moves upwards at any time during the well installation process, the driller should not be allowed to set drilling equipment (downhole hammers, drill pipe, etc.) on the well string to prevent further movement.

Filter pack should be added until its height is approximately two feet above the top of the screen (unless otherwise specified), and verification of its placement (by sounding) should be conducted. The filter pack should then be gently surged using a surge block or swab in order to settle the pack material and reduce the possibility of bridging.

The height of the filter pack will then be re-sounded and additional filter pack placed as necessary. Once the placement of the filter pack is completed, the depth to the top of the pack is measured and recorded on the Well Completion Form or other appropriate forms.

A three-foot thick (unless otherwise specified) bentonite seal is then installed on top of the filter pack. If pellets or chips are used, they should be added gradually to avoid bridging. Repeated depth soundings will be taken using a weighted tape to ascertain the top of the bentonite seal. The seal should be allowed to hydrate for at least one hour before proceeding with the grouting operation.

After hydration of the bentonite seal, grout is then pumped through a tremie pipe and filled from the top of the bentonite seal upward. The bottom of the tremie pipe should be maintained below the top of the grout to prevent free fall and bridging. When using drive casing or hollow-stem auger techniques, the drive casing/augers should be raised in incremental intervals, keeping the bottom of the drive casing/augers below the top of the grout. Grouting will cease when the grout level has risen to within approximately one to

two feet of the ground surface, depending on the surface completion type (flush mount versus aboveground). Grout levels should be monitored to assure that grout taken into the formation is replaced by additional grout. If settling of the grout occurs, additional topping off of the grout may be necessary.

For aboveground completions, the protective steel casing will be centered on the well casing and inserted into the grouted annulus. Prior to installation, a 2-inch deep temporary spacer shall be placed between the PVC well cap and the bottom of the protective casing cover to keep the protective casing from settling onto the well cap.

After the protective casing has set, a drainage hole may be drilled into the protective casing. The drainage hole is positioned approximately two inches above ground surface. The protective casing will be painted with a rust-preventive colored paint.

The well head will be labeled to identify, at a minimum, the well number, depth, and date of installation.

A minimum of 24 hours after grouting should elapse before installation of the concrete pad and steel guard posts for aboveground completions, or street boxes or vaults for flush mount completions.

For aboveground completions, a concrete pad, usually 3-feet by 3-feet by 4-inches thick, is constructed at ground surface around the protective steel casing. The concrete should be sloped away from the protective casing to promote surface drainage from the well.

For aboveground completions, where traffic conditions warrant extra protection, three or four steel bucking posts (bollards) will be embedded to a depth approximately 1.5 feet below the top of the concrete pad. The posts will be installed in concrete filled post holes spaced equally around the well. Where removal of bucking posts is required for well access, mounting sleeves should be imbedded into the concrete.

For flush mount (or subgrade) completions, a street box or well vault is set and cemented in position. The top of the street box or vault will be raised slightly above grade and the cement sloped to grade to promote surface drainage away from the well.

Following well completion and demobilization of the rig, the well site should be cleared of all debris and trash and restored to a neat and clean appearance. All investigation-derived waste generated at the well site should be appropriately contained and managed per SOP 16.

6.0 Required Forms

Bound Field Logbook
Well Construction Diagram
Lithologic/Soil Boring Log

WELL DEVELOPMENT
STANDARD OPERATING PROCEDURE

1.0 Purpose

This Standard Operating Procedure (SOP) establishes guidelines for specifying, assessing and documenting the well development process. Monitoring wells are developed to remove skin (i.e., near-well bore formation damage), well drilling fluids, sediments, and to settle and remove fines from the filter pack. Wells should not be developed for 24 hours after completion when a cement bentonite grout is used to seal the annular space, nor after 7 calendar days beyond internal mortar collar placement.

2.0 References

2.1 U.S. Environmental Protection Agency (EPA), August 1988, Guidance for Conducting Remedial Investigation and Feasibility Studies under CERCLA, Interim Final OSWER Directive 9355.3-01.

2.2 U.S. Environmental Protection Agency (EPA), 1987, A Compendium of Superfund Field Operations Methods, EPA-540/P-87/001a, U.S. Government Printing Office, Washington D.C.

2.3 ASTM, 1988, Standards Technology Training Program - Groundwater and Vadose Zone Monitoring, Nielsen, et al.

2.4 U.S. Environmental Protection Agency (EPA), 1991, Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells, Environmental Monitoring Systems Laboratory Office of Research and Development U.S. Environmental Protection Agency, Las Vegas, Nevada, EPA160014-891034.

3.0 Responsibilities

3.1 The *Field Manager* is responsible for ensuring that monitoring wells are properly developed and that the development process is properly documented. This will be accomplished by staff training and by maintaining quality assurance/quality control (QA/QC). The *Field Manager* is responsible for periodic review of field generated documentation associated with well development. If deviations from project requirements occur, the *Field Manager* is also responsible for issuing notices of nonconformances and requests for corrective action.

3.2 The *Field Sampling Team is* responsible for conducting monitoring well development and documentation in accordance with the specifications outlined in this SOP.

4.0 Definitions/Materials

4.1 Well Development - The act of removing fine grained sediment and drilling fluids from the sand pack and formation in the immediate vicinity of the well, thus increasing the porosity and permeability of the materials surrounding the intake portion of the well.

4.2 Eductor Pipe - The pipe used to transport well discharge water to the surface.

4.3 Materials

- Submersible pump or bailer.
- Power source (e.g., generator), if required.
- Electronic water level indicator and/or oil/water interface probe.
- Temperature, conductivity, pH, and turbidity meters.
- Personal protective equipment as specified in the project health and safety plan.
- Photoionization detector (PID).
- Teflon-coated stainless steel cable or nylon cord.
- Well Development Logs.

5.0 Procedure

5.1 General

The most common methods used to develop monitoring wells consist of surging, bailing, and pumping. This SOP for field personnel to use in assessing and documenting well development is intended only for these development methods.

5.2 Well Development

Decontaminate the rig and development equipment in accordance with SOP 14 and 15, respectively.

Calibrate all field analytical test equipment (e.g., water quality multimeter) according to the instrument manufacturer's specifications and SOP 2. Instruments that cannot be calibrated according to the manufacturer's specifications will be removed from service, tagged with an out of calibration label, and segregated (when possible) from the calibrated equipment area.

Visually inspect the well to ensure that it is undamaged, properly labeled and secured. Any observed problems with the well should be noted in the bound field logbook or appropriate form and reported to the *Field Manager*.

Unlock the well and obtain a depth to water level measurement according to the procedures outlined in SOP 6. Calculate the volume of water in the well (cased well volume) as follows:

$$3.14 \; x \; (d/2)^2 \; x \; (h_1 - h_2) \; x \; 7.48$$ *cased well volume* (*in gallons*)

where:

d = inside diameter of well casing (in feet)

h_1 = depth of well from top of casing (in feet)
h_2 = depth to water from top of casing (in feet)

The depth to the bottom of the well should be sounded and then compared to the completion form or diagram for the well. If sand or sediment are present inside the well, it should first be removed by bailing. Do not insert bailers, pumps, or surge blocks into the well if obstructions, parting of the casing, or other damage to the well is suspected. Instead report the conditions to the *Field Manager* and obtain approval to continue or cease well development activities.

Begin development by first gently surging followed by bailing and pumping. This is then continued with alternate surging and bailing or pumping. At no time should the surge block be forced down the well if excessive resistance is encountered. During development, the bailer should not be allowed to free-fall or descend rapidly such that it becomes lodged in the casing or damages the end cap or sediment trap at the bottom of the well.

While developing via pumping, take periodic water level measurements (at least one every five minutes) to determine if drawdown is occurring and record the measurements on the Well Development Record.

While developing, calculate the rate at which water is being removed from the well. Periodically record the volume of water removed on the Well Development Record.

While developing, water is also periodically collected directly from the eductor pipe or bailer discharge and readings taken of the indicator parameters: pH, temperature, conductivity, and turbidity. Development is considered complete when three consecutive readings show pH values are within ± 0.1 pH units, temperature is within ± 1.0 degree Celsius, conductivity is within ± 10 percent (%), and turbidity is within ± 10% or less than 15 nephelometric turbidity units (NTU), and a minimum of three well volumes of water have been removed. In certain instances, for slow recharging wells, the parameters may not stabilize. In this case, well development is considered complete upon removal of the minimum of three well volumes or in instances with limited groundwater resources, the well may be purged dry on three separate occasions.

Obtain a water level and pH, temperature conductivity, and turbidity measurements at the completion of development.

Complete documentation of the well development event on the Well Development Record. At a minimum this record must contain:

- Project name;
- Name(s) of the personnel responsible for well development;
- Well identification number;
- Well depth, casing size, and completion date;
- Method(s) of development;

- Calculated well volume and total volume of water removed;
- Water levels (including the time of measurement);
- Physical description of the water (e.g., discoloration, turbidity, odor, etc.) and solids removed from the well; and
- Measurements of pH, temperature, conductivity, and turbidity (including the time of collection).

Collect and appropriately transport and dispose of IDW water removed from the well in accordance with SOP 16.

Allow the well to recover for at least 24 hours prior to sampling.

6.0 Required Forms
Bound Field Logbook
Well Development Record Form

SOP NO. 10
REVISION NO. 0
DATE: 2/23/21

DRILLING
STANDARD OPERATING PROCEDURE

1.0 Purpose

This Standard Operating Procedure (SOP) establishes guidelines and procedures for field personnel to use during the supervision of drilling operations involving hollow stem auger techniques.

2.0 References

2.1 U.S. Environmental Protection Agency (EPA), 1991, Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells, Environmental Monitoring Systems Laboratory Office of Research and Development U.S. Environmental Protection Agency, Las Vegas, Nevada, EPA160014-891034.

2.2 F.G. Driscoll, 1986, Groundwater and Wells, Johnson Filtration Systems Inc., St. Paul, Minnesota.

3.0 Responsibilities

3.1 The *Field Manager* is responsible for ensuring that all hollow stem auger drilling activities are conducted and documented in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC). The *Field Manager* is responsible for periodic review of field generated documentation associated with this SOP. The *Field Manager* is also responsible for the implementation of corrective action (i.e., retraining personnel, additional review of work plans and SOPs, variances to hollow stem auger drilling requirements, issuing nonconformances, etc.) if problems occur.

3.2 The *Project Geologist* assigned to hollow stem auger drilling activities are responsible for completing their tasks according to specifications outlined in this SOP and other appropriate procedures. All staff is responsible for reporting deviations from the procedures to the *Field Manager*.

4.0 Definitions/Materials

4.1 Hollow Stem Auger Drilling

A drilling method using rotating auger flights (typically in 5 foot joints) with a bit on the bottom of the lead flight (sometimes called the "lead auger"). The flights consist of a hollow pipe and an outer spiral plate, which when rotated, forces soil cuttings upward along the borehole wall to the surface. The auger string is advanced by rotation, with pressure exerted by the rig, forcing the bit to cut the soil at the bottom and direct cuttings to the augers.

A retractable plug with a pilot bit is placed at the bottom of the auger string to prevent cuttings from entering the hollow stem. When the plug is retracted, a sampler may be sent through the hollow center to sample soil at the bottom of the borehole without requiring the augers to be removed. A wireline sampler may also be attached to the inside of the lead auger for coring as the borehole is advanced.

This method is commonly used for drilling and sampling of soil borings, collection of soil gas and screening-level water samples, and installation of some smaller diameter wells. The well casing string may be placed through the hollow stem.

The hollow stem auger drilling method has advantages over other drilling techniques in certain circumstances, and disadvantages in others. This method is highly suitable for unconsolidated and consolidated fine-grained soils. Hollow-stem auger drilling can achieve the most rapid rates of penetration in soft sticky clay-dominated soils. However, coarse and consolidated gravels and hard bedrock may be too dense for adequate drill penetration. Soil cuttings are typically disaggregated and remolded, making bedding, fabric, and soil property determination difficult.

The most reliable method for logging of soils during hollow stem auger drilling is by collecting relatively intact samples through the hollow stem. An advantage of the hollow stem auger method is that soil samples can be readily obtained from the bottom of the hole without requiring the removal of the auger string (unlike air or mud rotary methods).

This drilling method may be used to install monitoring wells (limited by diameter) as there is good depth control, and the auger can be progressively pulled as well construction materials are added to the borehole. The methodology may also be used to drill out monitoring wells for abandonment.

Another advantage of the hollow stem auger method is that air or mud are not required as circulating media. Therefore, there is limited to no potential for flushing of soil samples collected for chemical analyses, and a reduction in volumes of investigated-derived waste (IDW) requiring costly handling and management procedures. Auger-type rigs can be significantly smaller than other types of rigs, making them the most suitable for some jobs with significant space constraints, including overhead clearance.

Additional disadvantages of the hollow stem auger method include a typical maximum depth of 100 to 200 feet (may be less depending on soil conditions). Hard soil horizons or very coarse gravel (cobbles and boulders) may be impenetrable with this method.

5.0 Procedure

5.1 Drilling Site Mobilization

5.1.1 Site Preparation

The logistics of drilling, logging, sampling, cuttings/fluid containment, and/or well construction should be determined before drilling. The site should be prepared as per the predetermined plan.

Before mobilization, the *Project Geologist* should assess the drilling site with the driller. This assessment should identify potential hazards (slip/trip/fall, overhead power lines, etc.), and determine how drilling operations may impact the environment (dust, debris, noise). Potential hazards should be evaluated and corrected, or the borehole location changed or shifted.

The *Project Geologist* or appropriate designee should ensure that all identifiable underground utilities around the drilling location have been marked, and the borehole location appropriately cleared. At a minimum, copies of the site clearance documents should be kept on-site.

5.1.2 Rig Decontamination and Preparation

All drilling and sampling equipment should be decontaminated before drilling as per SOP 14 and 15.

The driller and *Project Geologist* should inspect the drilling equipment for proper maintenance and appropriate decontamination prior to each time the rig is mobilized to a site. All clutches, brakes and drive heads should be in proper working order. All cables and hydraulic hoses should be in good condition. All auger joints and bits should also be in good condition (e.g., no cracked or bent blades, bits are not excessively worn, etc.). The Accident Prevention Plan (APP) describes the daily drill rig inspection procedure that should be followed.

Any observed leakage of fluids from the rig should be immediately repaired and the rig decontaminated again before it is allowed to mobilize.

5.1.3 Mobilization and Set-Up

Once the site is prepared, the rig is mobilized to the site and located over the borehole location. The rig is leveled with a set of hydraulic pads attached to the front and rear of the rig. The driller should always raise the mast slowly and carefully to prevent tipping or damaging the rig, and avoiding obstructions or hazards.

Appropriate barriers and markers should be in place prior to drilling, as per the APP. Visqueen (plastic) may be required beneath the rig.

Appropriate cuttings and other investigation-derived waste (IDW) containment should be set on site prior to commencement of drilling.

5.1.4 Health and Safety Requirements

Tailgate Safety Meetings should be held in the manner and frequency stated in the APP. All personnel at the site should have appropriate training and qualifications as per the APP.

During drilling all personnel within the exclusion zone should pay close attention to rig

operations. The rotating auger blades can snag or catch loose clothing and are a significant safety hazard.

Establishing clear communication signals with the drilling crew is mandatory since verbal signals may not be heard during the drilling process. The entire crew should be made aware to inform the *Lead Driller and Project Geologist* of any unforeseen hazard, or when anyone is approaching the exclusion zone.

5.2 Drilling Procedures

5.2.1 Breaking Ground

Prior to the commencement of drilling, all safety sampling and monitoring equipment will be appropriately calibrated. The *Project Geologist* should consult with the driller regarding the appropriate equipment to be used for penetration of the surface cover (e.g., asphalt, concrete, cement, etc.). In the event of breaking ground where a shallow subsurface hazard may exist (unidentifiable utility, trapped vapors, etc.), the driller should be informed of the potential hazard and drilling should commence slowly to allow continuous visual inspection and/or monitoring, and if necessary, stop for probing. In areas with unknown utility locations, or outside public rights-of-way, a private utility locate company may be required.

5.2.2 Borehole Drilling

During drilling operations, and as the borehole is advanced, the *Project Geologist* will generally:

- Observe and monitor rig operations;
- Conduct all health and safety monitoring and sampling, and supervise health and safety compliance;
- Prepare a lithologic log from soil samples or cuttings; and
- Supervise the collection of, and prepare soil, soil vapor, and groundwater samples.

As drilling progresses the *Project Geologist* should observe and be in frequent communication with the driller regarding drilling conditions. This includes relative rates of penetration (indicative of fast or slow drilling) and chattering or bucking of the rig. These conditions, including the relative drilling rate, should be recorded on the boring log. Drilling should not be allowed to progress faster than the *Project Geologist* can adequately observe conditions, compile boring logs, and supervise safety and sampling activities. The *Project Geologist* should instruct the *Lead Driller* to slow down or pause work as needed in order to keep up with all required tasks.

The *Project Geologist* should also observe the rig operations, including the make-up and tightening of connections as additional auger joints are added to the auger string. Any observed problems, including significant down time, and their causes are recorded in the bound field logbook.

Cuttings and fluids containment during drilling should be observed and supervised by the *Project Geologist*.

The *Project Geologist* will oversee or conduct appropriate health and safety sampling and monitoring. If any potentially unsafe conditions are evident from the above drilling observations and the health and safety sampling and monitoring, the *Project Geologist* may suspend drilling operations at any time and take appropriate actions as per the APP. In the event suspension of drilling activities occur:

- The *Field Manager* and *Project Manager* must be informed of the situation;
- Appropriate corrective action must be implemented before drilling may be continued; and
- The observed problem, suspension, and corrective action will be entered in the bound field logbook.

During drilling the *Project Geologist* will compile a boring log. The log will be compiled preferably from soil samples recovered while drilling. Logs should only be compiled from drill cuttings if this is the only option. Observations of drilling conditions are also entered on the log as discussed above and in SOP18. If total depth was reached prematurely due to refusal, the cause of refusal should be noted on the boring log form.

Subsurface soil samples may be collected with a split spoon sampler or Shelby tube during drilling. The sampling will be supervised by the *Project Geologist*. Soil samples (drive samples) can be readily obtained at discrete intervals with these methods.

Soil organic vapor sampling may be conducted at discrete intervals during hollow stem auger drilling. This is done by stopping at the desired depth and driving a sample probe through the hollow stem into the soil ahead of the bit and then collecting a vapor sample. The sampling should be supervised by the *Project Geologist.*

Groundwater screening (grab) samples can be obtained at discrete intervals during drilling. One method is to auger to the bottom of the selected interval or zone and pull the auger back to the top of the interval, allowing groundwater through the open borehole. A water sample is then collected with a bailer run through the inside of the augers. Another method is to stop the augers at a selected interval or zone and advance a hydropunch sampler beyond the lead auger to retrieve a water sample.

5.2.3 Borehole Abandonment

If the borehole is to be abandoned once drilling is completed, the abandonment will follow procedures approved by the state in which the work is completed. The abandonment will be supervised by the *Project Geologist*.

5.2.4 Monitoring Well Completion

If a monitoring well is to be installed in the borehole, the well completion will follow procedures outlined in SOP 7. The well installation activities will be supervised by the *Project Geologist.*

5.3 Demobilization/Site Restoration

After drilling, sampling, well installation, or borehole abandonment is completed the hollow stem rig is rigged down and removed from the borehole location. The demobilization/site restoration will be supervised by the *Project Geologist* or appropriate designee. All debris generated by the drilling operation will be removed and appropriately disposed. The site should be cleaned (ground washed if necessary) and surface conditions restored as required. All abandoned borings should be topped off and completed and all monitoring wells should have their surface completions finished. Any remaining hazards as a result of drilling activities will be identified and appropriate barriers and markers put in place, as per the APP. All soil cuttings and fluids will be properly contained, clearly labeled, and maintained. The *Project Geologist* or appropriate designee should inspect the site to make sure that post-drilling site conditions are in compliance with project requirements.

6.0 Required Forms

Bound Field Logbook
Boring Log

SOP NO. 11
REVISION NO. 0
DATE: 2/23/21

BOREHOLE ABANDONMENT
STANDARD OPERATING PROCEDURE

1.0 Purpose

This Standard Operating Procedure (SOP) establishes guidelines and procedures for field personnel to use in the supervision of borehole or soil boring abandonment and groundwater monitoring well abandonment activities. Additional specific borehole and well abandonment procedures and requirements will be provided in the project work plans.

2.0 References

U.S. Environmental Protection Agency (EPA), 1991, *Handbook of Suggested Practices for the Design and Installation of Ground-Water Monitoring Wells,* EPA/600/4-89/034, U.S. Environmental Protection Agency, Office of Research and Development, March.

3.0 Responsibilities

3.1 The *Field Task Manager* or his/her designee is responsible for ensuring that sample collection activities are conducted in accordance with this SOP and with any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

3.2 The *Project Manager* and *Field Task Manager* responsible for periodically observing field activities and review of field generated documentation associated with this SOP. The *Project Manager* and *Field Task Manager* are also responsible for the implementation of corrective action (i.e., retraining personnel, additional review of work plans and SOPs, variances to the abandonment requirements, issuing nonconformances, etc.) if problems occur.

3.3 The *Field Technician* assigned to borehole and well abandonment activities are responsible for completing their tasks according to specifications outlined in this SOP and other appropriate procedures. All staff are responsible for reporting deviations from the procedures to the *Project Manager* or *Field Task Manager*.

4.0 Definitions

4.1 Borehole

Any hole drilled into the subsurface for the purpose of identifying lithology, collecting soil or rock samples, and/or installing groundwater wells.

4.2 Borehole Abandonment

The process whereby boreholes or soil borings are grouted or sealed following completion of drilling, sampling and/or logging.

4.3 Cuttings
Pieces of soil, sediment, or rock cut by a bit in the process of drilling borings.

4.4 Tremie
A tubular device or pipe used to place grout, bentonite, or filter pack in the annular space of a borehole.

5.0 Procedure
After drilling, logging and/or sampling, boreholes should be backfilled by the method required by the applicable regulatory agency and described in the installation-specific work plans. This typically consists of backfilling to the surface with bentonite chips, pellets or bentonite-cement grout. If bentonite chips or pellets are used, they should be added to the borehole in 2-foot lifts and hydrated with water from a potable water supply. This process should be repeated until the entire borehole is plugged using no less than 5 gallons water per ten feet of borehole. If bentonite grout is used the following guidelines should be followed:

- Bentonite should be thoroughly mixed into the grout and within the percentage range specified in the work plans. If not otherwise specified in the work plans, the cement-bentonite grout mixture should be of the following proportions: 94 pounds of Portland cement, 5 pounds of powdered bentonite and a maximum of 8 gallons of water. The grout is usually tremied into the hole; however, for selected boreholes (e.g., shallow borings well above the water table) at certain sites, the grout may be allowed to free fall. In either case, care must be taken to ensure the grout does not bridge, forming gaps or voids in the grout column.
- The volume of the borehole prior to sealing should be calculated and compared to the grout volume used during abandonment to aid in verifying that bridging did not occur.
- When using a tremie pipe to place grout in the borehole, the bottom of the tremie should be submerged into the grout column and withdrawn slowly as the hole fills with grout. If allowing the grout to free fall (and not using a tremie), the grout should be poured slowly into the boring. The rise of the grout column should also be visually monitored or sounded with a weighted tape.
- If the method used to drill the boring utilized a drive casing, the casing should be slowly extracted during grouting such that the bottom of the casing does not come above the top of the grout column.
- During the grouting process, the drilling hands performing the task should be supervised to assure that potentially contaminating material (oil, grease, or fuels from gloves, pumps, hoses, et. al) does not enter the grout mix and that personnel are properly wearing personal protective equipment as specified in the project Health and Safety Plan.
- Following grouting, barriers should be placed over grouted boreholes as the grout is likely to settle in time, creating a physical hazard. Grouted boreholes will typically require at least a second visit to “top off” the hole.
- The surface hole condition should match the pre-drilling condition (asphalt, concrete, or smoothed flush with native surface), unless otherwise specified in the installation-specific work plans.

6.0 Required Forms

Bound Field Logbook

SOP NO. 14
REVISION NO. 0
DATE: 2/23/21

EQUIPMENT DECONTAMINATION
STANDARD OPERATING PROCEDURE

1.0 Purpose

This Standard Operating Procedure (SOP) establishes guidelines for use by field personnel in the decontamination of sample equipment. The details within this SOP are applicable as general requirements for sample equipment decontamination.

2.0 References

None

3.0 Responsibilities

3.1 The *Field Task Manager* has the responsibility for ensuring that the decontamination of sample equipment is properly performed through staff training and by maintaining quality assurance/quality control (QA/QC). The *Field Task Manager* has the responsibility for periodic review of procedures and documentation associated with the decontamination of sample equipment. If perceived variances occur, the CQCC is also responsible for issuing notices of nonconformances and requesting corrective actions.

3.2 The *Field Technician* assigned to sample activities is responsible for gathering rinsate blank samples from the contractor's field sampling equipment to demonstrate that the equipment is being properly decontaminated. The project staff are also responsible for documenting the decontamination activities on the appropriate form(s).

4.0 Definitions/Materials

4.1 Laboratory Grade Detergent - A standard brand of laboratory-grade detergent, such as "Alconox" or "Liquinox".

4.2 Potable Water - Water dispensed from a municipal water system.

4.3 Distilled or Deionized Water
Ion-free, analyte-free water produced on site or purchased from a supplier.

4.4 Non-sampling Equipment
Non-sampling equipment includes:

- Field logbook
- DPT equipment
- High-pressure pump soap dispenser or steam-spray unit
- Manual-pump sprayer (pump sprayer material must be compatible with the solution used)

- Gloves, goggles, boots, and other protective clothing as specified in the site-specific Accident Prevention Plan (APP).

4.5 Small Equipment
Small equipment includes:

- 5-gal plastic buckets
- Stiff-bristle brushes
- Nalgene**,** or Teflon**,** sprayers or wash bottles or 2- to 5-gal manual-pump sprayer (pump sprayer material must be compatible with the solution used).

5.0 Procedure
5.1 General
This section contains responsibilities, requirements, and procedures for sampling equipment decontamination. The decontamination is required to maintain proper quality and integrity of collected samples. The details within this SOP should be used in conjunction with the project specific Uniform Federal Procedures - Quality Assurance Project Plan (UFP-QAPP).

5.2 This section provides requirements for the set up of a decontamination facility for DPT equipment and the decontamination procedures to be followed. Site conditions and specific equipment used on site will determine the following:

- Types of equipment requiring decontamination under this SOP;
- Location of the decontamination station;
- Types and/or specifications on materials to be used in the fabrication of the decontamination station; and
- Types of materials and additional details on the procedures to be used in the decontamination process.

Field personnel associated with either the fabrication of the decontamination station or the decontamination of sample equipment must read this SOP prior to implementation of related decontamination activities.

5.2.1 Decontamination Facility
A decontamination station will be set up in an area exclusively for decontamination of different sample equipment. The location of the decontamination station will be determined in the field. All decontamination of sample equipment will be conducted within the appropriate station.

At a minimum, the station will be constructed such that all rinsates, liquid spray, soil, debris, and other decontamination wastes are fully contained and may be collected for appropriate waste management and disposal. The station may be as simple as a bermed, impermeable polyethylene sheeting, of sufficient thickness, with an impermeable sump for collecting rinse water. More sophisticated designs involving self-contained metal decontamination pads in combination with bermed polyethylene sheeting may also be used, depending on project-specific requirements.

5.2.2 Decontamination of Downhole Equipment

All downhole DPT equipment will be thoroughly decontaminated before mobilization onto site and between borings. The standard procedure will be performed as described below. Decontamination will be performed in accordance with this SOP.

Appropriate personal protective equipment (as specified in the Health and Safety Plan [HASP]) must be worn by all personnel involved with the task to limit personal exposure.

Equipment caked with soil, or other material will initially be scraped or brushed. The scrapings will be containerized and appropriately disposed.

Equipment will then be sprayed with potable water using a hot water, high pressure washer.

Washed equipment will then be rinsed with potable water.

Decontaminated downhole equipment (such as rods, probe-driven sampler, etc.) will be placed on clean plastic sheeting to prevent contact with contaminated soil and allowed to air dry. If equipment is not used immediately, it will be covered or wrapped in plastic sheeting to minimize airborne contamination.

Decontamination activities will be documented by the *Field Technician* on the appropriate form(s).

5.2.3 Decontamination of DPT Equipment

DPT equipment will be decontaminated between sample sites. The standard procedure will be performed as described below.

Appropriate personal protective equipment (as specified in the HASP) will be worn by all personnel involved in the task, in order to limit personal exposure.

Equipment caked with soil, or other material will be initially scraped or brushed. The scrapings will be containerized and appropriately disposed.

Equipment will then be sprayed with potable water using a hot water, high pressure washer.

Clean equipment will then be rinsed with potable water.

During the decontamination effort, fluid systems should be inspected for any leaks or problems which might potentially result in an inadvertent release at the site, thereby contributing to the volume of waste or contamination. Any identified problems should be immediately repaired and documented in the bound field logbook. Decontamination should then be completed before moving the equipment onto the site or next sample site.

Decontamination activities will be documented by the *Field Technician* on the appropriate form(s).

Between boreholes, the back-end of the DPT equipment will be washed with potable water until surfaces are visibly free of soil buildup.

5.3 Decontamination of Non-dedicated Sampling Equipment
Each piece of reusable, small or nondedicated sampling equipment will be decontaminated before the start of work at each location. The standard procedure will be performed as described below.

Suitable personal protective equipment (specified by the APP) must be worn by all personnel involved with the task to reduce personal exposure.

Heavily caked soil and/or other material will be scraped or brushed from equipment. The scrapings will be placed into an appropriate container for disposal. High pressure or steam cleaning of equipment may be required to remove material.

Equipment that will not be damaged by water should be placed into a wash tub containing a laboratory-grade detergent solution and potable water and scrubbed with a brush or clean cloth. Rinsing will then be conducted with fresh, potable water, followed by deionized water.

Any equipment that may be damaged by submersion into water will be wiped clean using a sponge and detergent solution. Cleaning will be followed by wiping the equipment with deionized water.

Place decontaminated equipment on clean plastic sheeting or in a plastic bag to prevent contact with contaminated soil. If equipment is not used immediately, cover or wrap the equipment to minimize airborne contamination.

5.4 Waste Disposal
All wash water and rinse water that has come in contact with contaminated equipment is to be handled, packaged, labeled, marked, stored, and disposed of as investigation derived waste unless other arrangements are approved in advance.

Small quantities of decontamination solutions may be allowed to evaporate to dryness.

Unless required, plastic sheeting and disposable protective clothing may be treated as a solid nonhazardous waste.

6.0 Required Forms
Bound Field Logbook.

SOP NO. 15
REVISION NO. 0
DATE: 2/23/21

FIELD EQUIPMENT DECONTAMINATION STANDARD OPERATING PROCEDURE

1.0 Purpose

This Standard Operating Procedure (SOP) describes the procedures required for decontamination of field equipment. Decontamination of field equipment is necessary to ensure the quality of samples by preventing cross-contamination. Further, decontamination reduces health hazards and prevents the spread of contaminants off site.

2.0 References

2.1 EPA, September 1987, EPA Compendium of Superfund Field Operations Methods, EPA 540/P-87/001a, OSWER 9355.0-14.

2.2 EPA, August 1988, EPA Guidelines for Conducting Remedial Investigation and Feasibility Studies under CERCLA, Interim Final OSWER Directive 9355.3-01.

3.0 Responsibilities

3.1 The *Field Manager* and *Subcontractor Field Leads* are responsible for ensuring that all sample collection activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

The sampling team may be required to collect and document rinsate samples to provide quantitative verification that decontamination procedures have been correctly implemented.

4.0 Definitions/Materials

4.1 Distilled or Deionized Water
Ion-free, analyte-free water produced on site or purchased from a supplier.

4.2 Potable Water
Treated municipal water.

4.3 Laboratory Grade Detergent
A standard brand of laboratory-grade detergent, such as Alconox or Liquinox.

4.4 Non-sampling Equipment
Non-sampling equipment includes:

- Field logbook
- Drilling rigs, backhoes, augers, drill pipe, bits, well casing, and well screen.
- High-pressure pump soap dispenser or steam-spray unit

- Manual-pump sprayer (pump sprayer material must be compatible with the solution used)
- Gloves, goggles, boots, and other protective clothing as specified in the site-specific Accident Prevention Plan (APP).

4.8 Small Equipment
Small equipment includes:

- Split spoons, bailers, spoons, and bowls.
- 5-gal plastic buckets
- Stiff-bristle brushes
- Nalgene**,** or Teflon**,** sprayers or wash bottles or 2- to 5-gal manual-pump sprayer (pump sprayer material must be compatible with the solution used).

5.0 Procedures

- This section contains responsibilities, requirements, and procedures for sampling equipment and well material decontamination. The decontamination is required to maintain proper quality and integrity of collected samples. The details within this SOP should be used in conjunction with the project specific Uniform Federal Procedures Quality Assurance Project Plan (UFP-QAPP) or field sampling plan.

All field personnel associated with decontamination of sampling equipment or well materials must read both this SOP prior to implementation of related decontamination activities.

5.1 Decontamination Facility
Decontamination of large equipment will take place in an area designed exclusively for decontamination. This area should be approved by the client prior to field work commencing.

The large equipment decontamination facility will be constructed so that the equipment, as well as all wastes generated during decontamination (e.g.: soil, rinsate, liquid spray, debris, etc.), are fully contained. In addition, chemical products used in the decontamination process must be properly containerized and labeled.

5.2 Decontamination of Non-dedicated Sampling Equipment
Each piece of reusable, small or nondedicated sampling equipment will be decontaminated before the start of work at each location. The standard procedure will be performed as described below.

Suitable personal protective equipment (specified by the APP) must be worn by all personnel involved with the task to reduce personal exposure.

Heavily caked soil and/or other material will be scraped or brushed from equipment. The scrapings will be placed into an appropriate container for disposal. High pressure or steam cleaning of equipment may be required to remove material.

Equipment that will not be damaged by water should be placed into a wash tub containing a laboratory-grade detergent solution and potable water and scrubbed with a brush or clean cloth. Rinsing will then be conducted with fresh, potable water, followed by deionized water.

Any equipment that may be damaged by submersion into water will be wiped clean using a sponge and detergent solution. Cleaning will be followed by wiping the equipment with deionized water.

Place decontaminated equipment on clean plastic sheeting or in a plastic bag to prevent contact with contaminated soil. If equipment is not used immediately, cover or wrap the equipment to minimize airborne contamination.

5.3 Decontamination of Well Materials

Well materials including well casing, well screens, centralizers, and end caps will be decontaminated prior to use in constructing monitoring wells. (If factory-cleaned, hermetically sealed materials are used, no decontamination will be necessary provided that laboratory decontamination certification is submitted with the equipment.) The standard procedure outlined below must be performed when decontaminating well materials.

Appropriate personal protective equipment will be worn by all personnel involved in the task, in accordance with the APP.

Materials will be thoroughly sprayed and washed with water using a high-pressure steam cleaner.

Air dry.

Decontaminated materials will be placed on clean metal racks or clean plastic sheeting. If equipment is not used immediately, cover or wrap the equipment in clean plastic sheeting to minimize airborne contamination.

5.4 Pump Decontamination

The following steps must be followed when decontaminating pumps:

Set up decontamination area and separate clean storage area. Set up three 10-plus gallon containers, one with dilute soapy water (initial wash), one with potable water (second wash), and a third with deionized water (final wash).

Pump should be set up in the same configuration as for sampling, unless the pump can be broken down easily into its component parts (like with a non-dedicated bladder pump where dedicated tubing is used). Submerge pump and all downhole wetted parts (tubing, piping, foot valve) in soapy water of the first container. Place the discharge outlet into an

investigation-derived waster (IDW) water container or well purge water bucket. Pump soapy water through the pump assembly until it discharges to the waste container.

Move pump assembly to the potable water container while leaving discharge outlet in the waste container. All downhole wetted parts must be immersed in the potable water rinse. Pump potable water through the pump assembly until it runs clear.

Move pump assembly to the deionized water container and move the discharge outlet to the potable water container. Pump at least one pump and tubing volume of deionized water through the system. Decontaminate the discharge outlet by hand using the soapy water, then the potable water, then the deionized water.

Record the decontamination in the appropriate bound field logbook.

5.5 Waste Disposal
All wash water and rinse water that has come in contact with contaminated equipment is to be handled, packaged, labeled, marked, stored, and disposed of as IDW unless other arrangements are approved in advance.

Small quantities of decontamination solutions may be allowed to evaporate to dryness.

Unless required, plastic sheeting and disposable protective clothing may be treated as a solid nonhazardous waste.

6.0 Required Forms
Bound field logbook

SOP NO. 16
REVISION NO. 0
DATE: 2/23/21

MANAGEMENT OF INVESTIGATION-DERIVED WASTE STANDARD OPERATING PROCEDURE

1.0 Objective

Investigation-derived waste (IDW) covered by this SOP include wastes generated from the site investigation. Evaluation of waste performed using this SOP is to determine the proper non-hazardous or hazardous waste characterization and ensure the waste is disposed of consistent with applicable solid and hazardous waste regulations. Prior to disposal, the wastes will be collected, transferred and stored in accordance with applicable regulations.

2*.0 Background*

Environmental investigations and site remediation may generate potentially contaminated IDW, including but not limited to the following types of materials:

- Saturated and unsaturated soil;
- Groundwater;
- Remediation waste;
- Decontamination water; and
- Personal Protective Equipment (PPE) and miscellaneous refuse.

3.0 References

None.

4.0 Responsibilities

Investigation Contractor - This is any contractor completing an environmental investigation. Examples of such investigations would include Resource Conservation and Recovery Act (RCRA) Facility Investigations (RFIs), Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) assessments and investigations, and groundwater monitoring programs under the direction of either EPA or state agencies.

The collection, on-site storage, transportation and disposal of solid and hazardous waste generated from the site investigation will be completed in accordance with applicable regulations. This includes the proper containerization and labeling of wastes; safe movement of waste containers; characterization of the waste; temporary storage of the waste; and the preparation of the hazardous waste manifests and the notifications required under the 40 CFR Part 268 Land Disposal Restrictions (LDRs).

Unless the waste has been previously characterized or can be identified using generator

knowledge, IDW containers will be sampled within 30 days of being filled. The IDW sample will be analyzed, as discussed in the paragraphs below, and characterized for proper disposal. The waste will then be disposed of accordingly based on waste characterization.

5.0 Procedures

5.1 Waste Minimization Procedures

To the extent that it is practical, waste minimization procedures will be followed during environmental investigations. Guidelines for waste minimization are:

- Minimize materials which are introduced into any exclusion zone in an investigation area.
- Combine similar wastes throughout an investigation area in a single container wherever possible.
- Combine decontamination water from multiple sites in one container provided the contaminants are reasonably expected to be from the same source or are of the same character.
- Use a container of the appropriate size (e.g., use a 5-gallon drum for small amounts of waste unless a 55-gallon drum is needed to hold all the waste).
- Decontaminate and reuse material and equipment whenever practical. Minimize the volume of decontamination water generated.
- With solid environmental media and materials, ensure that waste is tightly packed to minimize the number of containers.
- Use materials with the least possible hazard as possible.

5.2 Waste Containerization and Labeling

Investigation or remediation derived waste, which are known or suspected to be hazardous waste, will be placed in appropriate Department of Transportation (DOT) containers and labeled as hazardous waste. Labels for shipping waste on public roads will be used to properly label and track containers. Upon filling of a container, the start accumulation date will be annotated on the label and the container will be transferred off-site or to the 90-day storage facility within 3 days.

5.3 Waste Management and Sampling Procedures

Wastes are either managed in open-top or closed-top drums, roll-off containers, or in polyethylene tanks. In some instances liquid waste may be discharged to a bulk tanker for transport to a Treatment Storage and Disposal Facility (TSDF).

The sampling method selected for a given waste stream is based on the physical properties the waste exhibits. Liquids will be sampled with a scoop, bailer, or pump; solids will be sampled with a trowel or auger. Equivalent sampling equipment may be used provided it is suitable for obtaining a representative sample of all phases of the waste.

Each sample will be taken using a sampling tool(s) that will ensure the most representative sample. Multi-phase wastes may require that more than one sample be

collected from each container to adequately represent each distinct phase. When more than one container is represented by an IDW sample, the sample to be analyzed will be a composite sample comprised of proportional amounts taken from all the related waste containers. For example, the drill cuttings from a single well would be composited into one sample. However, if information pertaining to the waste stream indicates the contamination may vary significantly, then composites would only be utilized for those portions of the waste stream with similar characteristics or each container should be sampled separately.

5.4. Waste Characterization

Unless the waste has been previously characterized or the waste can be adequately characterized using generator knowledge, all containers will be sampled within 30 days of being filled. Analysis will occur at a DoD-accredited laboratory. The sample will be analyzed, as discussed in the paragraphs below, and characterized for proper disposal per the requirements in the project specific work plan. The waste will then be disposed of accordingly per the project specific work plan. The characterization will be used to prepare a disposal profile for disposal at an appropriately licensed disposal facility.

5.5 Parameter Test Methods

The type of analysis of each waste will depend upon the operations previously conducted at the site and information gained from previous investigative or remedial work performed. The analytical parameters that normally will be considered include the characteristics of Ignitability, Corrosivity, Reactivity, Toxicity Characteristic Leaching Procedure (TCLP) Metals, TCLP Pesticides/Herbicides and TCLP Volatile Organic Compounds (VOCs). Parameters for F- and K-listed hazardous wastes will only be analyzed for if information specific to the site indicates their possible presence. Total analyses may be used in lieu of TCLP extraction methods for solid samples at the contractor's discretion.

In addition to waste characterization parameters, analysis may also be required to satisfy 40 CFR 262.34 obligations for determining whether Subpart CC air emissions requirements apply to waste containers while in 90 day storage. For this purpose, analysis for total VOCs is appropriate. Such analysis is not applicable to wastes not reasonably expected to contain significant VOC concentrations.

Parameters will be eliminated when previously-gathered information for a site or the physical state of the waste generated would so justify. The EPA waste codes and the applicable SW-846 analytical method(s) will be determined.

5.6 Off-Site Transportation and Disposal of Hazardous Waste

Prior to transporting or offering a container of hazardous waste for transport off-site, each container must be labeled in accordance with DOT regulations for hazardous materials under 49 CFR Part 172. In addition, each container of 110 gallons or less must be labeled with the following words and displayed in accordance with 49 CFR 172.304:

HAZARDOUS WASTE Federal Law Prohibits Improper Disposal. If found, contact

the nearest police or public safety authority or the U.S. Environmental Protection Agency.

Generator's Name and Address _____.

Manifest Document Number _____.

Any hazardous wastes from the project site to a disposal facility will be accompanied with a uniform hazardous waste manifest prepared by the Project Manager or his/her properly trained designee in accordance with 40 CFR 262.20. Acquisition, copies and use of the manifest will be in accordance with 40 CFR 262.21 through 23. A client representative will sign the manifest as the generator.

Wastes not meeting the criteria of hazardous waste will be managed as non-hazardous materials as appropriate in accordance with 49 CFR Part 172. Containers of a capacity <110 gallons (non-bulk) will be appropriately labeled and marked and a non-hazardous materials shipper/bill of lading will be prepared and signed by a client representative. Containers with a capacity of 110 gallons (882 lbs) or greater will be placarded during storage/transportation. Trucks used to transport bulk non-hazardous materials or non-hazardous waste will also be placarded if at least 1,001 lbs gross weight of non-hazardous materials are loaded.

SOP NO. 17
REVISION NO. 0
DATE: 2/23/21

SAMPLE HANDLING AND CUSTODY STANDARD OPERATING PROCEDURE

1.0 Purpose

This Standard Operating Procedure (SOP) outlines the methods and responsibilities for field personnel to use in the packaging and shipping of environmental samples for chemical and physical analysis, and the maintenance and custody of environmental samples which are to be used to provide data which form a basis for making project related decisions. This SOP only applies to the packaging and shipping of limited quantity, low concentration environmental samples, and the general procedures for maintaining and documenting sample chain of custody (COC) from the time of sample collection through sample disposal. This procedure does not apply to those samples considered hazardous materials, hazardous waste, mixed waste, radioactive waste, and/or dangerous goods, which are specified in the Department of Transportation (DOT) 49 CFR 114-327 and International Air Transport Association (IATA) procedures. The details within this SOP are only applicable to the general requirements for sample packaging and shipping.

2.0 References

2.1 Code of Federal Regulations (CFR), 40 CFR Chapter I Subchapter I Part 261 Subpart C, 40 CFR Chapter I Subchapter I Part 261 Subpart D, 40 CFR Chapter I Subchapter I Part 262, and DOT 49 CFR Subtitle B Chapter I Subchapter A Parts 100 to 177. (http://www.ecfr.gov, accessed 21 March 2019).

3.0 Responsibilities

3.1 The *Field Task Manager* is responsible for ensuring that all sample collection activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC).

3.2 The *Project Chemist* is responsible for assuring proper COC is initiated at the time the sample(s) are collected and maintained throughout the sample handling, packing, shipping, subsequent transportation of the sample(s) to the designated laboratory and maintain QA/QC. Additionally, he/she is the project authority for determining the disposition and fate of sample(s) which have identified deficiencies (e.g., missed holding times, elevated temperature at receipt, etc.). The *Project Chemist* is responsible for periodic review of COC records and documentation generated in association with this SOP. The *Project Chemist* is also responsible for implementation of corrective action (i.e., retraining personnel, additional review of SOPs, variances to quality control (QC) sampling requirements, issuing nonconformances, etc.) if problems occur.

3.4 The *Field Technician* is responsible for properly documenting and maintaining the COC from the time of sample collection until the sample is delivered to the lab.

3.5 *Laboratory Personnel* are responsible for receipt and log in of samples into the laboratory which have been submitted under a COC document. Additionally, samples received will be entered into the laboratory COC procedures by properly documenting and maintaining COC from the moment that they take custody of the sample at the laboratory until the sample is disposed of or returned to the client.

4.0 Definitions/Materials

4.1 Chain of Custody

The COC documents are the written records that trace the sample possession from the time each sample is collected until its final disposal, sometimes called the "cradle to grave" record. COC is maintained by compliance with one of the following criteria:

- The sample is in the individual's physical possession;
- The sample is maintained in the individual's physical view after being in his/her possession;
- The sample is transferred to a designated secure area restricted to authorized personnel;
- The sample is sealed and maintained under lock and key to prevent tampering, after having been in physical possession.

4.2 Waybill

A document that contains a list of the goods and shipping instructions relative to a shipment.

4.3 Common Carrier

For the purpose of this procedure, the common carrier is any commercial carrier utilized for the transportation of the sample(s) from the field to the laboratory.

4.4 Environmental Sample

A limited quantity, low concentration sample that does not require DOT or IATA hazardous waste labeling as a hazardous waste or material.

4.5 Hazardous Waste Sample

Medium or high concentration samples requiring either DOT or IATA labeling as a hazardous waste or material.

4.3 Hazardous Waste

Any substance listed in 40 CFR Part 261 Subpart D or otherwise characterized as ignitable, corrosive, reactive, or toxic as specified in 40 CFR Part 261 Subpart C that would be subject to manifest and packaging requirements specified in 40 CFR 262. Hazardous waste is defined and regulated by the U.S. Environmental Protection Agency (USEPA).

4.4 Hazardous Material
A substance or material in a quantity or form which may pose an unreasonable risk to health, safety, and/or property when transported in commerce. Hazardous material is defined and regulated by DOT (49 CFR 173.2 and 172.101) and IATA.

4.5 Sample
Physical evidence collected from a facility or the environment which is representative of conditions at the point and time at which the sample is collected.

5.0 Procedure
5.1 General COC
An overriding consideration for data resulting from laboratory analyses is the ability to demonstrate that the samples were obtained from the locations stated and that they reached the laboratory without alteration. Evidence of collection, shipment, laboratory receipt, and laboratory custody until disposal must be documented to accomplish this.

Documentation will be accomplished through a COC form that lists each sample and the individuals performing the sample collection, shipment, and receipt. The COC document is a preprinted form. The original COC form will accompany the samples to the laboratory and a copy will be retained in the field project file.

5.2 Field Sample Custody
Sampling personnel, upon collection of samples for analysis, will properly complete a COC form. The COC form will be the controlling document to assure that sample maintenance and custody are maintained thereby assuring the sample(s) are representative of the environment from which they were collected. At a minimum, the following information will be recorded on the COC form:

- The unique identification number assigned to each sample;
- The sample matrix (e.g., water, soil, waste);
- The date and time of the sample collection;
- The total number of containers associated with each sample;
- Preservatives added to the samples;
- Requested analyses by method number;
- Special instructions to the laboratory including handling requirements, quality assurance/quality control, health and safety, and sample disposition;
- The project name and number;
- The names of all sampling personnel;
- The name of the project contact; and
- A unique COC form reference number.

The COC document will be initiated in the field by the person(s) collecting the sample and signed by each individual who has had the samples in their possession. Each time that sample custody is transferred, the former custodian must sign over the COC as

Relinquished By, and the new custodian must sign on to the COC as Received By. Each signature must be accompanied by the date and time.

If the sampling personnel deliver the samples to the laboratory, transfer of COC occurs as follows:

- The sample collector delivers the samples to the laboratory and relinquishes the sample directly to a laboratory representative.
- The collector signs the COC listing his/her name, the date, and time. Any person involved in the collection of the sample may act as the sample custodian.
- The laboratory representative must receive the samples by signing his/her name, the date, and time on the COC. The laboratory representative may decline to take receipt of the samples if the COC is not properly completed or if the samples are not properly packaged. All designated *Laboratory Personnel* may act as the sample custodian.
- One copy of the COC is given to the sample collector to be returned to the project files and one copy is maintained with the samples at the laboratory.

If the sampling personnel transfer sample(s) to the laboratory utilizing a common carrier, sampling personnel will retain COC responsibility and the common carrier is not responsible for maintaining sample custody. The sample collectors are responsible for packaging the samples in a manner that meets the COC definition criteria, that is, the samples are sealed to prevent tampering.

When transferring samples to the courier for transport, COC procedures are maintained as follows:

- The sample collector lists the courier affiliation and waybill number on the COC.
- The sample collector relinquishes custody by signing his name, date, and time. The collector keeps a copy or photograph of the relinquished COC for the project file.
- The relinquished original COC is sealed in a watertight plastic bag and taped to the inside of the lid of the container used for transportation.
- The transportation container is sealed to prevent tampering and given to the courier for delivery to the laboratory.
- The laboratory representative must receive the samples by signing his/her name, the date, and time on the COC. This copy is maintained with the samples at the laboratory.

5.3 Analytical Laboratory Custody

Upon receipt at the analytical laboratory, the field-generated COC document will be signed, dated, time marked, temperature marked, and laboratory identification will be provided in the appropriate spaces. The laboratory will complete a cooler receipt form documenting the condition of the samples upon receipt.

Laboratory receipt personnel will enter the samples into the laboratory by implementing the sample custody procedures addressed within their approved sample log-in SOP.

After completion of analytical testing, sample remnants not consumed during testing may be kept for up to six months beyond the completion of analysis, unless otherwise specified by a notation on the COC that samples are to be returned to the project site for disposal. Once this time period has elapsed, the samples will be disposed of and the disposal record number will be recorded in the laboratory documentation of the samples.

5.4 Sample Handling
5.4.1 Inspect the sampling containers (obtained from the analytical laboratory prior to the sampling event) to ensure that they are appropriate for the samples being collected, correctly preserved, and undamaged.

5.4.2 When collecting a sample always use approved/site specific personal protective equipment (PPE) (e.g., gloves) to prevent cross-contamination from sample to sample but also as a health and safety requirement.

5.4.3.2 Sediment samples will be visually inspected for NAPL prior to packaging in laboratory provided glass jars (see SOP 12). The field task manager will look for color changes, blebs, globs, sheen, staining, hardened tar, and other visible product. Petroleum odors will also be considered indicative of NAPL.

5.5 Field Packaging
5.5.1 Collect the samples in accordance with the site-specific work plans and applicable SOPs.

5.5.2 Place all containers in separate, appropriately sized, airtight, seam-sealing plastic bags (e.g., Ziploc^{TM} or equivalent). Seal the bag, pushing out any excess air.

5.5.3 Place the bagged container inside a cooler. This cooler should have ice inside to assure samples remain cool, 4°C ± 2°C, during transit from field to the packaging location.

5.5.4 Maintain the samples under chain of custody (COC) in accordance with the site-specific work plans.

5.6 Sample Packaging
5.6.1 Inspect the integrity of the shipping container. The container is generally a cooler constructed of heavy plastic or metal with appropriate insulating properties so that variations in temperature during shipping are minimized. Do not use severely damaged (e.g., cracked) or dirty coolers for storing or shipping samples.
5.6.2 Carefully check the COC form against the collected sample labels and containers to ensure that the sample identifications, date and time of collection, number of containers, preservative, and the required analytical methods are correct and in agreement.

5.6.3 Pack the cooler with samples, placing ice below, on top of and between samples. Blue ice should not be used for sample shipping; it does not maintain the 4°C temperature necessary for regulatory compliance. If the cooler does not contain plastic bottles, include a plastic bottle of tap water labeled "temperature blank" so that the laboratory can verify the temperature of the samples upon receipt. The remaining space will be filled with packing material.

5.6.4 All samples requiring temperature preservation at 4°C will be acceptable within the range of 4°C ± 2°C. The laboratory should record the temperature of receipt upon the COC and/or Laboratory Cooler Receipt Form. For all samples received at less than 2°C (note if frozen), or at greater than 6°C, the sample(s) and temperature will be identified on the COC and the *Project Chemist* notified, to determine whether to proceed with sample analysis.

5.7 Sample Shipping

5.7.1 The laboratory will be contacted at least two weeks prior to sample shipments. Sample deliveries on weekends and holidays will be confirmed in advance. The person in charge of sample custody will relinquish the samples on the COC form. When a common carrier is to be used for sample shipment, record the waybill number (tracking number) and the name of the carrier on the COC form. Place the original copy of the COC form in a sealed, clear plastic bag and tape the bag to the inside lid of the shipping container. Retain a hard copy or photograph of the COC form for tracking purposes.

5.7.2 Using nylon-reinforced strapping tape or mailing tape, seal the cooler.

5.7.3 Place signed and dated custody seals over opposite ends of the cooler lid.

5.7.4 Attach the waybill to the outside of the cooler, preferably using an adhesive tag attached to the cooler handle, as this is less likely to fall off during transport of the cooler.

5.7.5 Turn the sample over to the carrier for delivery to the laboratory. All samples should be shipped for next morning delivery.

NOTE: The courier or carrier is not responsible for sample custody and is not required to sign the COC.

5.7.6 Contact the appropriate laboratory personnel to advise them of the sample shipment.

5.7.7 The *Field Task Manager* or their designee should track the cooler shipment to ensure it reaches its destination and follow up the next day by calling the carrier to verify on-time delivery of all coolers.

6.0 Required Forms

Chain of Custody Record
Laboratory Cooler Receipt Form

FIELD DOCUMENTATION
STANDARD OPERATING PROCEDURE

1.0 Purpose

The purpose of this Standard Operating Procedure (SOP) is to define the minimum requirements for documenting field activities in the field logbooks. Field logbooks provide a detailed daily handwritten record, kept in real time, of field activities performed at an investigation site. Logbooks are permanently bound by glue or thread into a hard cover. Field logbooks may be assigned to specific activities, positions, or areas within the site. Field logbook covers must be numbered and indicate the position, task, activity, or area assigned to the logbook.

2.0 References

None.

3.0 Responsibilities

3.1 The *Field Task Manager* is responsible for ensuring that all sample collection activities are conducted in accordance with this SOP and any other appropriate procedures. This will be accomplished through staff training and by maintaining quality assurance/quality control (QA/QC). The *Field Task Manager* is also responsible for ensuring that field logbooks are completed daily in accordance with this procedure, and for implementation of corrective action (i.e., retraining personnel, additional review of SOPs, reporting nonconformance to the *Project Manager*) if problems occur.

3.2 The *Field Technician* is responsible for making timely and complete entries in the field logbook per this SOP.

4.0 Definitions/Material

4.1 Field logbook

A bound and ruled/gridded surveyor's book or field book with sequentially-numbered pages.

4.2 Waterproof black ink pens.

5.0 Procedures

5.1 Field Logbook Cover

Label the front cover of the field logbook with the project name and number, client name, the start date and, when complete, the finish date. The field logbooks must have an identifying number on the cover.

5.2 Field Logbook

The following steps must be followed when making entries in the field logbook:

1. Enter the date; time the task started; weather conditions; and the names and organizations of personnel performing the task.

2. Record the name and organization, time of arrival, and time of departure of all visitors to the work area.

3. Describe all site activities in specific detail or indicate which SOPs were followed or which forms were used to record such information (e.g., soil boring log).

4. Describe in specific detail any field tests that were conducted, and instruments used. Reference any forms that were used, other data records, and the SOPs or other procedures followed in conducting the test.

5. Changes in procedures or sample locations and reasons for change.

6. Describe in specific detail any samples collected and whether splits, duplicates, matrix spikes or blanks were prepared.

7. Upgrades or downgrades of personal protective equipment (PPE) and the rationale for such action, and health and safety information such as level of PPE used.

8. List the time, equipment type, and the SOP followed for all decontamination carried out.

9. List all equipment calibrations, the details of the calibration (e.g., initial pre-calibration readings, standards used, and post-calibration readings), and the person(s) performing calibration, either in the field logbook or on an equipment calibration form.

10. Record all photographs in the logbook and include a description of the subject, the direction the photographer is facing, and the photographer's initials. If the event photographed is the collection of a sample, record the sample ID number.

11. List any equipment failures or breakdowns that occurred, together with a brief description of repairs or replacements.

12. No pages may be removed from the field logbooks for any reason. Blank pages must be marked "page intentionally left blank".

13. Mistakes must be crossed out with a single line, initialed, and dated. Only persons authorized by the *Field Task Manager* may make entries in logbooks.

14. An authorized person present for the field activity must sign the field logbook at the bottom of each page.

15. The field logbook and any other additional field documentation will be shared with entities established at the beginning of the project. These items will be shared at least weekly, if not more frequently. Frequency will be determined by the *Project Manager* at the beginning of the project.

6.0 Required Forms

Bound Field Logbook
Soil Boring Log Form

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APPENDIX C

LABORATORY STANDARD OPERATING PROCEDURES

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Title: Acid Digestion of Aqueous Samples for Analysis by ICP-MS [EPA 200.8 and SW-846 3005A, 3020A, and 3050B]

Approvals (Signature/Date):

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Technical Director

Facility Distribution No. _____ Distributed To: _____

1.0 Scope and Application

1.1 This procedure describes the preparation of aqueous samples for the analysis of metals by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) using EPA Method 200.8 and SW-846 Methods 3005A and 3020A.

1.2 Aqueous samples also include TCLP & SPLP Leachates and aqueous equipment rinse blanks associated with soil sampling. In some cases, where the associated soil samples require the SW-846 Method 3050B, Section 7.5, optional treatment to improve solubility and recovery of Sb, Ag, and Sn, the client may require that the aqueous equipment blank receive the same treatment. Refer to section 10.14 for this prep.

1.3 The applicability of each of these preparation protocols to specific analytes is detailed in the Eurofins TestAmerica (ETA) LIMS System (TALS). Additional elements may be analyzed following digestion by these protocols, provided that the method performance criteria specified in Section 12.0 of this SOP are met.

1.4 This SOP provides procedures applicable to the preparation of dissolved, total recoverable, potentially dissolved, and total metallic elements in ground water, aqueous samples, aqueous sludges, aqueous wastes, aqueous air sampling media, and leachates/extracts. This SOP is not applicable to samples that contain or consist of oil or other immiscible organic solvents.

NOTE: Samples that are known to be immiscible with water, e.g., contain or consist of oil or other immiscible organic solvents should be logged with a waste matrix and subbed out to a different Eurofins TestAmerica laboratory for digestion and analysis.

1.5 SW-846 Method 3005A is used to prepare surface and groundwater samples for total recoverable and dissolved metals determination by ICP-MS. Although digestion is not specifically required by the method (SW-846 3005A, Section 2.2) for dissolved samples, the standard operating procedure at ETA Denver is for all matrices to be digested prior to analysis.

1.6 EPA Method 200.8 Section 11.2 is used to prepare surface water, drinking water, and domestic and industrial waste samples for total recoverable and dissolved metals.

1.7 SW-846 Method 3020A is used to prepare aqueous samples, TCLP leachates, SPLP leachates and aqueous wastes that contain suspended solids for total metals analysis by ICP-MS.

1.8 The following table lists the sample preparation methods that are covered in this SOP and the specific section of this SOP for each preparation method. Prepared samples are analyzed by inductively coupled plasma-mass spectrometry (ICP-MS).

PREPARATION METHOD	SOP SECTION	DETERMINATIVE METHOD	ANALYTICAL SOPS #
Method 3020A (Total)	10.11	ICP-MS	DV-MT-0018 DV-MT-0022
Method 3005A (Total Rec./Dissolved)	10.12	ICP-MS	DV-MT-0018 DV-MT-0022
Method 200.8 (Total Rec.)	10.13	ICP-MS	DV-MT-0002
Method 200.8 (Dissolved)	10.13	ICP-MS	DV-MT-0002
Method 3050B (Special Sb Prep)	10.14	ICP-MS	DV-MT-0018 DV-MT-0022

2.0 Summary of Method

2.1 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals.

This preparation method is used for total recoverable and dissolved metals analysis by ICP-MS method 6020 and 6020A. A representative aliquot of sample is heated with nitric acid and substantially reduced in volume. The digestate is diluted to volume and then filtered (if necessary).

2.2 Method 3020A, Acid Digestion of Aqueous Samples and TCLP/SPLP Leachates for Total Metals.

This preparation method is used for total metals analysis by ICP-MS methods 6020, 6020A, and 6020B. A representative aliquot of sample is refluxed with nitric acid. This step is repeated until the digestate is light in color or until its color has stabilized. The digestate is diluted to volume and then filtered (if necessary).

2.3 Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma - Mass Spectrometry.

This preparation method is used for metals analysis by ICP-MS method 200.8. A representative aliquot of sample is refluxed with nitric and hydrochloric acids. The digestate is diluted to volume and then filtered (if necessary).

3.0 Definitions

- Dissolved Metals: The concentration of metals determined in a sample after the sample is filtered through a 0.45-µm membrane (Method 3005A). (The sample is acidified after filtration).

- Total Metals: The concentration of metals determined in an unfiltered sample following digestion (Method 3020A).

- Total Recoverable Metals: The concentration of metals determined in an unfiltered sample following treatment with hot, dilute mineral acid (Method 200.8 and Method 3005A).

- Potentially Dissolved Metals: An acidified sample is filtered between 8 - 96 hours following acidification and the filtrate is digested using Method 3005A.

- Refer to the Glossary of the ETA Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P *Quality Control Program* for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include the following: metallic or metal-containing labware (e.g., latex gloves coated with talc, which contains high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.2 The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination. Refer to Appendix A for additional contamination control guidelines.

4.3 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrices may not be digested using these methods if they are not soluble in acids. If physical interferences are present, they should be documented.

4.4 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.5 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals. If this occurs, the sample must be re-prepared.

4.6 Specific analytical interferences are discussed in the ICP-MS determinative method SOPs, e.g., DV-MT-0002 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by USEPA Method 200.8*, DV-MT-0018 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020*, and DV-MT-0022 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020A/B*.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, and this document.

5.1 Specific Safety Concerns

5.1.1 Samples that contain high concentrations of carbonates, organic material, or that are at elevated pH can react violently when acids are added.

5.1.2 The digestion solution must be cooled sufficiently before adding hydrogen peroxide (H_2O_2) to avoid a reaction and possible violent effervescence or boiling over of the digestion solution.

5.1.3 Care must be taken when handling the digestion tubes. The tubes may become very hot during the digestion procedure. Allow the tubes to cool before attempting to handle the sample digestate.

5.1.4 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm (TWA)	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

(1) Always add acid to water to prevent violent reactions.

(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Digestion block, with adjustable heating, capable of maintaining a sample temperature of 85 - 95 °C.

6.1.2 Thermometer that covers a temperature range of at least 80 - 110 °C.

6.1.3 Centrifugation equipment (if desired method of removing particulate material is centrifugation).

6.2 Supplies

6.2.1 Disposable digestion tubes, with volume accuracy verified to ± 3% gravimetrically prior to use. See SOP DV-QA-0008 *Volumetric Verification.*

6.2.2 Watch glasses, ribbed or equivalent, or disposable digestion tube covers.

6.2.3 Whatman GD/XP - PVDF membrane, 0.45 micron syringe filters (No. 6973-2504), for trace metal analysis, or equivalent. When used to filter any sample in a preparation batch or analytical batch, filters of the same type are also used to filter the method blank and the LCS in the batch. Acceptable results for the QC samples demonstrate that the filters neither add nor subtract analytes.

6.2.4 Syringes or equivalent filtration apparatus.

6.2.5 Repipettors or suitable reagent dispensers.

6.2.6 Calibrated automatic pipettes with pipette tips or Class A glass volumetric pipettes.

6.2.7 Class A volumetric flasks.

6.2.8 pH indicator strips (pH range 0 - 6).

6.2.9 Plastic digestate storage bottles.

7.0 Reagents and Standards

7.1 Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks as defined in the determinative method SOPs, e.g., DV-MT-0002 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by USEPA Method 200.8*, DV-MT-0018 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020*, and DV-MT-0022 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020A/B*.

7.2 Laboratory control sample (LCS), matrix spike, and matrix spike duplicate (MS/MSD) spike solutions are purchased as custom ETA Denver standards. Standards are logged into the Reagents module in TALS and are assigned unique identification numbers that can be used to access traceability information. The TALS identification numbers are recorded on the metals prep bench sheet.

7.2.1 All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. These plastic bottles may be stored in a glass jar.

7.2.2 Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.2.3 See TALS for the list of spiking levels. A volume of 0.1 mL of each working spike solution is added to the 50 mL final sample volume.

7.3 Nitric Acid (HNO_3), concentrated, trace-metal grade or better.

7.4 Nitric Acid, 1:1

Dilute concentrated HNO_3 with an equal volume of reagent water.

NOTE: When preparing dilute acids, always add acid to water. If the water is added to the acid, the sudden increase in temperature may cause splashing.

7.5 30% Hydrogen Peroxide (H_2O_2), ultra-pure grade.

7.6 Hydrochloric Acid (HCl), concentrated, trace metal grade or better.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE or Glass	500 mL	HNO_3, pH < 2	180 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS login, sample, or method comments and/or program Quality Assurance Summaries (QAS) to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in ETA Denver Policy DV-QA-003P *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in ETA Denver Policy DV-QA-024P *QA/QC Requirements for Federal Programs*.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified appropriately. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 *Non-Conformance and Corrective Action System*. This is in addition to the corrective actions described in the following sections.

9.2 Table 2 provides a summary of quality control requirements, including type, frequency, acceptance criteria, and corrective action. Detailed information regarding these criteria and required corrective actions are found in each determinative method SOP.

9.3 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst for each method being performed. Ongoing proficiency

must be demonstrated by each analyst on an annual basis. See Section 12.1 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.4 Preparation Batch

A preparation batch is a group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank (MB), an LCS, a matrix spike (MS), and a matrix spike duplicate (MSD). For samples logged in under Method 200.8, there must be two MS/MSD pairs for every batch containing more than ten samples. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify samples for the MS/MSD pair, then the batch may contain multiple MS/MSD pairs to accommodate client requests. Clients may also request a duplicate LCS (LCSD). In cases where the client has not provided sufficient sample to prepare an MS and MSD, an LCS and LCSD will be prepared to provide evidence of batch precision.

9.5 Sample Count

Laboratory-generated QC samples (e.g., MBs, LCSs) are not included in the sample count for determining the size of a preparation batch. The MS and MSD are also not usually included in the sample count, but may be included at client request.

9.6 Method Blank (MB)

The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. When samples are filtered in the laboratory for determination of dissolved metals, then the blank is filtered using a filter of the same type that was used for the samples. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false-positive data.

9.7 Laboratory Control Sample (LCS)

One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. When samples are filtered in the laboratory for determination of dissolved metals, then the LCS is filtered using a filter of the same type that was used for the samples. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.8 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of

the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client-specific data quality objectives (DQOs) may require the use of sample duplicates in place of or in addition to MS/MSDs. At least one MS/MSD pair must be processed for each preparation batch. Some client programs require a 10% MS/MSD analysis frequency. If insufficient sample is available to process an MS/MSD pair, then a second LCS must be processed. The LCS/LCSD pair is then evaluated according to the MS/MSD criteria.

The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Samples identified as field blanks, equipment blanks, or rinse blanks cannot be used for MS/MSD analysis.

9.9 Quality Assurance Summaries (QAS)

Certain clients may require specific project or program QC that may supersede the SOP requirements. Quality Assurance Summaries (QASs) are developed to address these requirements.

10.0 Sample Preparation Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervisors to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 *Non-Conformance and Corrective Action System*. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

10.3 All samples are to be checked out of Sample Control with the Internal Chain of Custody in TALS properly completed.

10.4 Proper sample identification is extremely important in any preparation procedure. Labeling of beakers, digestion tubes, and bottles must be done in a manner to ensure connection with the proper sample.

10.5 Samples are typically logged in as either waters or soils. Wastes such as organic liquids or sludges and tissues (animal/vegetable) are usually logged in with solid test codes. When initiating sample preparation, examine the sample to see if the sample matches the matrix designation. If the sample is logged in as aqueous, but it appears more like a waste (biphasic, visible oil, sludge-like, organic liquid, lots of sediment, etc.), then contact the project manager and the laboratory group leader for further instructions.

10.6 If possible, prepare all the samples of a project at the same time to minimize the QC required and streamline the flow of the project through the lab, data review, and reporting groups.

10.7 Guidelines are provided in Appendix A on procedures to minimize contamination of samples and standards.

10.8 All data shall be recorded directly on the described forms, logbooks, electronic forms, or directly in TALS at the time of data generation. It is not acceptable to record data on loose papers, scraps of paper, gloves, sample vials, or "Post-It" notes. Data may be recorded on paper bench sheets if the sheets are subsequently scanned and saved in a designated folder on the company server.

10.9 Aqueous Sample Preparation Setup

The following setup procedure must be followed for all aqueous samples prior to performing the specific digestion procedure:

10.9.1 Sample pH must be verified prior to preparation and analysis. Follow the procedure below when new samples are received for metals analysis.

10.9.1.1 Scan the entire cart of metals samples into the TALS internal chain of custody (ICOC) module. Change the current location of the samples to Metals. Export the ICOC to the Metals drive and print a paper copy.

10.9.1.2 Measure the sample pH with pH paper using a separate aliquot of sample. This can be done using disposable plastic droppers or by pouring a small amount of sample onto the pH paper. Do not put the pH paper directly into to bottle. Record the pH on the paper copy of the ICOC. When all of the samples have been tested, initial and date the copy of the ICOC, scan it, and save it to the Metals folder on the G: drive.

10.9.1.3 If the pH > 2 for a sample requiring acidic preservation, add 1 - 2 mL of concentrated HNO_3 to the sample. Replace the lid and mix.

10.9.1.4 Recheck the pH of the sample. If the pH < 2, record the sample information in the Laboratory Sample Filtration and Preservation Logbook. If the pH > 2, repeat Section 10.9.1.3 until the pH < 2 or 5 mL of acid has been added. If the sample still has a pH greater than 2 do not add any additional acid, proceed with sample digestion and create an NCM to document the inadequate preservation. Record the result in the preservation logbook.

10.9.1.5 Allow the sample to sit for 24 hours following acidification.

10.9.1.6 Recheck the pH of the sample. If the pH < 2, proceed with the appropriate digestion procedure. Note the date/time of this pH recheck in the preservation logbook.

10.9.1.7 If after 24 hours the pH > 2, repeat steps 10.9.1.3 through 10.9.1.6 until the pH remains < 2 following the 24-hour period

or 5 mL of HNO_3 has been added. If the sample still has a pH greater than 2 do not add any additional acid, proceed with sample digestion and create an NCM to document the inadequate preservation. Record the result in the preservation logbook.

Note: Acid must be added at least 24 hours before analysis.

10.9.2 Select the unfiltered fraction for a total or total recoverable analysis or the filtered fraction for a dissolved analysis. For SPLP select the proper sample leachates.

NOTE: Samples requiring dissolved metals determination are either filtered and preserved in the field or are filtered and preserved by the laboratory as soon as possible after receiving the samples. When filtered in the laboratory, the filtration and preservation are recorded in the Laboratory Sample Filtration and Preservation Logbook, including the preservative type and lot number. Filter acceptability is demonstrated by using filters of the same type to filter samples and batch QC samples when preparation batches include samples that were filtered in the laboratory. The results of the analysis of the batch QC samples are used to demonstrate that the filtration process neither adds nor subtracts target analytes from the sample results.

10.9.3 Mix the sample by shaking the container.

10.9.4 Measure and transfer 50 mL of the sample into a digestion tube. When using calibrated digestion tubes, pour the sample into the tube to the 50-mL mark. Record the lot number of the digestion tubes in TALS.

10.9.5 Measure two extra aliquots of the sample that is selected for the MS/MSD analysis. Spike each aliquot with 0.1 mL of each spiking solution (see TALS). Record the standards and pipette identifications in TALS.

10.9.6 Measure and transfer 50 mL of reagent water into a digestion tube for the method blank. If a determination of dissolved metals is requested, use filtered reagent water for the method blank.

10.9.7 Measure and transfer 50 mL of reagent water into a digestion tube for the LCS and add 0.1 mL of spiking solution (see Table 2). Record the standards and pipette identifications in TALS. If determination of dissolved metals is requested (preparation method 3005A), and one or more samples were filtered in the laboratory, then filter the LCS and Method Blank using a filter of the same type that was used to filter the sample(s).

10.10 Proceed to the appropriate Section of this SOP for the desired preparation method as follows:

Preparation Method*	SOP Section	Analytical Method
3020A Total Metals	10.11	Method 6020
3005A Total Recoverable	10.12	Method 6020
3005A Dissolved Metals	10.12	Method 6020
200.8 Total Recoverable Metals	10.13	Method 200.8
200.8 Dissolved Metals	10.13	Method 200.8
3050B Special Sb prep	10.14	Method 6020

(See also WI-DV-017 *Preparation of Water Samples for ICP-MS*)

10.11 Method 3020A - Preparation for Total Metals Analysis by ICP-MS Methods 6020, 6020A, or 6020B

10.11.1 To each sample in a digestion tube, add 1.5 mL of concentrated HNO3.

10.11.2 Place each rack of samples onto a Hot Block. Heat at 90 - 95 °C until the volume is reduced to approximately 5 mL. Record the start time and the Hot Block temperature in TALS.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared and reanalyzed.

10.11.3 Remove the sample racks from the Hot Blocks and allow the digestion tubes to cool in a fume hood.

10.11.4 Add 1.5 mL of concentrated HNO3 to each sample. Cover each sample tube with a disposable plastic watch glass and reflux gently.

10.11.5 Continue heating, adding additional acid as necessary in 1 - 2 mL increments to ensure a complete digestion. Record the start and stop times and the Hot Block temperature in TALS.

NOTE: Digestion is complete when the digestate is light in color and does not change in appearance with continued refluxing.

10.11.6 Evaporate to low volume, approximately 3 - 5 mL.

10.11.7 Allow the digestion tubes to cool, then add about 10 mL of reagent water to each tube.

10.11.8 Replace the covers and continue warming for 10 to 15 minutes to allow additional solubilization of any residue to occur. Record the stop time in TALS.

10.11.9 Allow the samples to cool and rinse the watch glasses into the digestion tubes with reagent water.

10.11.10 Re-Volume to 50 mL with reagent water, cap and mix thoroughly.

10.11.11 The sample is now ready for ICP-MS analysis. Refer to SOPs DV-MT-0018 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020* or DV-MT-0022 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020A/B.*

NOTE: If insoluble materials are present in a sample digestate, the ICP-MS analyst may filter the sample prior to analysis. Refer to DV-MT-0018 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020* or DV-MT-0022 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020A/B* for additional details.

10.12 Method 3005A - Preparation for Total Recoverable and Dissolved Metals Analysis by ICP-MS Method 6020, 6020A, or 6020B

10.12.1 To each sample in a digestion tube, add 2.0 mL of concentrated HNO3.

10.12.2 Place each rack of samples onto a Hot Block. Heat the samples to 90 - 95 °C and cautiously evaporate to approximately 10 mL, while ensuring that no portion of the sample container is allowed to go dry. Record the start and stop times and the Hot Block temperature in TALS.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be re-prepared.

10.12.3 Remove the sample racks from the Hot Blocks and allow the samples to cool in a fume hood.

10.12.4 Rinse the digestion tubes with reagent water.

10.12.5 Re-Volume to 50 mL with reagent water, cap and mix thoroughly.

10.12.6 The sample is now ready for ICP-MS analysis. Refer to SOP DV-MT-0018 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020*.

NOTE: If insoluble materials are present in a sample digestate, the ICP-MS analyst may filter the sample prior to analysis. Refer to DV-MT-0018 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020* or DV-MT-0022 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020A/B* for additional details.

10.13 Method 200.8 - Preparation for Total Recoverable, Potentially Dissolved, and Dissolved Metals Analysis by ICP-MS

10.13.1 To each sample, add 0.5 mL of concentrated HNO3 and 0.25 mL of concentrated HCl.

10.13.2 Adjust the digestion block temperature so the solution in a covered container rises to approximately 90 - 95 ºC. Record the temperature in TALS.

10.13.3 Heat the sample until it evaporates to approximately 10 mL, while ensuring that no portion of the bottom of the digestion tube is allowed to go dry.

CAUTION: DO NOT ALLOW SAMPLE TO BOIL OR GO DRY. Doing so will result in the loss of analyte and the sample must be reprepared.

10.13.4 Cover the sample with a disposable watch glass and gently reflux for an additional 30 minutes. Avoid vigorous boiling to prevent the loss of the HCl-H2O azeotrope. Record the start and stop times and the Hot Block temperature in TALS.

10.13.5 Remove the sample racks from the Hot Blocks and allow the samples to cool in a fume hood.

10.13.6 Rinse the watch glass or cover into the container and re-volume to 50 mL with reagent water. Cap and mix thoroughly.

10.13.7 The sample is now ready for ICP-MS analysis. Refer to SOP DV-MT-0002 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by USEPA Method 200.8*.

NOTE: If insoluble materials are present in a sample digestate, the ICP-MS analyst may filter the sample prior to analysis. Refer to SOP DV-MT-0002 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by USEPA Method 200.8* for additional details.

10.14 Method 3050B – Special Prep for Sb, Sn and Ag for Analysis by ICP-MS Methods 6020, 6020A, or 6020B

10.14.1 To 25 mL of sample in a 100 mL digestion tube, add 2.5 mL of HNO3 and 2.5 mL of HCl.

10.14.2 Heat at 90 - 95 °C until the sample has reduced to a volume of 10 - 15 mL ensuring that no portion of the sample container is allowed to go dry. Record the start and stop times and the Hot Block temperature in TALS.

10.14.3 Remove the sample racks from the Hot Blocks and allow the samples to cool in a fume hood.

10.14.4 Add 1.0 mL of HCl to each digestion tube and cover with a ribbed watch glass.

10.14.5 Replace the watch glass and heat the sample for 15 minutes. Record the start and stop times and the Hot Block temperature in TALS.

10.14.6 Remove the sample racks from the Hot Block and allow the samples to cool in a fume hood.

10.14.7 Re-volume to 100 mL with reagent water, cap and mix thoroughly.

10.15 Calibration

The digestion block temperature must be maintained between 90 and 95 °C. The temperature must be monitored continuously while in use and must be recorded on the metals preparation bench sheet. The temperature must be monitored by measuring the temperature of reagent water contained in a digestion tube that is placed in each digestion block. The thermometer used and the start and end temperatures are recorded in TALS. The thermometer is calibrated in accordance with SOP DV-QA-0001 *Thermometer Calibration*.

11.0 Calculations / Data Reduction

11.1 Not applicable. See the determinative method SOPs, DV-MT-0002 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by USEPA Method 200.8*, DV-MT-0018 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020*, and DV-MT-0022 *Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020A/B* for data analysis and applicable calculations.

11.2 Documentation

11.2.1 All of the preparation information is recorded and stored in TALS.

11.2.2 The preparation information includes:

11.2.2.1 Batch number, job and sample numbers, preparation date, and analyst name;

11.2.2.2 Matrix and prep type;

11.2.2.3 Initial sample pH, Initial sample volume and final volume;

11.2.2.4 Reagent manufacturer and lot number for each reagent used;

11.2.2.5 Digestion tube lot information;

11.2.2.6 Standard identification number for each standard used;

11.2.2.7 Start and stop times for digestions;

11.2.2.8 Observed and corrected temperature readings during digestion;

11.2.2.9 Identification numbers of calibrated measuring equipment used (thermometers, balances, pipettes, etc.).

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the corporate MDL policy described in CA-Q-S-006 *Detection and Quantitation Limits*. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024 *Training*.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024 *Training*.

13.0 Pollution Control

13.1 This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

13.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Safety Manual, and DV-HS-001P *Waste Management Plan.*

14.2 The following waste streams are produce when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards: Contact Waste Coordinator

14.2.2 Acidic waste from sample digests: Waste Stream J.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.

15.1.2 Method 3020A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy, Revision 1, July 1992.

15.1.3 Method 6020, Inductively Coupled Plasma - Mass Spectrometry, Revision 0, September 1994.

15.2 Methods for the Chemical Analysis of Water and Waste (MCAWW), 1983.

15.3 Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectroscopy, Revision 5.4, May 1994.

16.0 Method Modifications:

16.1 Modifications and Interpretations Applicable to SW-846 Reference Methods

16.1.1 Chapter 1 of SW-846 states that the method blanks should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above one-half of the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

16.1.2 The referenced methods, as well as Table 3-1 of SW-846, refer to the use of a 100 mL aliquot for digestion. This SOP requires the use of a 50 mL sample size to reduce waste generation. The use of reduced sample volumes is supported in EPA's document "Response to Public Comments Background Document, Promulgation of the Second Update to SW-846, Third Edition", dated November 3, 1994. This document stated, "...flexibility to alter digestion volumes is addressed and 'allowed' by the table (3-1) and is also inherently allowed by specific digestion methods. Table 3-1 is only to be used as guidance when collecting samples..."

EMSL-Ci has also taken the stance that "reduction in sample size and appropriate corresponding reduction in sample volume is not considered a significant change in the methodology." Additionally, in written correspondence from the Office of Solid Waste, Oliver Fordham stated. "As a 'representative sample' can be assured, scaling causes no loss of precision and accuracy in the analysis."

16.2 Modifications Specific to Method 3005A

An additional 1.0 mL of HNO_3 was included to replace the 5.0 mL of HCl. HCl was eliminated to reduce interferences from chloride.

16.3 Modifications and Interpretations Specific to Method 3020A

16.3.1 Section 10.11.6 of this SOP requires that the sample be reduced to a volume of 3 - 5 mL. Section 7.2 of Method 3020A states that the volume

should be reduced to 3 mL, but also states that no portion of the bottom of the digestion tube should go dry. The volume required by this SOP is a closer approximation of the volume required to provide an adequate covering of the bottom of the digestion tube so as to prevent the loss of critical analytes through volatilization.

16.3.2 The scope of 3020A has been expanded to include silver, based on comparison studies with 7760A. Method 3020A consistently demonstrated improved accuracy and precision over Method 7760A in the matrices tested (reagent water, surface water, and TCLP leachate) up to a concentration of 1 ppm silver.

17.0 Attachments

Table 1. TCLP Reporting Limits, Regulatory Limits, and Matrix Spike Levels
Table 2. Summary of Quality Control Requirements
Appendix A. Contamination Control Guidelines

18.0 Revision History

This section has been added beginning with Revision 0. Only details of the last two revisions are incorporated into this SOP. Prior revisions are documented in the QA files and available upon request.

- Revision 15, Dated ~~09~~ 18 February 2022
  - Updated 10.13.6, re-volume to 50mL rather than 25mL

- Revision 14, Dated 30 December 2021
  - Annual Review
  - Updated copyright information.
  - Updated language and formatting throughout

- ~~Revision 13, Dated 31 December 2020~~
  - ~~Annual Review~~
  - ~~Minor language and formatting corrections throughout~~
  - ~~Added references to Method 6020B throughout~~
  - ~~Added clarification to Section 9.5~~
  - ~~Updated Section 10.9 and subsections to clarify pH checking procedure~~
  - ~~Added language to Sections 10.11.3 and 10.11.4 to clarify procedure~~
  - ~~Removed reference to drinking water in Section 10.13.6~~

Table 1.

TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	RL (µg/L)	Regulatory Limit (µg/L)	Spike Level (µg/L)
Arsenic	500	5,000	3,200
Barium	10,000	10,0000	10,200
Cadmium	100	1,000	1,200
Chromium	500	5,000	5,200
Lead	500	5,000	5,200
Selenium	250	1,000	1,200
Silver	500	5,000	1,200

Table 2.

Summary of Quality Control Requirements

QC Parameter	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per sample preparation batch of up to 20 samples	Refer to Determinative SOP: 200.8: DV-MT-0002 6020: DV-MT-0018 6020A/B: DV-MT-0022	Re-digest and reanalyze samples associated with the method blank
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples	Refer to Determinative SOP: 200.8: DV-MT-0002 6020: DV-MT-0018 6020A/B: DV-MT-0022	Re-digest and reanalyze samples associated with the method blank
Matrix Spike (MS)	One per sample preparation batch of up to 20 samples	Refer to Determinative SOP: 200.8: DV-MT-0002 6020: DV-MT-0018 6020A/B: DV-MT-0022	Re-prep not required unless preparation error suspected.
Matrix Spike Duplicate (MSD)	See Matrix Spike frequency	Refer to Determinative SOP: 200.8: DV-MT-0002 6020: DV-MT-0018 6020A/B: DV-MT-0022	See Corrective Action for Matrix Spike

Appendix A.

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered or latex gloves must not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.
- Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Separate glassware if an unusually high sample is analyzed and soak with sulfuric acid prior to routine cleaning.

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Title: Acid Digestion of Solids [Method EPA 3050B]

Approvals (Signature/Date):

Doug Gomer 6/8/21
Doug Gomer Date
Technical Specialist

Reed Pottruff 6/8/2021
Reed Pottruff Date
Health & Safety Manager / Coordinator

Maria Fayard 6/9/21
Maria Fayard Date
Quality Assurance Officer

Scott Hall 6/8/21
Scott Hall Date
Laboratory Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This is a strong acid digestion procedure for the preparation of sediments, sludge, soils, and other types of solid materials by EPA Method 3050B for analysis by inductively coupled plasma atomic emission spectroscopy (ICP) or inductively coupled plasma-mass spectrometry (ICP/MS).

1.2 Method 3050B is designed to determine the concentration of "environmentally available" metals, and is not a true "total metals" digestion (see discussion below). The procedure is used primarily for hazardous waste characterization and other Resource Conservation and Recovery Act (RCRA) compliance testing.

1.3 The elements approved for Method 3050B are shown in Table I. The source method also mentions that other elements may be prepared by the method if the quality control requirements are met. The complete list of elements routinely included in this procedure by Eurofins TestAmerica Denver is shown in Table II.

1.4 If sample preparation utilizing the Incremental Sampling Method is required, see SOP DV-OP-0013 Incremental Sampling Methodology for Soils and Sediments for the procedure required prior to acid digestion for metals incorporating this procedure.

2.0 Summary of Method

A representative 1 to 2 gram portion of sample is digested with two cycles of nitric acid additions, followed by hydrogen peroxide digestion. For ICP analysis, the sample is also refluxed with hydrochloric acid. The resulting solution is filtered and diluted to 100 mL with reagent water. For the Incremental Sampling Method, 10 g of sample is used and brought to a final volume of 500 ml.

3.0 Definitions

3.1 Refer to the Glossary of the Eurofins TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P Quality Control Program for definitions of general analytical and QA/QC terms.

3.2 **Total Metals** - Although Method 3050B is often referred to as a "total metals" digestion, it is important to understand that there are many compounds formed from these elements that are not efficiently dissolved using this digestion procedure. It is more accurately termed a strong acid digestion procedure. The limitations are discussed further in Section 4 (Interferences) below. The method itself states, "This method is not a total digestion technique for most samples." There are a variety of total digestion procedures used for metal assay, geochemical analysis, etc., that involve more vigorous digestions than 3050B.

3.3 **Preparation Batch** - A group of up to 20 samples that are of the same matrix and are processed together using the same lots of reagents and standards. The minimum QC elements in a batch are outlined in Section 9.

3.4 **Reagent Water –** Water that is free of the analytes of interest. In the Metals group, reagent water is obtained from a Barnstead E-Pure water purification system.

3.5 Other quality control terminology used in this procedure is based on SW-846, and is defined in the glossary section of the Eurofins TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P Quality Control Program.

4.0 Interferences

4.1 There are common compounds formed by the elements of interest (e.g., barium sulfate, beryllium oxide, silicon dioxide, crystalline silicates, titanium dioxide, etc.) that are not efficiently dissolved using this EPA approved procedure.

4.2 Silicon or silica are occasionally requested as part of the Method 3050B digestion. However, this digestion will include only acid-soluble silicon, and will not dissolve crystalline silica. The analysis is for silicon, but the final result is sometimes expressed as silica rather than silicon.

4.3 Antimony and silver have poor solubility in dilute nitric acid solution. Therefore it is strongly recommended that these elements are determined by the ICP-MS procedure that includes HCl as the final digestion acid. See Section 11.12 of this SOP.

4.4 Potential sources of trace metals contamination include metallic or metal-containing labware (e.g., powdered gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. See Attachment 1 for more information regarding contaminant control.

4.5 The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.

4.6 For critical low-level determinations of boron and silica, only quartz and/or plastic labware should be used.

4.7 Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents, and other matrix materials may not be digested using these methods if they are not soluble in acids. If physical interferences are present, they should be documented.

4.8 Allowing samples to boil or go dry during digestion may result in the loss of volatile metals or conversion of metals to insoluble forms. For example, antimony is easily lost by volatilization from hydrochloric media. If this occurs the sample must be re-prepared.

4.9 Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.

4.10 Samples Requiring Additional Digestion Reagents

A few examples of types of samples that might require additional digestion reagents follow. It is very important to note situations where samples are not behaving normally. However, do not assume that adding additional reagents will be acceptable for the project, even if it is obvious that the digestion will be incomplete without it. The situation must be discussed with the project manager and documented in a Nonconformance Memo (NCM), whether or not the variations suggested in the following examples are approved.

4.10.1 Samples with high organic content may require additional nitric acid and/or hydrogen peroxide for a thorough digestion, but these oxidizing reagents should be added very carefully to avoid violent reactions.

4.10.2 Samples with high concentrations of metal in the elemental form or refractory oxides may require additional hydrochloric acid for a thorough digestion. As an example, blasting sand used to remove paint from the hull of ships typically consists of 30% cupric oxide. Following 3050B exactly will produce results as low as 0.1% without additional hydrochloric acid. Increasing the volume of hydrochloric acid can produce results approaching the true copper concentration. Samples that appear to have nonstandard matrices or have visible metal particles should be documented in an NCM.

4.10.3 Highly alkaline materials may require larger volumes of acid than specified in this procedure.

4.10.4 If the use of extra digestion reagents is approved, the same volume of reagents must be added to all field samples and QC samples in the batch. Usually the method blank results will not be elevated. To ensure that the QC sample results accurately reflect sample results, the QC samples must be treated exactly like the samples.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Samples that contain high concentrations of carbonates or organic materials or samples that are at elevated pH can react violently when acids are added. If any solid sample appears to be a chemical

substance rather than an environmental sample, consult with the group supervisor or the Project Manager (PM) before adding acid.

5.3.2 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit(2)	Signs and Symptoms of Exposure
Hydrogen Peroxide, H_2O_2	Oxidizer Corrosive Poison	1 ppm TWA 1.4 mg/m^3 TWA 75 ppm IDLH	Contact with other materials may cause fire. Eye contact may result in permanent eye damage. Causes eye and skin burns. Corrosive: May cause severe respiratory tract irritation. Harmful if swallowed, may cause digestive tract irritation or burns.
Nitric Acid, HNO_3	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid, HCl	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

(1) Always add acid to water to prevent violent reactions.

(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1 Instrumentation

6.1.1 Top-loading balance capable of accurately weighing to the nearest 0.01 grams.

Note: Balances are serviced annually and the accuracy checked daily using three standard masses. See SOP DV-QA-0014 Selecting and Using Balances for details.

6.1.2 Digestion "Hot Block" or equivalent heating device capable of maintaining a temperature of 90-95 °C. The Hot Block temperature must be monitored separately for each unit. The temperature of each Hot Block is checked by placing a calibrated thermometer through a cap on a digestion tube that is partially filled with water. The water in the tube must be high enough to cover the thermometer past the minimum immersion line. The temperature is directly recorded in the Batch Information area in the Eurofins TestAmerica LIMS (TALS).

6.2 Supplies

6.2.1 Thermometers (non-mercury liquid filled or digital) that cover a temperature range including 80-110 °C with clearly visible 1 °C increments.

Note: Thermometers are calibrated before use and periodically as described in SOP DV-QA-0001 Thermometer Calibration.

6.2.2 Disposable digestion tubes, with volume accuracy verified to ± 3% gravimetrically prior to use. See SOP DV-QA-0008 Volumetric Verification.

6.2.3 Watch glasses, ribbed or equivalent, or disposable digestion tube covers.

6.2.4 Whatman 541 (acid washed) filter paper, or equivalent.

6.2.5 Whatman GD/XP - PVDF membrane, 0.45-micron syringe filters, No. 6973-2504, for trace metal analysis, or equivalent. When used to filter any sample in a preparation batch or analytical batch, filters of the same type are also used to filter the method blank and the LCS in the batch. Acceptable results for the QC samples demonstrate that the filters neither add nor subtract analytes.

6.2.6 Syringes or equivalent filtration apparatus.

6.2.7 Disposable plastic funnels.

6.2.8 Disposable wooden spatulas for subsampling.

6.2.9 Centrifuge, capable of at least 2,000 rpm.

6.2.10 Graduated cylinders, 100 mL and 500 mL, capable of ± 3% accuracy.

6.2.11 Calibrated automatic pipettes with corresponding pipette tips or Class A glass volumetric pipettes.

Note: Mechanical pipettes are calibrated before use as described in SOP DV-QA-0008 Volumetric Verification.

6.2.12 Class A volumetric flasks.

6.2.13 pH indicator strips (pH range 0 – 6).

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Reagent water – Millipore DI system or equivalent, 10-18.2 megohm-cm. See SOP DV-QA-0026 for daily water monitoring procedure.

7.2 Nitric acid (HNO_3), concentrated - Trace metal grade or better.

7.3 Nitric acid (HNO_3), 5% - Add 50 mL of concentrated HNO_3 to approximately 900 mL of reagent water and dilute to 1 liter.

7.4 Hydrochloric acid (HCl), concentrated - Trace metal grade or better.

7.5 30% Hydrogen peroxide (H_2O_2) - Reagent grade used for ICP analysis.

7.6 30% Hydrogen peroxide (H_2O_2) – Ultra pure used for ICP-MS analysis.

7.7 Glass beads, ≤ 1 mm diameter, washed with aqua regia (for DoD projects).

7.8 Standards

7.8.1 All standards must be NIST traceable. Unless purchased directly from NIST, the accuracy of each standard is verified before the initial use, as described in SOP DV-QA-0015 Verification and Storage of Chemical Standards and Reagents.

7.8.2 Storage and Shelf Life of Metal Standards

7.8.2.1 Standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. They are stored at room temperature.

7.8.2.2 Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.8.3 LCS and MS Spike Solutions

7.8.3.1 ICP and ICP/MS spike solutions are purchased as custom-made solutions from a commercial vendor at ready-to-use concentrations. No further dilutions are needed.

7.8.3.2 If a non-routine element is required that is not contained in the custom-made solution, single-element solutions from a commercial vendor may also be used.

7.8.3.3 Intermediate standards prepared in the laboratory may be used for spiking as long as the procedures for standard recording and verification outlined in SOP DV-QA-0015 Verification and Storage of Chemical Standards and Reagents are followed.

Typical LCS and MS/MSD spike standard concentrations are shown below.Analysis	Standard	Elements	Conc. (mg/L)
ICP	Spike Mix #1	As, Se, Si, Sn, Tl Ba, Be, Cd, Co, Cr, Li, Mn, Mo, Ni, Pb, Sr, Ti, V	200 100
ICP	Spike Mix #2	Ca, K, Mg, Na Al, Fe	5000 1000
ICP	Spike Mix # 3	P S U W	2000 1000 200 100

Typical LCS and MS/MSD spike standard concentrations are shown below.Analysis	Standard	Elements	Conc. (mg/L)
ICP	Spike Mix # 4	Bi B, Th Zn, Zr	200 100 50
ICP	Spike Mix # 4B	Ag	5
ICP	Spike Mix # 5	Sb	100
ICP-MS	MS CALSTD-1	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Si, Sn Sr, Ti, Tl, V	100
ICP-MS	ICP CALSTD-2	Al, Ca, Fe, K, Mg, Na	2000
ICP-MS	MS Spike 2	Ag, Sr, Th, U, W, Zn	20

Note: ICP or ICP-MS digestions may select different combinations of spikes in order to satisfy client requests. All spikes used for sample digestion will be recorded in the Reagent module in TALS.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Sample holding time for metals included under the scope of this SOP is 180 days from the date of collection to the date of analysis.

8.2 Soil samples do not require chemical preservation, but are stored at ≤6 °C until the time of analysis.

Matrix	Sample Container	Min. Sample Size	Preservation[1]	Holding Time[2]	Reference
Soils	Glass	3 grams	Cool ≤ 6 °C	180 Days	N/A

[1] Although ICP analysis of soil does not require refrigeration of the samples, mercury analysis does require refrigeration. Samples which will be used to aliquot for both analyses must be refrigerated.

[2] Inclusive of digestion and analysis.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical

results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in Eurofins TestAmerica Denver policy DV-QA-003P Quality Control Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in Eurofins TestAmerica Denver policy DV-QA-024P QA/QC Requirements for Federal Programs. This procedure meets all criteria for DoD QSM unless otherwise stated. Any deviation or exceptions from QSM requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 Non-Conformance and Corrective Action System. This is in addition to the corrective actions described in the following sections.

9.2 Preparation batches may consist of up to 20 field samples. Laboratory generated QC samples (method blanks, LCS, MS/MSD) are not counted towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

9.3 Minimum QC Requirements

Each preparation batch must contain a method blank (MB), a laboratory control sample (LCS), and a matrix spike/matrix spike duplicate (MS/MSD) pair. Note that some programs require an unspiked duplicate sample in place of or in addition to the duplicate matrix spike. Be sure to check special instructions in TALS. If clients specify specific samples for the MS and MSD, the batch may contain multiple MS/MSD pairs.

9.3.1 Method Blank (MB)

One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents

specific to the method that is carried through the entire analytical procedure, including preparation and analysis. Soil method blanks are prepared by taking 5 mL or 5 g of reagent water through the procedure described in Section 11. Add 1.0 g of prewashed glass beads to the blank if required by the client to better simulate a solid matrix.

The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data.

Acceptance Criteria: Criteria for the acceptance of blanks are contained within the individual analytical method SOPs.

Corrective Action: If the method blank does not meet the criteria contained within the analytical method SOPs, the blank and all associated samples in the batch must be re-digested and reanalyzed.

9.3.2 Laboratory Control Sample (LCS)

One aqueous LCS must be processed with each preparation batch. The LCS contains reagent water that is spiked with all the analytes of interest and is carried through the entire analytical procedure. A duplicate LCS (LCSD) must be prepared when there is insufficient sample volume to perform an MS/MSD. The LCS is used to monitor the accuracy of the analytical process. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. Add 1.0 g of prewashed glass beads to the LCS if required by the client to better simulate a solid matrix.

The spike solutions described in Section 7.8.3 are used to prepare LCSs as follows:

- Routine ICP: Add 1.0 mL of spike
- DoD ICP: Add 1.0 mL of spike to 1.0 g of glass beads
- Routine ICP-MS: Add 1.0 mL of spike
- DoD ICP-MS: Add 1.0 mL of spike to 1.0 g of glass beads

The resulting spike concentrations for each element are given in Table 2 and Table 3.

Incremental Sampling Method LCSs are spiked with 5 ml of spike.

Acceptance Criteria: Criteria for the acceptance of LCS results are contained within the individual analytical method SOPs.

Corrective Action: When LCS results fail to meet control limits, the LCS and all associated samples in the batch must be re-prepared and reanalyzed.

9.3.3 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a second aliquot of a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a third aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Samples identified as field blanks cannot be used for MS/MSD analysis. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process.

The spike solution described in Section 7.7.3 is also used to prepare matrix spikes, as follows:

- ICP: Add 1.0 mL of spike
- ICP-MS: Add 1.0 mL of spike

The resulting spike concentrations for each element are given in Table 2 and Table 3. Incremental Sampling Method MS/MSD pairs are spiked with 5 ml of spike.

NOTE 1: The spike must be added after the sample aliquot but before any reagents.

NOTE 2: This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD precision is preferred as not all samples will contain measurable concentrations of the target analytes. Samples that have target analytes at low concentrations or non-detectable levels do not provide useful precision data. When an MS/MSD pair is not available, an LCS and LCSD are used to measure precision.

10.0 Calibration

Not applicable. This SOP addresses sample preparation only for subsequent ICP or ICP/MS analysis. Calibration of the measurement system is covered in the SOPs for the determinative methods.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client

can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 Non-Conformance and Corrective Action System. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.3 Sample Custody

11.3.1 Samples are transferred from the Sample Control group to the Metals group and the transfer is documented using the Sample Transfer function of the Internal Chain of Custody in TALS (see SOP DV-QA-0003 Sample Management and Chain of Custody for details).

11.3.2 Proper sample identification is extremely important in any preparation procedure. Labeling of digestion tubes and bottles must be done in a manner to ensure connection with the proper sample.

11.4 Subsampling

11.4.1 It is not acceptable to simply collect 1.0 g off of the top of the sample. Samples must be mixed and incrementally subsampled to obtain a representative portion. At a minimum, mix by stirring with a disposable wooden spatula. If there is insufficient room in the sample container to allow for proper mixing, refer to SOP DV-QA-0023 Subsampling, for directions.

11.4.2 Select at least three incremental subsamples from different locations in the original sample and place them in a tared 50 mL digestion tube. The final sample weight should be between 1.0 and 1.5 g. Record the weight to the nearest 0.01 g.

11.4.3 Measure additional aliquots for QC samples required in the batch and spike as required (see Section 9 for details).

NOTE: When adding glass beads to the Method Blank and LCS digestion tubes, the nominal weight must be entered into the Initial Amount field in TALS. The true weight of glass beads should be recorded in the Notes field on the Worksheet tab in the preparation batch.

11.5 Incremental Sampling Method Digestion

For the Incremental Sampling Method approximately 10 g of sample is weighed out by the Organic Prep group following the procedure described in SOP DV-OP-0013 Incremental Sampling Methodology for Soils and Sediments. This pre-weighed sample is then delivered to the Metals group for digestion and analysis. The sample weight is recorded on the ISM Worksheet and attached to the incremental sampling batch in TALS. The pre-weighed aliquots are delivered in 125 mL digestion tubes which are ready for spike standards and reagents to be added. The Method 3050B digestion reagents are increased 5x to maintain the

same proportions as are used for a 1-2 gram sample. When required, 10 g of glass beads are added to the Method Blank and LCS prior to digestion.

11.6 Initial Digestion Cycle with 1:1 Nitric Acid

11.6.1 Add approximately 5 mL of reagent water to each digestion tube.

11.6.2 Add 5 mL of concentrated HNO_3.

11.6.3 After all of the acid has been added to the preparation batch, gently swirl the samples to mix and then place the sample rack on the Hot Block.

11.6.4 Place a ribbed cover on each tube.

11.6.5 Heat samples to 90-95 °C, and reflux for 15 minutes without boiling.

NOTE: **DO NOT ALLOW SAMPLES TO BOIL OR GO DRY** during any part of the digestion. Doing so will result in the loss of analyte and the sample must be re-prepared.

11.6.6 Remove the samples from the Hot Block and allow them to cool before proceeding with the next step.

11.6.7 Record the start time, starting temperature, end time, and ending temperature in TALS.

11.7 Second Digestion Cycle Using Concentrated Nitric Acid

11.7.1 Add 5 mL of concentrated HNO_3, and replace the ribbed cover.

11.7.2 Place samples back on the Hot Block and reflux at 90-95 °C for 30 minutes. Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.

11.7.3 If brown fumes are observed, this means that material in the sample is actively being oxidized. There may not be enough HNO_3 acid to complete the oxidation, and there could be violent reaction of the sample with peroxide in the third digestion step. For that reason, it is necessary to repeat the previous two steps until no more fumes are evolved.

11.7.4 Heat the samples at 90-95 °C for 2 hours.

11.7.5 Allow the samples to thoroughly cool before proceeding.

11.8 Third Digestion Cycle Using Hydrogen Peroxide

11.8.1 Add 2 mL of reagent water to each tube.

11.8.2 Add 3 mL of 30% H_2O_2 a few drops at a time. Care must be taken to

ensure that losses do not occur due to excessively vigorous effervescence.

11.8.3 Replace the ribbed cover and heat samples until effervescence subsides.

11.8.4 Allow the samples to cool.

11.8.5 Continue adding 30% H_2O_2 in 1 mL increments with warming until effervescence is minimal or sample appearance is unchanged. If additional peroxide is added to a sample then it must also be added to the method blank and LCS.

NOTE: Do not add more than a total of 10 mL of 30% H_2O_2. If 10 mL have been added and the samples are still vigorously effervescing, document the situation with an NCM and continue with the digestion.

11.8.6 Heat the samples at 90-95 °C for 2 hours.

11.8.7 Allow the samples to cool.

11.8.8 If samples will be analyzed by ICP, continue on with the fourth digestion step using HCl in Section 11.8. If the samples will be analyzed by ICP-MS, skip the HCl digestion step and go to step 11.10.

11.9 Fourth Digestion Cycle for ICP Using Concentrated Hydrochloric Acid

11.9.1 If the samples are being prepared for ICP analysis, add 10 mL of concentrated HCl to the samples in the digestion tubes and cover with ribbed covers.

11.9.2 Reflux for an additional 15 minutes without boiling.

11.9.3 Allow the samples to cool.

11.10 Separating Undigested Solids from the Digestion Solution

11.10.1 Filter samples through Whatman 541 or equivalent fiber filters into a graduated 125 mL digestion tube whose accuracy is documented to be better than ± 3%.

NOTE: In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

11.10.2 For samples digested by the Incremental Sampling Method use a 500 mL poly bottle that has been measured after measuring out 500 mL of DI water from a graduated cylinder.

11.10.3 Wash the original digestion tube and ribbed cover with reagent water to

ensure quantitative transfer of all of the digestion solution into the new digestion tube.

11.10.4 Rinse the funnel and filter paper with reagent water to ensure complete sample transfer into the new digestion tube.

11.10.5 Re-volume sample to 100 mL with reagent water. This must be done volumetrically, rather than by weight. Record the final volume in TALS. For Multi-Incremental samples the final volume is 500 mL.

11.11 Documentation and Record Management

The following information must be recorded for each preparation batch. This information is directly entered into TALS.

- Initial sample weight and final digestion volume
- Preparation analyst and date
- Identification of all reagents and standards
- Identification of all measuring and test equipment used (e.g., balances, thermometers, pipettes)
- Glass beads lot number
- Filter paper lot number
- Digestion tube lot number
- Hot Block ID number
- Fume Hood ID number

11.12 Alternate Antimony Preparation for Analysis by ICP-MS

11.12.1 Weigh out 1.0-1.5 g soil samples according to the procedure in Section 11.3.

11.12.2 Add approximately 5 mL of reagent water to each digestion tube.

11.12.3 Spike the LCS, LCSD, MS, and MSD with 1.0 mL of the MS spike 2 standard.

11.12.4 Add 2.5 mL concentrated HNO_3 and 2.5 mL concentrated HCl to each sample and batch QC.

11.12.5 Cover each tube with a watch glass and reflux on hot block at 90-95 °C for 15 minutes.

11.12.6 Filter through Whatman 541 or equivalent filter paper into a new 125 mL

digestion tube while still hot.

11.12.7 Rinse the filter and funnel with 1.25 ml of hot (~95 °C) concentrated HCl.

11.12.8 Rinse three times with hot (~95 °C) reagent water (5 mL rinses.)

11.12.9 Place the filter paper and soil residue back into the original sample digestion vessel. Add 2.5 mL concentrated HCl, cover and reflux on the hot block for 20 minutes or until paper dissolves.

11.12.10 Filter through a fresh filter into the original filtrate. Rinse three times with reagent water (5 mL rinses).

11.12.11 Bring to final volume of 100 mL with reagent water.

12.0 Calculations / Data Reduction

Not applicable. Calculations of final results are described in the determinative analytical SOPs.

13.0 Method Performance

13.1 Method Detection Limit (MDL)

An MDL must be determined for each analyte/matrix prior to the analysis of any samples. See the SOPs for the determinative analysis methods for details.

13.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024 Training.

13.3 Training Requirements

The group leader or supervisor is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered in the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024 Training.

14.0 Pollution Control

14.1 It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.2 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P Waste Management Plan.

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Aqueous Acidic (Metals) - Corrosive – Waste Stream J

15.2.2 Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

16.0 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update III, December 1996; Method 3050B.

16.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/25/2010.

16.3 Department of Defense Quality Systems Manual for Environmental Laboratories Version 5.0, July 2013.

17.0 Method Modifications:

Item	Method	Modification
1	3050B	Method 3050B Section 7.1 states that a 1-2 g aliquot is to be used. The amount specified by Eurofins TestAmerica Denver in this procedure is limited to 1-1.5 g in order to prevent increased instrument maintenance and sample reruns due to dilutions.

18.0 Figures, Tables, and Attachments

Table 1: Method 3050B Approved Analyte List for ICP/ICP-MS

Table 2: Soil LCS and MS/MSD Spikes for ICP

Table 3: Soil LCS and MS/MSD Spikes for ICP-MS

Attachment 1: Contamination Control Guidelines

19.0 Revision History

This section has been added beginning with Revision 0. Only details of the last two revisions are incorporated into this SOP. Prior revisions are documented in the QA files and available upon request.

- Revision 16 dated 11 June 2021
    - Removed QSM versions and instead referenced SOP DV-QA-024P QA/QC Requirements for Federal Programs for information about DoD QSMs.
- Revision 15 dated 23 April 2021
    - Annual Review
    - Updated copyright information
    - Changed TestAmerica to Eurofins TestAmerica throughout
    - Updated language and formatting throughout

Table 1.

Method 3050B Approved Analyte List for ICP/ICP-MS

Element	Symbol	CAS Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Thallium	Tl	7440-28-0
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6

Table 2.

Soil LCS and MS/MSD Spikes for ICP

ELEMENT	Stock Standard (mg/L)	Sample Spike (mg/kg)	Final Digested Solution (mg/L)
Aluminum	1,000	1,000	10.0
Antimony	100	100	1.0
Arsenic	200	200	2.0
Barium	200	200	2.0
Beryllium	100	100	1.0
Bismuth	200	200	2
Boron	100	100	1.0
Cadmium	100	100	1.0
Calcium	5,000	5,000	50
Chromium	100	100	1.0
Cobalt	100	100	1.0
Copper	100	100	1.0
Iron	1,000	1,000	10.0
Lead	100	100	1.0
Lithium	100	100	1.0
Magnesium	5,000	5,000	50
Manganese	100	100	1.0
Molybdenum	100	100	1.0
Nickel	100	100	1.0
Phosphorous	2,000	2,000	20
Potassium	5,000	5,000	50
Selenium	200	200	2.0
Silicon	200	200	2.0
Silver	5	5	0.05
Sodium	5,000	5,000	50
Strontium	100	100	1
Sulfur	1,000	1,000	10
Thallium	200	200	2
Thorium	100	100	1.0
Tin	200	200	2.0
Titanium	100	100	1.0
Uranium	200	200	2.0
Vanadium	100	100	1.0
Zinc	50	50	0.50
Zirconium	50	50	0.50

NOTE: Final soil spike concentration based on the addition of 1.0 mL stock standard to 1.0 g of sample, which is then digested to produce a 100 mL final volume.

Table 3.

Soil LCS and MS/MSD Spikes for ICP-MS

ELEMENT	Stock Standard (mg/L)	Sample Spike (mg/kg)	Final Digested Solution (µg/L)
Aluminum	400	400	4,000
Antimony	20	20	200
Arsenic	20	20	200
Barium	20	20	200
Beryllium	20	20	200
Cadmium	20	20	200
Chromium	20	20	200
Cobalt	20	20	200
Copper	20	20	200
Iron	400	400	4,000
Lead	20	20	200
Manganese	20	20	200
Molybdenum	20	20	200
Nickel	20	20	200
Selenium	20	20	200
Silver	20	20	200
Strontium	40	40	400
Thallium	20	20	200
Thorium	20	20	200
Tin	20	20	200
Tungsten	20	20	200
Uranium	20	20	200
Vanadium	20	20	200
Zinc	20	20	200

NOTE: Final soil spike concentration based on the addition of 1.0 mL stock standard to 1.0 g of sample, which is then digested to produce a 100 mL final volume.

Attachment 1

Contamination Control Guidelines

The following procedures are strongly recommended to prevent contamination:

- All work areas used to prepare standards and spikes should be cleaned before and after each use.
- All glassware should be washed with 5% HNO_3 according to the procedure described in SOP DV-IP-0005 Glassware Washing - Inorganic.
- Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.
- Powdered should not be used in the metals laboratory since the powder contains silica and zinc, as well as other metallic analytes.
- Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

The following are helpful hints in the identification of the source of contaminants:

- Yellow pipette tips and volumetric caps can sometimes contain cadmium.
- Some sample cups have been found to contain lead or cobalt.
- New glassware can be a source of silica and boron.
- Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.
- Improper cleaning of glassware can cause contamination.
- Latex gloves contain over 500 ppb of zinc.

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Title: Polynuclear Aromatic Hydrocarbons by GC/MS Selected Ion Monitoring (SIM) [SW-846 Method 8270C/D/E]

Approvals (Signature/Date):

5/14/21	5/14/2021
Heather Fiedler Date	Reed Pottruff Date
Department Specialist	Health & Safety Manager / Coordinator
5/17/21	5/16/21
~~Maria Fayard~~ Thomas Lill Date	~~Scott~~ Hall Date
Quality Assurance Officer	Laboratory Director
Signing for Maria Fayard. 5/17/21	

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1.0 Scope and Application

1.1 This procedure is a Gas Chromatography/Mass Spectrometry (GC/MS) technique for the analysis of polynuclear aromatic hydrocarbons (PAH) and heterocyclic compounds at the part per trillion (ng/L or ng/kg) level in waters or solids. This procedure follows the general guidelines of EPA Methods 8270C/D/E for Selected Ion Monitoring (SIM) analysis.

1.2 The SIM technique optimizes quantitative information at the expense of qualitative information gained from other methods of analysis. It is important to note that this procedure is intended for the analysis of samples previously characterized by another method such as open-scan 8270C/D/E. The initial characterization is necessary to avoid misidentification of the parent compounds producing the ions used for this analysis.

1.3 In addition, this procedure is appropriate only for sample analytes of interest at less than 10,000 ng/L or 330,000 ng/kg. Samples containing semivolatile organics at concentrations greater than 10,000 ng/L and 330,000 ng/kg should be analyzed by a method designed to detect at higher (part per billion) levels. Samples at these levels may still be analyzed by this procedure; however, extra measurement uncertainty would be introduced because of the sample dilutions that would be required.

1.4 This procedure is applicable to water and soil samples. For water samples, 1 liter of water is extracted. It is also possible to extract 250 mL of water and analyze by an LVI (large volume injection) method designed to maintain reporting limits while reducing the initial volume of sample required for extraction. For soil samples, a sample aliquot of 30 g is extracted.

1.5 Analytes, Matrix(s), and Reporting Limits

The standard list of compounds that can be analyzed by this procedure is shown in Table IV. Typical reporting limits are 100 ng/L for aqueous samples and 5.0 μg/kg for soil samples for the PAH compounds.

2.0 Summary of Method

2.1 Sample Preparation

2.1.1 Aqueous Samples

Analytes of interest are extracted from water samples using separatory funnel extraction (EPA 3510C or 3510C_LVI) described in SOP DV-OP-0006 Extraction of Aqueous Samples by Separatory Funnel. The PAH compounds are extracted from the sample without any adjustment to pH. The concentration of organic extracts is covered in SOP DV-OP-0007 Concentration and Clean-up of Organic Extracts.

2.1.2 Solid Samples

Solid samples are extracted by sonication (EPA 3550C), which is covered in SOP DV-OP-0016 Ultrasonic Extraction of Solid Samples or by microwave extraction (EPA 3546) described in SOP DV-OP-0015 Microwave Extraction of Solid Samples. The extraction solvent is a 1:1 mixture of methylene chloride and acetone. The concentration of organic extracts is covered in SOP DV-OP-0007 Concentration and Clean-up of Organic Extracts.

2.2 Instrumental Analysis

2.2.1 Quantitation of the extracted compounds is performed by gas chromatography - mass spectrometry (GC/MS) in the selected ion monitoring mode (SIM). Routine instrument conditions and the ions used for analysis are shown in Tables I and IV, respectively.

2.2.2 Development of a successful SIM method requires identifying the ions to be monitored, the ion dwell times, the ions in each group, and the timing for switching between groups. A quantitation ion is selected with confirmation ions being monitored for identification purposes (see Table IV). Switching times are set where there is adequate resolution (a gap of 1-2 minutes) between peaks. If there is inadequate time between eluting peaks, small retention time shifts may cause peaks to partially or completely disappear as there are changes in the ions monitored. Dwell times will be set by default once the ions per group and the switching times are identified in the data acquisition method. These can be adjusted manually in order to optimize sensitivity as needed.

3.0 Definitions

3.1 Refer to Eurofins TestAmerica Denver's Quality Assurance Manual (QAM) and SOP DV-QA-003P Quality Control Program for definitions of the quality control terms used in this document.

3.2 Selected Ion Monitoring - A mass spectrometry technique that provides lower detection level capability by monitoring fewer mass scans for longer periods of time than is done in open-scan methods.

3.3 Primary Ion Area - The signal chosen for quantitation purposes.

3.4 Secondary Ion Area - The signal chosen for identification and confirmation purposes.

3.5 LVI – Large Volume Injection – An analysis method designed to maintain reporting limits while reducing the initial volume of sample required for extraction by increasing the volume of sample extract introduced onto the GC column.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. The use of high purity reagents and solvents helps to minimize interference problems.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the environment being sampled.

4.3 An interference that is unique to selected ion monitoring techniques can arise from the presence of an interfering compound which produces the same ion used for quantitation of one of the PAHs. This event results in a positive interference to the reported value for the compound of interest. This interference is controlled to some degree by acquiring data for a confirmation ion. If the ion ratios between the quantitation ion and the confirmation ion are not within the specified limits, then interferences may be present. Open scan analysis to identify compounds throughout the mass range is the most reliable assurance against reporting false positives.

4.4 Contamination by carryover can occur whenever high–level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, typically with compound concentrations well in excess of the high calibration standard, the sample analysis that immediately follows the high level sample should be evaluated for carryover. If detections are observed for the compounds that were over the calibration range in the prior sample this sample should be reanalyzed to rule out carryover unless some other objective evidence indicates that carryover is not an issue.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned

immediately. Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile or similar gloves must be used.

5.3.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.3.3 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.3.4 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect the instrument from its source of power.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Materials with Serious or Significant Hazard Rating

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm - TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm - TWA 125 ppm - STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
PAH standards can contain all or some of the following:			Standards contain low concentrations of compounds known to be or suspected to be carcinogens. All PAH compounds are considered to be hazardous, toxic, and irritants. Some or all are reported human carcinogens, mutagens, and/or teratogens.
benzo(a)anthracene	Carcinogen		
benzo(b)fluoranthene	Carcinogen		
benzo(k)fluoranthene	Carcinogen		
benzo(a)pyrene	Carcinogen		
chrysene	Carcinogen	0.2 mg/m^3 - PEL	
dibenz(a,h)anthracene	Carcinogen		
indeno(1,2,3-cd)pyrene	Carcinogen		
naphthalene		10 ppm - PEL	

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Gas Chromatograph (See Table I for operating conditions)

The analytical system includes a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for on-column injection when using packed columns and for split or splitless injection when using

capillary columns. Instruments F (Agilent 6890 with a 5973 MSD), G5 (Agilent 6890 with a 5975 MSD), and X4 (Agilent 6890 with a 5973 MSD) may be used for this analysis. Equivalent instruments may be used.

6.1.2 Mass Spectrometer (See Table I for operating conditions)

A mass spectrometer operating at 70 eV (nominal) electron energy in the electron impact ionization mode and tuned to maximize the sensitivity of the instrument to the compounds being analyzed. The GC capillary column is fed directly into the ion source of the mass spectrometer.

6.1.3 A computer system interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. The computer allows acquisition at pre-selected mass windows for selected ion monitoring.

6.1.4 Please refer to the Master List of Documents, Software, and Hardware (or current revision) located on R:\QA\Read\Master List of Documents for the current software and hardware to be used for data processing.

6.2 Supplies

6.2.1 All glassware used, both within the scope of this SOP and for the initial sample extraction (see SOPs DV-OP-0006 Extraction of Aqueous Samples by Separatory Funnel, DV-OP-0007 Concentration and Clean-up of Organic Extracts, DV-OP-0015 Microwave Extraction of Solid Samples, and DV-OP-0016 Ultrasonic Extraction of Solid Samples), must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. This should be followed by detergent washing with hot water, and rinses with tap water, reagent water, and finally with acetone.

6.2.2 Glassware should <u>not</u> be oven dried or heated in a muffle furnace. Successive solvent rinses of the separatory funnel, sonication, and Kuderna-Danish glassware are required to minimize low level contamination of samples.

6.2.3 Store glassware inverted or in sealed containers capped with aluminum foil.

6.2.4 Gas-tight syringes, various sizes, and SMI pipettors.

6.2.5 Serological pipettes are used for final extract volume measurement.

6.2.6 Micro reaction vessels, 1.8 mL vials with Teflon caps, for storing concentrated extracts.

6.2.7 Column – A Varian VF-5MS 30-meter fused silica capillary column, 0.5 μm film thickness, 0.25 mm ID, plus 10-meter EZguard, or equivalent.

6.2.8 Agilent Ultra Inert splitless single taper liners.

6.2.9 Amber crimp cap vials with Sil/PTFE aluminum seals.

6.2.10 Hamilton 10 μL autosampler syringes.

7.0 Reagents and Standards

7.1 Reagents

All solvents are reagent grade or higher unless specified otherwise. See SOP CA-Q-S-001 Acid and Solvent Testing and Approval Program for a description of the program for testing solvents prior to use. The manufacturer expiration applies to all solvents and when not specified by the manufacturer the expiration will be recorded as one year after opening the solvent for use.

7.1.1 Methanol, reagent grade.

7.1.2 Methylene chloride, reagent grade.

7.1.3 Helium gas, 99% + purity.

7.2 Standards

Commercial standards are received in flame-sealed ampoules or as neat, 100% concentration solutions. Standards are verified before use. Details concerning verification of standards are given in SOP DV-QA-0015 Verification and Storage of Chemical Standards and Reagents. Stock standards are stored refrigerated at ≤6°C. All stock standards must be protected from light. Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually from the date of opening or earlier, if the vendor indicates an earlier date. Dilutions or working level standards that are prepared from stock standards are assigned an expiration date according to the earliest expiring stock or one year from the date of preparation, whichever date is earlier.

For the PAH compounds and the additional compounds that are mentioned in this SOP the following stock standards are currently used: **MS-48925** Supelco cat. # 48925 at 1,000 µg/mL (surrogates), **MS-31009** Restek cat. # 31009 SV Calibration Mix #3 at 2,000 µg/mL, **MS-31010** Restek cat. # 31010 SV Calibration Mix #4 at 2,000 µg/mL (has 2-methyl naphthalene), **MS-31853** Restek cat. # 31853 at 2,000 µg/mL (1,4-Dioxane), **MS-31995** Restek cat. #31995 8270 Calibration Mix #5 at 2,000 µg/mL (has all PAH compounds including 2 methyl naphthalene), **MS-APP914820X** Accustandard cat. #APP-9-148-20x at 2,000 µg/mL (n-nitrosodiethylamine), and **MS-47643-U** Supelco cat. # CRM47643 8270 Ether/Phthalate mix at 2,000 µg/mL. Other vendors and mixes may be substituted for these stocks but an NCM must be written for the SOP deviation.

7.2.1 GC/MS Tuning Standard

A methylene chloride solution containing decafluorotriphenylphosphine (DFTPP) at a concentration of 50 µg/mL (25 µg/mL for LVI) is prepared by diluting 0.5 mL of the stock to a final volume of 10ml. The current vendor for the tuning standard is Supelco cat. # 47548-U at a concentration of 1,000 µg/mL.

7.2.2 Calibration Standards

Calibration standards for the initial calibration (ICAL) are prepared at 7 concentrations to cover the calibration range by diluting vendor stock standard solutions using methylene chloride. The standards are prepared directly in autosampler vials by using syringes to deliver the appropriate volumes of stock standard solution, internal standard solution, and methylene chloride. The following tables summarize a typical set of calibration standards:

MS-SIM SL_Stk is a 200 µg/mL calibration substock that is prepared by diluting 0.5 mL of **MS-31009, MS-31010, MS-31853, MS-31995, (and MS-APP914820X)** to a final volume of 5 mL.

Standard Method: Prepared using a PAH SIM stock standard **MS-SIMSL** with a concentration of 20 µg/mL for levels 4 through 7. The **MS-SIMSL** standard is prepared by diluting 0.2 mL of **MS-48925** (surrogates) and 1 mL of **MS-SIM SL_Stk** and 0.1 mL of **MS-47643-U** to a final volume of 10 mL. A secondary PAH SIM stock standard **MS-SIMSL Int** prepared by diluting 1 mL of the **MS-SIMSL** to a final volume of 10 mL with a concentration of 2 µg/mL is used to prepare levels 1 through 3:

Vol Stock (µL)	Methylene Chloride (µL)	Internal Standard (µL)	Final Volume (µL)	Conc PAH (µg/mL)
5	495	50	500	0.02
25	475	50	500	0.1
75	425	50	500	0.3
15	485	50	500	0.6
30	470	50	500	1.2
62.5	437.5	50	500	2.5
125	375	50	500	5.0

LVI Method: Prepared using a PAH SIM stock standard with a concentration of 20 µg/mL for levels 6 and 7. A secondary PAH SIM stock standard with a concentration of 2 µg/mL is used to prepare levels 1 through 5:

Vol Stock (µL)	Methylene Chloride (µL)	Internal Standard (µL)	Final Volume (µL)	Conc PAH (µg/mL)
1	499	50	500	0.004
5	495	50	500	0.02
15	485	50	500	0.06
30	470	50	500	0.12
60	440	50	500	0.24
12.5	487.5	50	500	0.5
25	475	50	500	1.0

7.2.3 Initial Calibration Verification (ICV) Standard (MS-SIM SSV)

A second source initial calibration verification (ICV) standard is prepared using a standard solution that is obtained from a source independent from the source that supplies the standard used for the initial calibration. It is prepared by diluting 30 µL of a substock that is at a concentration of 20 µg/mL to a final volume of 0.5 mL. The final PAH SIM concentration for this ICV standard is 1.2 µg/mL (0.24 µg/mL for LVI).

The substock for **MS-SIMSSV** (above) is prepared by diluting 1 mL of another substock, **MS-HSLB1_STK,** to 10 mL final volume.

MS_HSLB1_STK is prepared by diluting 2 mL of **MS-570666.SEC** (Restek cat. # 570666.sec 8270 List 1/Std#1 Mega Mix at 500, 1,000, 2,000 µg/mL) and 2 mL of **MS-569731SEC** (Restek cat. #769731.sec 8270 List 1/Std #10 at 2,000 µg/ml) to a final volume of 10 ml. The final concentration of this stock varies as either 200 µg/mL or 400 µg/mL depending upon the compound.

The final PAH SIM concentration for this ICV standard is 1.2 µg/mL (0.24 µg/mL for LVI).

7.2.4 Continuing Calibration Verification (CCV) Standard

A standard with the same analytes and concentrations as the 600 ng/mL (120 ng/mL for LVI) calibration standard. The standard may be from the same preparation as the initial calibration or prepared at a later date.

7.2.5 Surrogate Spiking Solutions (8270SIM Surr)

The surrogate spike solution contains neutral surrogates at concentrations of 500 ng/mL in methanol. It is prepared by diluting 0.1 mL of **8270SurrStkHL** (Restek cat. #567685 at 5,000 µg/mL) to a final

volume of 1,000 mL with acetone. Table II lists the surrogate compounds for the standard list of PAHs.

- 1.0-liter water extractions, add 1.0 mL of the surrogate spike solution
- 250-mL LVI water extractions, add 0.250 mL of the surrogate spike solution
- 30-gram soil sample extractions, add 1.0 mL of the surrogate spike solution

7.2.6 Internal Standard (IS) Solutions (MS-SIM IS)

A 6,000 ng/mL solution of the internal standards is prepared in methylene chloride from vendor stock **MS-57604** (Restek cat. #567684 8270 Internal Standard at 2,000 µg/mL) by diluting 60 mL of this stock to 300 mL final volume. Then 1.5 mL of this stock, **MS-IS**, is diluted to 100 mL to yield the **MS-SIMIS** spiking solution. Table III lists the IS compounds.

To each sample extract, 20 µL of the respective IS solution is added to a 200 µL aliquot of the sample extract for both standard (1 L sample) and LVI extracts.

7.2.7 LCS, MS, and MSD Spike Solution (8270BO-SIMLCS)

A methanol solution containing the requested spike compounds at a concentration of 900 ng/mL each is prepared from the vendor stock solution by diluting 0.225 mL of **MS-570666** (Restek cat. #570666 HSL Mega Mix at 1,000 µg/mL) to a final volume of 250 mL with P&T methanol. Following are the final sample concentrations of the spiked compounds for the water and solid extractions:

- 1.0-liter water extractions, add 1.0 mL of the spike solution, [PAH] = 900 ng/L
- 250-mL LVI water extractions, add 0.250 mL of the spike solution, [PAH] = 900 ng/L
- 30-gram soil sample extractions, add 1.0 mL of the spike solution, [PAH] = 30 µg/kg

7.3 All stock and working standards are stored according to the manufacturer's instructions. Dilutions from stocks may not be assigned expiration dates that exceed the stock standard expiration date set by the manufacturer.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Sample Amounts

8.1.1 Water samples are collected in pre-cleaned amber glass bottles fitted with a Teflon-lined cap. To guarantee the ability to meet routine reporting limits, two full bottles of sample should be provided. Additional bottles are

needed to satisfy the requirements for matrix spikes and duplicate matrix spikes. For the standard method, each bottle should be 1.0 L; for the LVI method, each bottle should be 250 mL.

8.1.2 Soil samples are collected in an 8-ounce, pre-cleaned, wide-mouth jar with a Teflon-lined lid.

8.2 Samples are chilled to a temperature between 0 and 6 °C immediately after collection and shipped via overnight carrier to the laboratory.

8.3 Samples and excess sample volume must be stored refrigerated at ≤ 6 °C from when the log-in process is completed (see SOP DV-QA-0003 Sample Management and Chain of Custody) to storage after analysis.

8.4 Water samples must be extracted within 7 days of the time of sample collection, while solid samples must be extracted within 14 days of sampling. Extracts must be analyzed within 40 days from the start of the sample extraction.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the Eurofins TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in Eurofins TestAmerica Denver Policy DV-QA-003P Quality Control Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), and Department of Energy (DOE), are described in Eurofins TestAmerica Denver Policy SOP DV-QA-024P QA/QC Requirements for Federal Programs.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 Non-Conformance and Corrective Action System. This is in addition to the corrective actions described in the following sections.

9.2 Method Blank (MB)

A method blank is processed and analyzed with each analytical batch, not to exceed 20 samples. For aqueous samples, the method blank consists of reagent water spiked with surrogates. For soil samples, the method blank is Ottawa sand spiked with surrogates. This sand is mixed with sodium sulfate for extraction by ultrasonication. Method blanks are used to assess whether the laboratory has contributed contamination to the sample analysis process that adversely affects the accuracy of the determination of target analytes. The goal is to have no detectable contaminants in the method blank. However, due to the sensitivity of this analysis, it is not uncommon to detect target analytes at levels above the method detection limit (MDL).

Acceptance Criteria: MB results must be less than ½ the reporting limit.

Corrective Action: If the MB exceeds ½ the RL for any target analyte, then one of the following must apply for acceptance of the batch:

The blank contamination is less than $^{1}/_{10}$ of the measured concentration of any sample in the associated preparation batch, or

The blank contamination is less than the concentration present in the samples and is less than $^{1}/_{10}$ of the regulatory limit, or

The same contaminants are <u>not</u> found in the associated samples.

NOTE: Positive method blank results below the reporting limit should be evaluated by the analyst for potential impact on sample results at or near the reporting limit.

9.3 Laboratory Control Samples (LCS)

A Laboratory Control Sample (LCS) is processed and analyzed with each analytical batch not to exceed 20 samples. For aqueous samples, the LCS consists of reagent water spiked with the analytes of interest and surrogates. For soil samples, the LCS is Ottawa sand spiked with analytes of interest and surrogates. For ultrasonic extraction, sodium sulfate is added to the reagent sand. The LCS spiking solution is described in Section 7.2.7. LCS results are used to determine whether the analytical system is in control. Depending on project requirements, a duplicate LCS (LCSD) may be required to assess the precision of the analytical system.

Acceptance Criteria: The percent recovery for each requested target analyte in the LCS must fall within the established control limits (found in TALS).

Note: SC DHEC compliance samples must recover within limits of 70-130%.

Corrective Action: If the percent recovery for any requested analyte in the LCS exceeds the upper control limit and the analyte is not detected in any of the associated samples, then no further action is required, and data are reported with an NCM.

If the percent recovery for any analyte in the LCS exceeds the upper control limit and the analyte is detected in any of the associated samples, then reanalyze the LCS. If similar results are obtained on the second attempt, then investigate and correct any problems. Re-extract and reanalyze the preparation batch.

If the percent recovery for any analyte in the LCS is below the lower control limit, reanalyze the LCS. If similar results are obtained on the second attempt, then investigate and correct any problems. Re-extract and reanalyze the preparation batch.

If re-extraction of samples is not possible or the client requests the samples not be re-extracted, qualify data and explain in a NCM.

9.4 Matrix Spike and Spike Duplicate (MS/MSD)

One matrix spike (MS) sample and one matrix spike duplicate (MSD) sample are prepared and analyzed for each preparation batch. An MS sample is a field sample to which known amounts of the target analytes, as well as the surrogates, have been added. An MSD is a second aliquot of the same sample that is spiked the same as the MS. The MS/MSD spiking solution is described in Section 7.2.7. MS results are used to assess the effects of the sample matrix on the accuracy of the analytical system. The MSD results are used to assess the effects of the sample matrix on the precision of the analytical system. Given the expected variability in sample matrix, the MS/MSD results are applicable to only the sample used to prepare the MS and MSD. MS/MSD results should not be extrapolated to other samples without extensive investigation and characterization to demonstrate similarity between samples. The DoD QSM requires that the MS/MSD be prepared from samples from the same site.

Acceptance Criteria: The MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results must be within the established control limits. Percent recovery control limits are set at ± 3 standard deviations around the historical mean of the LCS recovery data, unless otherwise dictated by the client or project. The RPD control limit is set at 3 standard deviations above the mean of the historical data.

NOTE: DOD QSM limits apply to projects performed under this program.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind

that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.

- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

9.5 Internal Standards

The internal standards listed in Table III and described in Section 7.2.6 are spiked at the same level in all field sample extracts, QC sample extracts, instrument blanks, and calibration standards.

Acceptance Criteria: The peak area for each internal standard in each field sample and QC sample extract should be between 50% and 200% of the peak area for the same internal standard in the midlevel standard of the initial calibration.

Corrective Action: If the internal standard fails acceptance criteria, then perform the following corrective actions:

- Inspect system for malfunction and correct as needed.
- Reanalyze the affected samples.
- If the interference cannot be corrected for field samples, the earlier analysis is reported with discussion in an NCM.
- If QC samples have internal standard failures that are confirmed by re-analysis, the cause of the failures must be investigated.

9.6 Surrogate Compound Analysis

Surrogate compounds listed in Table II and described in Section 7.2.4 are added to all field and QC samples prior to extraction. Surrogate recoveries are used to assess individual sample matrix effects on sample preparation and analysis.

Acceptance Criteria: Surrogate recoveries must fall within established control limits. QC sample results are not acceptable unless the surrogate recoveries for those samples are in control.

Corrective Action: Corrective action must be considered for any surrogate failure and may depend on project-specific instructions. Lacking instructions to the contrary the following actions shall be taken:

- Evaluate sample chromatogram and other QC.
- If the surrogate(s) fail in the LCS and/or method blank, then re-prepare and reanalyze all associated samples. Samples may be excepted where the surrogate recovers high in the MB and the MB does not have detection above ½ of the RL. Likewise, if the surrogate is out of control in the LCS but the LCS compounds recover in control then the samples may be reportable but the program requirements must be checked to see if this is acceptable. In any case an NCM must be written to describe the situation.
- For surrogate failures in field samples, re-prepare and reanalyze the samples, unless matrix interference is evident from earlier analysis or from chromatograms in which case the samples are reported with an NCM.

9.7 Instrument QC

9.7.1 Instrument Optimization

9.7.1.1 The GC/MS system must be tuned to meet manufacturer's specifications, using a suitable calibration such as perfluorotri-n-butylamine (FC-43). This is performed through the auto-tune feature in the software. The mass calibration and resolution of the GCMS system is then verified by the analysis of DFTPP prior to the analysis of any standards or samples. In some instances the laboratory will opt to omit the DFTPP. The DFTPP tune check is less useful for SIM analysis than it is for full scan analysis because the DFTPP analysis must necessarily be done in full scan mode. When this check is omitted, the FC-43 check will be performed daily.

9.7.1.2 The instrument is tuned for DFTPP (decafluorotriphenylphosphine), calibrated initially with a seven-point calibration curve, and verified each 12-hour shift that samples are to be run with one or more continuing calibration verification (CCV) standard(s).

9.7.2 Instrument Tuning

At the beginning of every 12-hour shift when analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria (Table VI) are achieved for DFTPP.

9.7.2.1 Inject 1 µL of the 50 µg/mL GC/MS tuning standard (see Section 7.2.1) into the GC/MS system.

9.7.2.2 The mass spectrum of the DFTPP must be obtained in the following manner: three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is also required, and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of the DFTPP. Do not subtract part of the DFTPP peak. A procedure compliant with these requirements is programmed into a Macro used to evaluate the DFTPP spectrum. Confirm that all the key m/z criteria in Table VI are achieved.

9.7.2.3 If all the criteria are not achieved, the analyst must adjust or retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

9.7.3 Initial Calibration (ICAL)

9.7.3.1 A new calibration curve must be generated initially, after major changes to the system, or when continuing calibration criteria cannot be met. Major changes include installation of new columns and source maintenance.

9.7.3.2 A minimum five-point initial calibration curve must be established for linear fit calibrations (weighted or unweighted). Six points or more are required for second order curve fits. See Section 9.7.4 for Calibration Acceptance Criteria.

- The concentrations of standards commonly used to construct the PAH calibration curve are 20, 100, 300, 600 (often analyzed before the rest of the standards and called the ICIS), 1,200, 2,500, and 5,000 ng/mL.

- For the LVI method, the concentrations of standards commonly used to construct the PAH calibration curve are 4, 20, 60, 120 (often analyzed before the rest of the standards and called the ICIS), 240, 500, and 1,000 ng/mL.

9.7.3.3 If the concentration of any target compound in a sample exceeds the calibration range, the extract must be diluted with methylene chloride so that the concentrations of all target compounds fall within the range of the calibration curve, and be reanalyzed. Any samples analyzed immediately following the sample that exceeded the linear range may require reanalysis due to possible carryover from the high-level sample.

9.7.3.4 Generally, it is NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or the linear range is supported or adjusted accordingly. The only exception is that a level may be removed from the calibration if the reason can be clearly documented, for example a broken vial. A minimum of five levels must remain in the calibration. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 12 hours.

9.7.3.5 Calculate the response factor (RF) for each analyte for each calibration standard level as described in Section 11.3. Calculate the mean RF and relative standard deviation (RSD) for each analyte.

9.7.4 Calibration Acceptance Criteria and Corrective Action:

Acceptance Criteria 8270C:

The RSD of the initial calibration for each analyte of interest must be ≤ 35%.

Acceptance Criteria 8270D:

Refer to Table VII for the acceptance criteria for minimum response factor and RSD. Two target compounds and surrogates may fail to meet the minimum RRF criteria listed in Table VII but must still meet the minimum RRF criteria of 0.010 (excluding compounds with a minimum RRF requirement of 0.010). In addition, two target compounds and surrogates may fail to meet the RSD criteria listed in Table VII but must still meet the maximum RSD requirement of 40% (excluding compounds with a maximum RSD requirement of 40%). Refer to SOP DV-QA-024P QA/QC Requirements for Federal Programs for requirements for federal programs.

Acceptance Criteria for DoD:

For DoD QSM specific criteria see SOP DV-QA-024P QA/QC Requirements for Federal Programs.

Corrective Actions:

If these criteria cannot be met, least-squares weighted or unweighted linear regression may be used to establish a calibration function as described in Section 11.4. In this case, the correlation coefficient (r) must be greater than 0.995 (equivalent to $r^2 \geq 0.99$) or a second-order regression fit with coefficient of determination (COD, r^2) greater than 0.99 may be used. If these linearity criteria are not achieved, verify the standard preparation and instrument conditions, and then recalibrate the instrument. If technical acceptance criteria are not met, it may be necessary to clean the ion source, perform injector maintenance, change the column, or take other corrective actions.

Note: Quadratic regression may not be used, all target analytes and surrogates must pass the minimum RF criteria per the method, and percent error or relative standard error (RSE) checks must be performed with each ICAL per EPA 8000D requirement for SC DHEC compliance samples.

9.7.5 In the event that a least-squares regression is used, the analyst should evaluate the bias at the lower portion of the curve. This can be accomplished by re-fitting the low point standard back into the curve. The recalculated concentration should be within ± 50% of the standard's true concentration. If these criteria are not met, the analyst may have to evaluate the concentration range of the standards, or the lower limit of quantitation.

9.8 Initial Calibration Verification (ICV)

The Initial Calibration Verification (ICV) is a second-source, mid-level standard that is analyzed immediately following the initial calibration standards.

Acceptance Criteria: The absolute value of the difference between the measured PAH analyte concentration and the true value must be ≤ 30 %. For DoD QSM specific criteria see SOP DV-QA-024P QA/QC Requirements for Federal Programs.

Corrective Action: If the ICV recovery fails, then take the following actions:

- Verify standard preparation, and if incorrect, re-prepare the ICV standard solution.
- If preparation of the ICV standard was correct, then re-prepare the initial calibration standards and recalibrate.

9.9 Continuing Calibration Verification (CCV)

Every 12 hours, the mass spectrometer response for each PAH relative to the internal standard is determined by analyzing a 600 ng/mL calibration standard (120 ng/mL for the LVI method). The RF for each compound in the continuing calibration verification (CCV) analysis is compared to the RF for that compound in the ICAL.

9.9.1 Acceptance Criteria 8270C

The absolute value of the difference between the CCV RF for each PAH analyte and the corresponding ICAL value must be ≤ 35 %.

9.9.2 Acceptance Criteria 8270D

The absolute value of the difference between the CCV RF for each PAH analyte and the corresponding ICAL value must meet the criteria in Table VII. The compounds must also meet the minimum response factor criteria listed in Table VII. Two target compounds and surrogates may fail to meet the minimum RRF criteria in Table VII (excluding compounds with a minimum RRF requirement of 0.010) but must still meet the minimum RRF criteria of 0.010. In addition, two target compounds and surrogates may fail to meet the difference criteria in Table VII (excluding compounds with a maximum percent difference requirement of ± 40%) but must still meet the maximum difference requirement of ± 40%. Refer to SOP DV-QA-024P QA/QC Requirements for Federal Program for requirements for federal programs.

Note: All target analytes and surrogates must pass the minimum RF criteria and pass the %D criteria of EPA 8270D for SC DHEC compliance samples.

9.9.3 Acceptance Criteria for DoD QSM

For DoD QSM specific criteria see SOP DV-QA-024P QA/QC Requirements for Federal Programs.

9.9.4 Acceptance Criteria 8270C & 8270D

9.9.4.1 The internal standard response of the CCV must be within 50 - 200% of the internal standard response in the mid-level (ICIS) of the most recent ICAL sequence.

9.9.4.2 The internal standard retention time must be within ± 30 seconds of the internal standard retention time in the corresponding level of the most recent ICAL sequence.

9.9.5 Corrective Action:

9.9.5.1 If, for any analyte, the CCV RF does not meet the stipulated acceptance criteria, a five-point calibration curve must be repeated for that analyte prior to the analysis of samples.

9.9.5.2 If any internal standard retention time in the CCV changes by more than 30 seconds from that of the corresponding level of the most recent ICAL sequence, the chromatographic system must be inspected for malfunctions and corrections made, as required.

9.10 Closing CCV (DoD QSM only)

DoD QSM 5.0 and subsequent versions requires a closing CCV, injected within 12 hours of the DFTPP injection. See SOP DV-QA-024P QA/QC Requirements for Federal Programs for specific criteria.

9.10.1 Acceptance Criteria

All reported analytes and surrogates must be within ± 50%.

9.10.2 Corrective Action

Recalibrate and reanalyze all affected samples since the last acceptable CCV

Or

Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails perform column maintenance and recalibrate; then reanalyze all affected samples sine the last acceptable CCV.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 Non-Conformance and Corrective Action System. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

10.3.1 Aqueous Sample Extraction and Concentration

10.3.1.1 Instructions for the extraction of aqueous samples may be found in SOP DV-OP-0006 Extraction of Aqueous Samples by Separatory Funnel.

10.3.1.2 Instructions for the concentration of extracts may be found in SOP DV-OP-0007 Concentration and Clean-up of Organic Extracts.

10.3.2 Soil Sample Extraction and Concentration

10.3.2.1 Instructions for the ultrasonic extraction of soil samples may be found in SOP DV-OP-0016 Ultrasonic Extraction of Solid Samples.

10.3.2.2 Instructions for the microwave extraction of soil samples may be found in SOP DV-OP-0015 Microwave Extraction of Solid Samples.

10.3.2.3 Instructions for the concentration of extracts may be found in SOP DV-OP-0007 Concentration and Clean-up of Organic Extracts.

10.4 Sample Analysis

10.4.1 All aliquotting, extract dilutions, and spike additions must be performed in the trace fume hood using equipment dedicated to PAH-SIM analysis. An aliquot of 200 µL of each sample extract is placed into a two-milliliter GC/MS autosampler vial. Sufficient volume of extract remains should reanalysis be necessary.

10.4.2 Prior to analysis, 20 µL of internal standard is added to the sample vial giving a final internal standard concentration of 600 ng/mL (150 ng/mL for LVI) in the extract.

10.4.3 Representative aliquots are injected into the gas chromatograph/mass spectrometer using similar conditions to those summarized in Table I. The injection volume is 1 µL (5 µL for LVI).

10.4.4 Whenever an unusually concentrated sample is encountered, it may be necessary to reanalyze the subsequent sample extracts after analyzing an instrument blank to demonstrate that there is no cross contamination.

10.4.5 The following is a typical analytical sequence:

- Solvent rinses, as needed
- MS tune
- ICAL plus ICV or CCV
- Instrument blank

- MB, LCS
- LCSD (if requested by client)
- Sample extracts
- MS and MSD are interspersed with sample extracts, and usually run after the sample from which they are produced.
- The last sample extract must be injected within 12 hours of the tune.

10.4.6 The sequence may be altered to accommodate reanalysis or additional instrument blank and calibration evaluations. At a minimum, an instrument blank or a method blank shall be included in the sequence. Refer to Policy DV-QA-003P Quality Control Program for additional details.

10.4.7 The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the selected ion monitoring (SIM) mode using appropriate windows to include the quantitation and confirmation masses for each analyte as shown in Table IV.

10.4.8 All compounds detected at concentrations above the method MDL are checked to ensure that the confirmation ion is present at the appropriate ratio.

10.4.9 All compounds detected at concentrations above the highest calibration standard require dilution and reanalysis. In addition, any samples that were analyzed immediately following a high-level sample should be reanalyzed to rule out carryover from the high-level sample, unless they are preceded by an acceptable instrument blank or the high compound(s) were not detected in the subsequent samples.

10.4.10 Manual Integrations

10.4.10.1 Upon completion of the analytical sequence, transfer the raw instrument data to Chrom for further processing. Review the chromatograms to ensure correct assigning of peaks and correct integration of each peak.

10.4.10.2 Note that certain compounds (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene) may require frequent manual integrations. Special attention must be exercised by the analyst and secondary reviewer for compounds that are commonly mis-integrated in automated software or are manually integrated. If manual data manipulations are necessary, they must be justified and documented. See DV-QA-011P Acceptable Manual Integration Practices.

10.5 Troubleshooting and Maintenance

10.5.1 Daily Instrument Maintenance

In addition to the checks listed in Appendix II, the following daily maintenance should be performed.

- Clip column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Install new or cleaned gold seal and washer as necessary.
- Perform mass calibration as necessary.

10.5.2 Major Maintenance

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the repeller, cleaning the source, replacing the multiplier, and replacing the “topboard” or RF-related electronics. Refer to the manufacturer's manual for specific guidance.

11.0 Calculations / Data Reduction

11.1 Qualitative Identification

Obtain electronic ion current profiles (EICP) for the primary mass ion and the confirmatory ion for detected compounds. The following criteria must be met to make a qualitative identification:

11.1.1 The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.

11.1.2 The retention time (RT) of unknown peaks must fall within ± 0.2 minutes of the RT for the compound in the daily calibration standard (mid-point ICAL or daily CCV).

11.1.3 The relative peak areas of the primary ion compared to the confirmation or secondary ion masses in the EICPs must fall within ± 20% of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library. A compound that does not meet secondary ion confirmation criteria may still be determined to be present in a sample after close inspection of the data by the mass spectroscopist. Supportive information includes correct relative retention time (RRT) and the presence of the secondary ion, but the ratio falls outside of ± 20% of the primary ion, which may be caused by an interference of the secondary ion.

11.1.4 Structural isomers that have very similar mass spectra and less than a 30-second difference in retention time can be explicitly identified only if

the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if there is a definitive inflection between the two peaks, according to the analyst's judgment. Otherwise, structural isomers are identified as isomeric pairs.

11.2 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-P-003 Calibration Curves and the Selection of Calibration Points and the public folder, *Arizona Calibration Training*.

11.3 Average Response Factor Calibration

The following formula is used to calculate the response factor for each analyte of interest relative to the applicable internal standard for each of the calibration standards:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s	=	Area of the characteristic ion for the target analyte in the calibration standard
A_{is}	=	Area of the characteristic ion for the internal standard
C_{is}	=	Concentration of the internal standard, (ng/mL)
C_s	=	Concentration of the target analyte in the calibration standard (ng/mL)

The calibration uses the average response factor for each target analyte, which is calculated as follows:

$$\text{average (mean) RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Where:

RF_i	=	Response factor for the i^{th} calibration level
n	=	Number of calibration levels

The standard deviation for the mean RF for each target analyte is calculated as follows:

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

The relative standard deviation (RSD) for the average response factor for each target analyte is calculated as follows:

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

The concentration of each target analyte in the sample extract is calculated using the average response factor that was calculated in the equation above as follows:

$$C_e = \frac{A_e \times C_{is}}{A_{is} \times \overline{RF}}$$

Where:

C_e	=	Concentration of target analyte in the sample extract, ng/mL
A_e	=	Area of the characteristic ion for the target analyte in the sample extract.
A_{is}	=	Area of the characteristic ion for the internal standard
C_{is}	=	Concentration of the internal standard, (ng/mL)
$\overline{RF}$	=	Average response factor for the target analyte as determined by calibration

11.4 Linear Least-Squares Regression Calibration (Unweighted)

A linear least-squares regression is performed using the concentration of the target analyte in the calibration standard as the independent variable (x) and the instrument response as the dependent variable (y). The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = mx + b$$

Where:

y	=	instrument response (e.g., peak area)
x	=	concentration of target analyte in calibration standard
m	=	slope of the line
b	=	intercept of the line

For the internal standard calibration, the regression equation is rewritten as follows:

$$\frac{A_s C_{is}}{A_{is}} = mC_s + b$$

Where:

A_s	=	Area of the characteristic ion for the target analyte in the calibration standard
A_{is}	=	Area of the characteristic ion for the internal standard
C_s	=	Concentration of the target analyte in the calibration standard, (ng/mL)
C_{is}	=	Concentration of the internal standard, (ng/mL)
m	=	slope of the line
b	=	intercept of the line

The concentration in an unknown extract is then calculated by rearranging the calibration equation as follows:

$$C_e = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{m}$$

Where C_e is the concentration of the target analyte in the sample extract, and A_e is the area of the characteristic ion for the target analyte in the sample extract.

The actual sample concentration (C) for each compound is calculated as follows:

$$C = C_e \times \left(\frac{V_e}{V_o}\right) \times DF$$

Where:

C	=	Concentration of the target analyte in the original sample, ng/L (water sample) or ng/kg (solid sample)
C_e	=	Concentration of the target analyte in the sample extract, ng/mL
V_e	=	Final extract volume, mL.
V_o	=	The original volume or weight of the sample that was extracted, in L (aqueous sample) or kg (solid sample).
DF	=	Dilution factor, if appropriate.

11.5 Additional Regression Calibration Models

As needed, weighted linear least-squares or second order regressions may be utilized for this analysis. See Corporate SOP CA-Q-P-003 Calibration Curves and the Selection of Calibration Points and the public folder, *Arizona Calibration Training*, for calculations and further explanations.

11.6 A second-level technical review of the organic data is performed prior to data reporting. This review is performed by a peer or supervisor using the guidelines and checklists detailed in SOP DV-QA-0020 Data Review.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-S-006 Detection and Quantitation Limits. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024 Training.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024 Training.

12.4 Retention Time Study

12.4.1 Expected absolute retention times (RTs) are initially determined by analyzing all target analytes in the open-scan mode. Example RTs are listed in Table V.

12.4.2 Relative retention times (RRTs) are then calculated for samples in each analytical run based on the RTs found in the continuing calibration verification standard (CCV).

12.4.3 RTs are re-established after any significant instrument maintenance, including source cleaning and changing columns, or whenever compounds are not adequately detected in CCVs or LCSs.

13.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Environmental Health and Safety Manual, and DV-HS-001P Waste Management Plan.

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

14.2.2 Methylene chloride solvent rinse waste – Waste Stream B

14.2.3 Expired extract vial waste – Waste Stream A

14.2.4 Radioactive and potentially radioactive waste must be segregated from non-radioactive and mixed waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Soil Waste Physical/Chemical Methods (SW-846), Third Edition, September 1986, Final update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final update IIB, January 1995; Final Update III, December 1996, Final Update IV January 2008.

15.1.1 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

15.1.2 Method 8000C, Determinative Chromatographic Separations, Revision 2, February 2007.

15.1.3 Method 8000D, Determinative Chromatographic Separations, Revision 5, March 2018.

15.1.4 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 3, December 1996.

15.1.5 Method 8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 4, February 2007.

15.1.6 Method 8270E, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 6, June 2018.

15.1.7 Method 3510C, Separatory funnel Liquid-Liquid Extraction, Revision 3, December 1996.

15.1.8 Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996.

15.1.9 Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

15.1.10 Method 3546, Microwave Extraction, Revision 0, February 2006.

15.2 CLP Statement of work for Multi-Media, Multi-Concentration Organics Analysis, SOM01.2. June 2007.

16.0 Method Modifications

16.1 The CLP SOW referenced in 8270D does not require the analysis of DFTPP prior to the analysis of samples. The method relies on the successful analysis of calibration standards to verify acceptable function of the mass spectrometer. Eurofins TestAmerica Denver utilizes the DFTPP check to identify any operational issues with the mass spectrometer prior to the analysis of the calibration standards. This allows the analyst to identify possible problems independent of the GC. As a result, the laboratory will start the 12 hour clock with the injection of the DFTPP, not the calibration standard as required in the method.

16.2 Method 8270C serves as the basis for this SOP, but the method has been modified extensively for low-level analysis using selected ion monitoring (SIM) and optimizing instrument conditions for the low-level analysis. Consequently the sensitivity of the method has been enhanced and it is not uncommon to detect low-level contamination in the method blank at levels well below the limits of detection for the less sensitive GC/MS method. For example, Method 8270C states that the RSD of the initial and continuing calibration must be less than or equal to 15% and 20% respectively. Due to the low-level nature of the analysis, this SIM procedure allows both of these criteria to be less than or equal to 35%.

16.3 Method 8270C stipulates qualitative identification based on relative retention time (RRT), which is calculated by dividing the retention time (RT) of the target analyte by the RT of the internal standard. The RRT of the suspected target analyte in the sample extract must be within ± 0.06 RRT units of the RRT for that analyte in the calibration standard. This SOP stipulates qualitative identification based on an absolute RT. Namely the RT of the suspected target analyte in the sample extract must be within ± 0.2 minute of the RT for that analyte in the calibration standard. Additionally, the RT for the internal standard in the sample extract must also be within ± 0.2 minute of the RT for the internal standard in the calibration standard. The criteria used in this SOP are more restrictive than those imposed by the referenced method. For the earliest eluting compounds, the RT for the internal standard is typically 8 minutes. The earliest eluting target analyte must be at a RRT of at least 0.8, which translates to a RT of 6.4 minutes. Assuming a worst-case scenario where the RT of the internal standard is 0.2 minute higher (i.e., 8.2

minutes) and the RT of the target analyte is 0.2 minute lower (i.e., 6.2 minutes), the calculated RRT is 0.76. The total deviation from the expected RRT is 0.04 RRT units, which is smaller than what is allowed by Method 8270C.

17.0 Attachments

Table I:	Routine Instrument Operating Conditions
Table II:	Surrogates for Standard List Analysis
Table III:	Internal Standards for Standard List Analysis
Table IV:	PAH Compounds and Ions Used for Analysis
Table V:	Example Retention Times, IS and Surrogate Associations
Table VI:	DFTPP Key Ions and Ion Abundance Criteria for 8270C and 8270D
Table VII:	8270D Relative Response Factor Criteria for Initial and Continuing Calibration
Appendix I:	Extended List PAHs
Appendix II:	Suggested Instrument Maintenance Schedule – Mass Spectrometer & Gas Chromatograph
Appendix III:	Mass Spectrometer Settings for Single Ion Monitoring

18.0 Revision History

This section has been added beginning with Revision 0. Only details of the last two revisions are incorporated into this SOP. Prior revisions are documented in the QA files and available upon request.

- Revision 16: 21 May 2021
  - Annual Review
  - Updated copyright information
  - Changed TestAmerica to Eurofins TestAmerica throughout
  - Removed Table VIII and references to that table and instead referenced SOP DV-QA-024P QA/QC Requirements for Federal Programs for the information included in that table.
  - Removed QSM versions and instead referenced SOP DV-QA-024P QA/QC Requirements for Federal Programs for information about DoD QSMs.
  - Updated language and formatting throughout.
- Revision 15: 13 March 2020
  - Added reference to 8270E throughout.

Table I: Routine Instrument Operating Conditions

GC Conditions[1]	
Inlet	Split or Pulsed Split at 275 °C Split ratio - 3.1 : 1 Split Flow – 10.4 mL / min
Capillary Column	Varian Vf-5MS, 30 m length, 0.25 mm ID, 0.5 μm film
Column Mode	Constant flow, 3.4 mL/min
Temperature Program	Initial temp = 55 °C 30 °C/min ramp to 256 °C 4 °C/min ramp to 296 °C 30 °C/min ramp to 340 °C and hold for at least 1 minute past the elution time of the last compound.
Run Time	About 20 minutes with a new column.
Carrier Gas	Helium Purge flow = 25.0 mL/min, 3.00 min Total flow ≈ 31 mL/min
Injection Volume	Injection volume will be 1.0 µL or 5.0 µL depending on the logged method chain. 1. 8270C_SIM/3510C = 1.0 µL 2. 8270C_SIM/3510C_LVI = 5.0 µL 3. 8270D_SIM/3510C = 1.0 µL 4. 8270D_SIM/3510C_LVI = 5.0 µL 5. 8270D_SIM_DOD5/3510C = 1.0 µL 6. 8270D_SIM_DOD5/3510C_LVI = 5.0 µL 1.0 µL injection uses Standard Method Calibration standards (Section 7.2.2) 5.0 µL injections uses LVI Method Calibration standards (Section 7.2.2)
Transfer Line	290 °C
Mass Spectrometer Conditions[1,2]	
MS Source	230 °C
MS Quadrupole	200 °C
Dwell Time per Ion	Ranges from 30 to 100 milliseconds
Ions	See following tables

[1] The conditions listed above are subject to final fine adjustments to maximize instrument sensitivity. Changes to the above conditions are acceptable as long as method criteria are met.

[2] Details on the mass assignments in each window along with start and dwell times are given in Appendix III.

Table II: Surrogates for Standard List Analysis

PAH Surrogates	Mass Ion	Confirmation Ion
Nitrobenzene-d_5	82	128
2-Fluorobiphenyl	172	171
Terphenyl-d_{14}	244	122

Table III: Internal Standards for Standard List Analysis

Compound	Mass Ion	Confirmation Ion
Acenaphthene-d_{10}	164	162
Phenanthrene-d_{10}	188	94
Chrysene-d_{12}	240	120

Table IV: PAH Compounds and Ions Used for Analysis

Compound	Mass Ion	Confirmation Ions
Acenaphthene	153	152, 154
Acenaphthylene	152	151, 153
Anthracene	178	179,176
Benzo(a)anthracene	228	226, 229
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Chrysene	228	226, 229
Dibenzo(a,h)anthracene	278	139, 279
Dibenzofuran	168	139, 84
Fluoranthene	202	101, 203
Fluorene	166	165, 167
Indeno(1,2,3,cd)pyrene	276	138, 277
1-Methylnaphthalene	142	141, 115
2-Methylnaphthalene	142	141, 115
Naphthalene	128	129, 127
Phenanthrene	178	179, 176
Pyrene	202	101, 200
Morpholine	57	87

Table V: Example Retention Times, IS and Surrogate Associations

Compound	RT[1] (min.)	IS #	Surrogate #
Morpholine	4.001	1	1
Naphthalene	5.921	1	1
2-Methylnaphthalene	6.595	1	1
1-Methylnaphthalene	6.700	1	1
Acenaphthylene	7.512	1	2
Acenaphthene	7.686	1	2
Dibenzofuran	7.861	1	2
Fluorene	8.210	1	2
Phenanthrene	9.194	2	2
Anthracene	9.255	2	2
Fluoranthene	10.768	2	2
Pyrene	11.166	2	2
Benzo(a)anthracene	13.827	3	3
Chrysene	13.924	3	3
Benzo(b)fluoranthene	17.004	3	3
Benzo(k)fluoranthene	17.089	3	3
Benzo(a)pyrene	18.034	3	3
Indeno(1,2,3,cd)pyrene	21.509	3	3
Dibenz(a,h)anthracene	21.583	3	3
Benzo(g,h,i)perylene	22.306	3	3
Acenaphthene-d_{10} (IS)	7.657	1	-
Phenanthrene-d_{10} (IS)	9.177	2	-
Chrysene-d_{12} (IS)	13.856	3	-
Nitrobenzene-d_5 (Surr)	5.201	1	1
2-Fluorobiphenyl (Surr)	6.945	1	2
Terphenyl-d_{14} (Surr)	11.38	2	3

[1]Retention times may vary depending upon chromatographic conditions.

Table VI: DFTPP Key Ions and Ion Abundance Criteria

8270C

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	< 2% of mass 69
69	Mass 69 relative abundance
70	< 2% of mass 69
127	40 - 60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass ion 198
275	10 - 30% of mass 198
365	> 1% of mass 198
441	Present, but less than mass 443
442	40 - 100% of mass 198
443	17 - 23% of mass 442

With the exception of mass 442, the tune criteria for SW846 method 8270D are less stringent for the criteria required in SW846 method 8270C. For 8270D, the 442 mass must be greater than 50% of mass 198 to meet the tune criteria. By using the 8270C criteria, the rest of the data will be within the 8270D criteria.

Table VII: 8270D Relative Response Factor Criteria for Initial and Continuing Calibration

Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
Acenaphthene	0.900	20	20
Acenaphthylene	0.900	20	20
Anthracene	0.700	20	20
Benzo(a)anthracene	0.800	20	20
Benzo(a)pyrene	0.700	20	20
Benzo(b)fluoranthene	0.700	20	20
Benzo(g,h,i)perylene	0.500	20	20
Benzo(k)fluoranthene	0.700	20	20
Chrysene	0.700	20	20
Dibenzo(a,h)anthracene	0.400	20	20
Dibenzofuran	0.800	20	20
Fluoranthene	0.600	20	20
Fluorene	0.900	20	20
Indeno(1,2,3,cd)pyrene	0.500	20	20
1-Methylnaphthalene	0.400	20	20
2-Methylnaphthalene	0.400	20	20
Naphthalene	0.700	20	20
Phenanthrene	0.700	20	20
Pyrene	0.600	20	20

Appendix I: Extended List PAH Analysis by GC/MS

Summary of Method

This is the extended list for the SIM analysis that some clients require. All of the compounds listed in this appendix are analyzed for in addition to the standard compounds discussed throughout this SOP.

Modifications from the SIM analysis are as follows:

- The DFTPP tune has tailing factors that are calculated for Pentachlorophenol and Benzidine and a DDT breakdown check is performed.
- The instrument is calibrated at eight concentration levels. The calibration levels are made by diluting two stock standards with concentrations of 20 µg/mL [PAHXSIM stock (#1)] and 2 µg/mL [PAHXSIM 2º stock (#2)] down to the concentrations listed below, in methylene chloride. All phthalate compounds and 2-methylnaphthalene are at a ratio of 2:1 in the stock standards. Therefore, if the concentration is 0.02 µg/mL for the target analytes, the phthalates are at 0.04 µg/mL.

Level (µg/mL)	Stock ID	Stock Amt (µL)	Solvent amount (µL)	IS amount (µL)	Final Volume (µL)
0.02 µg/mL	#2	5	495	50	500
0.1 µg/mL	#2	25	475	50	500
0.3 µg/mL	#2	75	425	50	500
0.6 µg/mL	#1	15	485	50	500
1.2 µg/mL	#1	30	470	50	500
2.5 µg/mL	#1	62.5	437.5	50	500
5.0 µg/mL	#1	125	375	50	500
10.0 µg/mL	#1	250	250	50	500

Response factors for each compound must be ≤ 20% RSD. If any compound is > 20% RSD, must use the best curve fit.

Initial Calibration Verification

- The second source calibration stock is also at 20 µg/mL (PAHSIM SSV stock).
- The second source verification (SSV or ICV) is analyzed at 1.2 µg/mL.
- The acceptance criterion for the ICV is ± 25%D.

Continuing Calibration Verification

- The CCV is run at 0.6 µg/mL
- The criterion: The Average %D for all compounds must be < 20 %D, with no single compound exceeding 30 %D.

Sample extraction: See DV-OP-0006 Extraction of Aqueous Samples by Separatory Funnel (aqueous), DV-OP-0015 Ultrasonic Extraction of Solid Samples (soil), and DV-OP-0016 Microwave Extraction of Solid Samples (soil).

Sample concentration: See DV-OP-0007 Concentration and Clean-up of Organic Extracts.

Sample analysis:

- Internal Standard final concentration is 6 µg/mL in standards and extracts. The stock is at 400 µg/mL
- For the MS/MSD, the recovery for the spike pair must be within the control limits stored in TALS. The MS/MSD pair is generally aliquotted and run two times on the instrument, to confirm the results. If the results to be reported are from the first analysis, it is not required that the second analysis be within the 12 hour tune clock.

Instrument Configuration:

The GCMS instrumentation is configured the same as in the SIM analysis.

Extended List Compounds, Reporting Limits and Ions Used for Analysis:

Compound	Water Reporting Limit (ng/L)	Soil Reporting Limit (µg/kg)	Mass Ion	Confirmation Ion
1,4-Dioxane	NA	20	88	58
N-Nitrosodiphenylamine	1,000	20	169	168
N-Nitrosodimethylamine	400	18	74	42
N-Nitrosodiethylamine (LVI)	100	--	102	44
N-Nitrosodi-n-propylamine (LVI)	100	--	70	42
Butyl Benzyl Phthalate	1,000	20	149	91
Dimethyl Phthalate	1,000	20	163	164
Diethyl Phthalate	1,000	20	149	177
Bis(2-Ethylhexyl) Phthalate	1,000	20	149	167
Di-n-octyl Phthalate	1,000	20	149	150
Di-n-butyl Phthalate	1,000	20	149	150

Extended List Compounds Example Retention Times, IS and Surrogate Associations:

Compound	RT[1] (min.)	IS #	Surrogate #
1,4-Dioxane	1.60	1	2
N-Nitrosodiphenylamine	6.75	2	2
N-Nitrosodimethylamine	2.16	1	2
N-Nitrosodiethylamine (LVI)	2.72	1	1
N-Nitrosodi-n-propylamine (LVI)	3.69	1	1
Butyl Benzyl Phthalate	10.33	2	2
Dimethyl Phthalate	5.92	1	2
Diethyl Phthalate	6.51	1	2
Bis(2-Ethylhexyl) Phthalate	11.67	2	2
Di-n-octyl Phthalate	13.69	3	2
Di-n-butyl Phthalate	7.95	2	2
Acenaphthene-d_{10} (IS)	7.657	1	-
Phenanthrene-d_{10} (IS)	9.177	2	-
Chrysene-d_{12} (IS)	13.856	3	-
Nitrobenzene-d_5 (Surr)	5.201	1	1
2-Fluorobiphenyl (Surr)	6.945	1	2
Terphenyl-d_{14} (Surr)	11.38	2	3

[1]Retention times may vary depending upon chromatographic conditions.

APPENDIX II

Instrument Maintenance Schedules
Mass Spectrometer & Gas Chromatograph

MASS SPECTROMETER Instrument Maintenance Schedule				
Daily	**Weekly**	**As Needed**	**Quarterly**	**Annually**
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration (PFTBA or FC-43).	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between service contract maintenance.	Check vacuum, relays, gas pressures, and flows.	Replace the exhaust filters on the mechanical rough pump every 1 to 2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.		Change the oil in the mechanical rough pump.
Check inlets, septa.		Clean source, including all ceramics and lenses. Source cleaning is indicated by a variety of symptoms, including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.		Relubricate the turbomolecular pump-bearing wick.
Check baseline level.		Repair/replace jet separator.		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates the need for replacement.		

APPENDIX II (continued)

Instrument Maintenance Schedules
Mass Spectrometer & Gas Chromatograph

GAS CHROMATOGRAPH Instrument Maintenance Schedule (For GC/MS only.)	
Daily	***As Needed***
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or guard column or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance indicates it is required (e.g., peak tailing, poor resolution, high backgrounds, etc.).
Check temperatures of injectors and detectors. Verify temperature programs.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.
Check inlets, septa. Clean injector port.	Replace septa.
Check baseline level.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Repair or replace flow controller if constant gas flow cannot be maintained.
	Reactivate flow controller filter dryers when the presence of moisture is suspected.
	Autosampler: Replace syringe, fill wash bottle, dispose of waste bottle contents.

APPENDIX III
Mass Spectrometer Settings for Single Ion Monitoring

Group ID	Group Start Time[1] (min)	Analyte	Masses	Dwell Times
1	1.45	N-Nitrosodimethylamine	74, 42	50, 50
		1,4-Dioxane	88, 58	50, 50
		Morpholine[2]	57, 87	50, 50
		N-Nitrosodiethylamine (LVI)[3]	102, 44	50, 50
2	2.60	Nitrobenzene-d_5	82, 128	50, 50
		Naphthalene	128, 129, 127	50, 50
		N-Nitrosodiethylamine (LVI)[3]	102, 44	50, 50
		N-Nitrosodi-n-propylamine (LVI)	70, 42	50, 50
3	4.79	2-Fluorobiphenyl	172, 171	50, 50
		2-Methylnaphthalene	142, 141, 115	50, 50
		1-Methylnaphthalene	142, 141, 115	50, 50
4	5.46	Dimethyl Phthalate	163, 164	50, 50
		Acenaphthene- d_5	164, 162	50, 50
		Acenaphthene	153, 152, 154	50, 50
		Acenaphthylene	152, 151, 153	50, 50
		Dibenzofuran[4]	168, 139, 84	50, 50
5	6.06	Diethyl Phthalate	149, 177	50, 50
		N-Nitrosodiphenylamine	169, 168	50, 50
		Fluorene	166, 165, 167	50, 50
		Dibenzofuran[4]	168, 139, 84	50, 50
6	6.78	Phenanthrene-d_{10}	188, 94	50, 50
		Phenanthrene	178, 179, 176	50, 50
		Di-n-butyl Phthalate	149, 150	50, 50
		Anthracene	178, 179, 176	50, 50
7	8.05	Butyl Benzyl Phthalate	149, 91	50, 50
		Terphenyl-d_{14}	244, 122	50, 50
		Fluoranthene	202, 101, 203	50, 50
		Pyrene	202, 101, 200	50, 50
8	10.48	Chrysene- d_{12}	240, 120	50, 100
		Bis(2-Ethylhexyl) Phthalate	149, 167	100, 100
		Chrysene	228, 226, 229	50, 50
		Benzo(a)anthracene	228, 226, 229	50, 50
9	12.33	Di-n-octyl Phthalate	149, 150	50, 50
		Benzo(a)pyrene	252, 253, 125	50, 50
		Benzo(b)fluoranthene	252, 253, 125	50, 50
		Benzo(k)fluoranthene	252, 253, 125	50, 50
10	16.48	Dibenzo(a,h)anthracene	278, 139, 279	50, 50
		Indeno(1,2,3-cd)pyrene	276, 138, 277	50, 50
		Benzo(g,h,i)perylene	276, 138, 277	50, 50

[1]Group start times may vary due to chromatographic conditions.
[2]Morpholine method detection limit verifications not kept current. Laboratory does not stock standards.
[3]N-Nitrosodiethylamine (LVI) elutes between windows 1 and 2 and was therefore included in both.
[4]Dibenzofuran elutes between windows 4 and 5 and was therefore included in both.

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Title: Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020A/B

Approvals (Signature/Date):

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Doug Gomer Date
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Reed Pottruff Date
Health & Safety Manager / Coordinator

Maria Fayard 10/1/21
Maria Fayard Date
Quality Assurance Manager

Scott Hall 10/1/21
Scott Hall Date
Technical Director

Facility Distribution No. _____ **Distributed To:_____**

1.0 Scope and Application

1.1 This procedure describes multi-elemental analysis by inductively coupled plasma-mass spectrometry (ICP-MS) based on EPA Method 6020A and 6020B.

1.2 Method 6020A and 6020B lists twenty-three elements approved for analysis by ICP-MS. The laboratory has implemented analysis by these methods for : Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mg, Mn, Ni, Se, Ag, Sr, Tl, Th, V, and Zn. Additional elements may be included provided that the method performance criteria presented in Sections 9 and 12 are met. Project approval may be required from the controlling agencies for compliance testing beyond the elements included in the promulgated methods and for those elements which may require state-specific accreditation.

1.3 The procedure is applicable to the analysis of acid digested waters, sediments, sludges and soils. Standard reporting limits are listed in Attachment 1 for water and soil. The preliminary acid digestion for aqueous samples is described in SOP DV-IP-0014 Acid Digestion of Aqueous Samples for Analysis by ICP-MS for Methods 3005A and 3020A and the digestion procedure for solids is given in SOP DV-IP-0015 Acid Digestion of Solids for Method 3050B.

2.0 Summary of Method

2.1 Aqueous digestates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through the quartz torch and injects it into a radiofrequency (RF) plasma. There the sample is decomposed and desolvated.

2.2 The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrapole mass spectrometer capable of providing a mass resolution better than or equal to 0.9 amu (see Section 3) peak width at 10% of the peak height. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

2.3 A collision/reaction cell utilizing He and (optionally) H_2 gases is used to remove molecular interferences. As the ion beam passes through the cell chamber, a diffuse cloud of He or H_2 gas is injected into its path. Collisions between the ions and the atoms in the gas deflect and remove interferences. See Section 4.2.3 for more information.

2.4 Interferences must be assessed and valid corrections applied, or the data flagged to indicate problems. Interference corrections must include compensation for background ions contributed by the plasma gas, reagents, and the constituents of the sample matrix. Recommended elemental equations, which correct for many of these interferences, are listed in Attachment 2. Interference equations may vary or be unnecessary depending on the instrument setup and choice of collision/reaction gas.

2.5 Use of the internal standard technique is required to compensate for suppressions and enhancements caused by sample matrices. Internal standard assignments are listed in Attachment 4.

3.0 Definitions

3.1 **Atomic Mass Unit (amu) –** Obsolete term replaced by "unified atomic mass unit (u)" or "dalton (Da)", which denotes a small unit of mass that is used to express atomic and molecular masses. It is defined to be 1/12 of the mass of one atom of carbon-12.

3.2 **Batch –** The batch is a set of up to 20 samples of the same or similar matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches are defined at the sample preparation stage. See Policy DV-QA-003P Quality Control Program for further details.

3.3 **Dissolved Metals -** Those elements which pass through a 0.45-μm membrane filter (sample is acidified after filtration).

3.4 **Total Metals -** The concentration determined on an unfiltered sample following vigorous acid digestion.

3.5 **Total Recoverable Metals -** The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acids.

3.6 **Instrument Detection Limit (IDL) -** See Section 12.3.

3.7 **Sensitivity -** The slope of the analytical curve (i.e., the functional relationship between raw instrument signal and the concentration).

3.8 **Tuning Solution -** This is a multi-element solution containing analytes which are representative of the entire mass range capable of being scanned by the instrument. It is used to optimize the sensitivity of the instrument and to verify the mass resolution meets method criteria.

3.9 **Initial Calibration Verification / Quality Control Standard (ICV) -** A multi-element standard of known concentrations prepared to verify instrument calibration. This solution must be an independent standard prepared near the mid-point of the calibration curve, and at a concentration other than that used for instrument calibration.

3.10 **Continuing Calibration Verification (CCV) -** A multi-element standard of known concentrations prepared to monitor and verify the instrument daily continuing performance.

3.11 **Interference Check Standard (ICS) -** A solution containing both interfering and analyte elements of known concentration that is used to correction factors.

3.12 **Laboratory Control Sample / Laboratory Fortified Blank (LCS/LFB) -** A multi-element standard of known concentrations that is carried through the entire

sample preparation and analysis procedure. This solution is used to verify the accuracy of the sample preparation.

3.13 **Reagent Blank -** High purity (> 18 megohm-cm) water containing the same acid matrix as the calibration standards that is carried through the entire digestion process.

3.14 **Calibration Blank -** High purity (> 18 megohm- cm) water acidified with the same acid concentrations present in the standards and samples. Also referred to as the Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB).

3.15 **Method Detection Limit (MDL) -** The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

3.16 **Low Level ICV (LLICV) / Continuing Calibration Verification (LLCCV) -** A multi-element standard of known concentrations prepared to monitor and verify the instrument performance at the reporting limit (RL).

3.17 Refer to the Glossary of the Eurofins TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P Quality Control Program for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Elemental Isobaric Interferences

Elemental isobaric interferences in the ICPMS are caused by isotopes of different elements forming ions with the same nominal mass-to-charge ratio (m/z). Most interferences of this type are corrected for by the instrument software and by the careful selection of isotopes for analysis.

4.2 Isobaric Molecular Interferences

4.2.1 Polyatomic interferences are derived from the plasma gas, reagents or sample matrix. Isobaric molecular interferences are caused by ions consisting of more than one atom or charge. These molecular interferences are minimized by use of the collision cell utilizing He and/or H_2 gases. (See Attachment 2) When these interferences cannot be avoided by the use of another isotope with sufficient natural abundance, corrections must be applied and the data flagged to indicate the presence of interferences.

4.2.2 Chloride in samples can produce low recoveries for antimony and silver. If chloride interference is a concern, 1% HCl can be added during digestion, but calibration standards must be adjusted to include 1% HCl also. The use of hydrochloric and sulfuric acids should be minimized due to higher incidence of molecular-ion interferences with the presence of these acids. Excessive amounts of nitric acid can also lead to molecular interferences.

4.2.3 Collision cell interference removal works both by causing the interfering molecular ion to dissociate and by reducing the kinetic energy of the ion. The latter is termed Kinetic Energy Discrimination (KED), and is the primary mechanism for interference removal. Polyatomic ions are larger than elemental ions and so collide with the helium atoms in the collision cell more frequently than the smaller elemental ions. Each collision reduces the energy of the ion, so the molecular ions lose energy more quickly. At the end of the collision cell a positive voltage plate prevents passage of the now low energy molecular ions. Thus, the interference is eliminated because the molecular ions do not reach the detector.

4.3 Doubly Charged Ion Interferences

Doubly charged elemental ion interferences are possible in cases where the second ionization potential of the element is significantly below the first ionization potential for argon (15.7 eV). If a doubly charged ion is formed, it will cause a response at half of its elemental mass, potentially causing interference. Most elements have high enough second ionization potentials that formation of doubly charged ions is not an issue. The percentage of doubly charged ions being formed in the plasma is monitored on a daily basis during the instrument tuning process.

4.4 Physical Interferences

4.4.1 Physical interferences are associated with the transport and nebulization process. Internal standards are used to compensate for these types of interferences.

4.4.2 Internal standards should be added at a level to give approximately 100,000 – 20,000,000 counts of raw signal intensity. The mass of the internal standard should ideally be within 50 amu of the mass of the measured analyte.

4.4.3 Matrix effects are monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the calibration blank. When performing method 6020A, the internal standard recoveries in samples can not fall below 70% of the intensity of the calibration standard. For method 6020B, the internal standard recoveries in samples cannot fall below 30% while the requirement for DoD is 30-120%. If they fall outside the applicable window, a five-fold dilution (1:4) is performed on the sample to correct for matrix effects and the sample is reanalyzed.

4.4.4 Memory effects or carry-over can occur when there are large relative concentration differences between samples and/or standards which are analyzed sequentially. The rinse period between samples must be long enough to eliminate significant memory interference.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.2 The ICP-MS plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma. The RF Generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) capable of providing resolution less than or equal to 0.9 amu at 10% peak height and 1.0 amu at 5% peak height in the mass range from 6-253 with a data system that allows corrections for isobaric interferences and the application of the internal standard technique. The ICP-MS must be equipped with a collision cell for the removal of molecular interferences.

6.1.2 A four-channel peristaltic pump.

6.1.3 Autosampler with autosampler tubes.

6.1.4 Appropriate water cooling device.

6.2 Supplies

6.2.1 Argon gas: High purity grade (99.99%).

6.2.2 Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.2.3 Class A volumetric flasks and 100ml Graduated Cylinder

6.3 Computer Software and Hardware

Please refer to the master list of documents and software located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. All standards must be entered into the Eurofins TestAmerica LIMS (TALS) Reagent Module. Reagents that are not used for calculating results may either be recorded in the Reagent Module or may be entered into batch worksheets.

7.1 Storage and Shelf-Life

7.1.1 All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Standards stored at concentrations as received from the vendor and mid-level dilutions must be replaced prior to the expiration date assigned by the vendor. If no expiration date is provided, the stocks and mid-level standards may be stored for up to one year. They must be replaced sooner if verification from an independent source indicates a problem.

7.1.2 Working standards, i.e., all standards at concentrations ready to analyze on the ICP-MS are prepared fresh daily.

7.1.3 For more information on standard storage and shelf-life, see SOP DV-QA-0015 Verification and Storage of Chemical Standards and Reagents.

7.2 Standards

Detailed instructions regarding the preparation of standards and reagents are given in this section. Alternate procedures are allowed as necessary to accommodate volume requirements as long as final concentrations are maintained and an accurate description of the standard or reagent used is entered into the Reagent Module in TALS.

7.2.1 Tuning Solution

The parent tuning solution is purchased as a custom multi-element mix. The elements and concentrations of the constituents are shown in Attachment 7. Prepare the working tuning Solution as detailed below.

7.2.1.1 Obtain a clean 1 L volumetric flask

7.2.1.2 Place 500 mL of reagent water and 10 mL of conc. HNO_3 in the flask

7.2.1.3 Pipette 1mL of the Tuning Solution Stock into the flask

7.2.1.4 Add 50 µL of a 500 mg/L Mg solution and 30 µL of a 1000 mg/L Be solution.

7.2.1.5 Dilute to volume with reagent blank (See Section 7.3). Stopper and mix.

7.2.2 P/A factor solution

7.2.2.1 The Pulse/Analog (P/A) solution is used to monitor the correlation between the Pulse counting and Analog modes of the electron multiplier. The diluted solution must be prepared at different concentrations depending on the current instrument conditions. Multiple dilutions may be required to cover the required intensity range for all elements.

7.2.2.2 The P/A solution may be commercially purchased as a custom multi-element mix. See Attachment 7 for a list of the constituents and concentrations.

7.2.2.3 Prepare and use the P/A solution as recommended by the instrument manufacturer. The P/A solution should be analyzed daily.

7.2.3 Calibration Standard

Stock calibration standards are purchased as custom multi-element mixes or as single element solutions. Each day of analysis, the standards are diluted to working levels using reagent blank (see Section 7.3). The concentrations are given in Attachment 10. Prepare the Daily Working Calibration Standard as shown.

7.2.3.1 Daily Working Calibration Standard for Instruments 077 and 078 (ms 77 cal std)

7.2.3.1.1 Obtain a clean 200 mL volumetric flask.

7.2.3.1.2 Place 100 mL of reagent blank in the flask.

7.2.3.1.3 Pipette 0.25 ml of each MS CALSTD 1 thru 6 into the flask.

7.2.3.1.4 Dilute to volume with reagent blank. Stopper and mix.

7.2.4 Initial Calibration Verification (ICV) Standard

The ICV stock is from a source different than the source for the calibration standards. Each day of analysis, the ICV standards are prepared new in reagent blank to the concentrations shown in Attachment 10. Prepare the ICV as shown below:

7.2.4.1 Initial Calibration Verification Standard for Instruments 077 and 078 (MS 77 ICV)

7.2.4.1.1 Obtain a clean 250 mL volumetric flask.

7.2.4.1.2 Place 100 mL of reagent blank in the flask.

7.2.4.1.3 Pipette 0.1 mL of each MS ICVMIX 1 thru 6 into the flask.

7.2.4.1.4 Dilute to volume with reagent blank. Stopper and mix.

7.2.5 Continuing Calibration Verification (CCV) Standard

The CCV is prepared from the same source as the calibration standards. The CCV standards are prepared fresh each day of analysis in reagent blank. The concentration is shown in Attachment 10.

7.2.5.1 Continuing Calibration Verification for Instruments 077 and 078 (ms 77 ccv)

7.2.5.1.1 Using a graduated cylinder pour 100 ml each of the MS CAL Daily and reagent blank for a final volume of 200 ml.

7.2.6 Reporting Limit (RL) Standards

The reporting limit standards are prepared fresh daily from the same stock as the calibration standards using the reagent blank. The analyte concentrations must be less than or equal to the respective reporting limits. Multiple solutions may be required in order to satisfy all of the project and client specific reporting limits. Alternate reporting limit concentrations may be used as necessary to meet client requirements as long as an accurate description of the standard used is entered into the Reagents module in TALS. Prepare the Reporting Limit Standard for the Agilent 7700 as detailed below.

7.2.6.1 RL Standard for Instruments 077 and 078 (ms 77 RL)

7.2.6.1.1 Obtain a clean 50 mL volumetric flask.

7.2.6.1.2 Place 30 mL of reagent blank in the flask.

7.2.6.1.3 Pipette 0.5 mL of the ms 77 cal std solution into the flask.

7.2.6.1.4 Dilute to volume with reagent blank. Stopper and mix.

7.2.7 Daily Linear Range Standard

The Linear Range standard is prepared from the same stock as the calibration standards using reagent blank.

7.2.7.1 Daily Linear Range Standard for Instruments 077 and 078 (MS 77 LR STD)

7.2.7.1.1 Obtain a clean 500 mL volumetric flask.

7.2.7.1.2 Place 50 mL of reagent blank in the flask.

7.2.7.1.3 Pipette 1.0 mL of a 1,000 mg/L As standard into the flask.

7.2.7.1.4 Pipette 2.5 mL of a 1,000 mg/L Ba standard into the flask.

7.2.7.1.5 Pipette 1.0 mL of a 1,000 mg/L Be standard into the flask.

7.2.7.1.6 Pipette 1.0 mL of a 1,000 mg/L Cd standard into the flask.

7.2.7.1.7 Pipette 1.0 mL of a 1,000 mg/L Co standard into the flask.

7.2.7.1.8 Pipette 2.5 mL of a 1,000 mg/L Cr standard into the flask.

7.2.7.1.9 Pipette 2.5 mL of a 1,000 mg/L Cu standard into the flask.

7.2.7.1.10 Pipette 1.0 mL of a 1,000 mg/l Li standard into the flask.

7.2.7.1.11 Pipette 5.0 mL of a 1,000 mg/L Mn standard into the flask.

7.2.7.1.12 Pipette 1.0 mL of a 1,000 mg/L Mo standard into the flask.

7.2.7.1.13 Pipette 2.5 mL of a 1,000 mg/L Ni standard into the flask.

7.2.7.1.14 Pipette 2.5 mL of a 1,000 mg/L Pb standard into the flask.

7.2.7.1.15 Pipette 0.5 mL of a 1,000 mg/L Sb standard into the flask.

7.2.7.1.16 Pipette 1.0 mL of a 1,000 mg/L Se standard into the flask.

7.2.7.1.17 Pipette 1.0 mL of a 1,000 mg/L Sn standard into the flask.

7.2.7.1.18 Pipette 1.0 mL of a 1,000mg/l Sr standard into the flask.

7.2.7.1.19 Pipette 0.5 mL of a 1,000 mg/L Tl standard into the flask.

7.2.7.1.20 Pipette 1.0 mL of a 1,000 mg/L U standard into the flask.

7.2.7.1.21 Pipette 1.0 mL of a 1,000 mg/L V standard into the flask.

7.2.7.1.22 Pipette 2.5 mL of a 1,000 mg/L Zn standard into the flask.

7.2.7.1.23 Pipette 0.05 mL of a 10,000 mg/L Th standard into the flask.

7.2.7.1.24 Dilute to volume with reagent blank. Stopper and mix.

7.2.8 Internal Standard (IS) Solution (77 I.S. / MS I.S. INT)

The internal standard solution is added continuously by peristaltic pump through a mixing tee. The concentrations and components are specified in Attachment 4. Prepare the IS solution as follows:

7.2.8.1 Obtain a clean 250 mL volumetric flask.

7.2.8.2 Place 100 mL of reagent blank in the flask.

7.2.8.3 Pipette 1.2 mL of the 1,000 mg/L Ge Standard into the flask.

7.2.8.4 Pipette 0.4 mL of the 1,000 mg/L Ho Standard into the flask.

7.2.8.5 Pipette 0.4 mL of the 1,000 mg/L In Standard into the flask.

7.2.8.6 Pipette 0.75 mL of the 1,000 mg/L Sc Standard into the flask.

7.2.8.7 Pipette 1.5 mL of the 1,000 mg/L ^{6}Li Standard into the flask.

7.2.8.8 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.9 Interference Check Standard Solutions (ICSA / ICSAB)

The interference check standard solution (ICSA) and the spiked interference check standard solution (ICSAB) are prepared as follows:

7.2.9.1 ICSA Standard (ms 77 icsa / MS ICSA)

7.2.9.1.1 Obtain a clean 100 mL volumetric flask.

7.2.9.1.2 Place 50 mL of reagent blank in the flask.

7.2.9.1.3 Pipette 10.0 mL of the MS ICSA STOCK standard into the flask.

7.2.9.1.4 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.9.2 ICSAB Standard (MS 77 ICSAB / MS ICSAB)

7.2.9.2.1 Obtain a clean 100 mL volumetric flask.

7.2.9.2.2 Place 50 mL of reagent blank in the flask.

7.2.9.2.3 Pipette 0.1 mL of each MS ICVMIX 1 thru 6 stock standards into the flask.

7.2.9.2.4 Pipette 10.0 mL of the MS ICSA STOCK standard into the flask.

7.2.9.2.5 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.10 6020A only -Low Level Initial Calibration Verification and Low-Level Continuing Verifications (ms 77 LLCCV / MS LCCV)

The low level ICV / low level CCV solution is prepared from the same source as the calibration standards. The low level standard is prepared fresh each day of analysis in reagent blank. The concentration is shown in Attachment 10. Prepare the low level standard solution as follows:

7.2.10.1 Obtain a 100 mL volumetric flask.

7.2.10.2 Place 50 mL of reagent blank in the flask.

7.2.10.3 Pipette 1 ml of MS LLCCV

7.2.10.4 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.3 Reagents

7.3.1 Reagent Water – Water free of the elements of interest, generated using an ion-exchange water polishing system capable of achieving 18.0 megohm-cm.

7.3.2 **Reagent Blank - Agilent 7700, 2% HNO3/0.5% HCl –** Carefully dilute 40 mL of concentrated HNO3 and 10 mL of HCl in 2.0 L of reagent water. This solution is used to dilute samples and it is used for the initial and continuing calibration blanks.

7.3.3 **Reagent Blank - Agilent 7700, 5% HNO3/5% HCL (Zr only) –** Carefully dilute 100 ml of concentrated HNO_3 and 100 ml of HCL in 2.0 L of reagent water. This solution is used to dilute samples and it is used for calibration blanks.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[1]	Reference
Water[2]	HDPE	50 mLs	HNO_3, pH < 2	180 Days	SW-846
Soil	Glass	4 oz	Cool ≤ 6°C[3]	180 Days	SW-846

[1]Samples must be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Holding Times are calculated from the date the sample was collected.

[2]Water samples collected for dissolved elements are filtered immediately on-site by the sampler before adding preservative.

[3]Although ICP analysis of soil does not require refrigeration of the samples, mercury analysis does require refrigeration per SW-846. Samples which will be used to aliquot for both analyses must be refrigerated. Therefore the laboratory routinely refrigerates samples to be analyzed by Methods 6020A or 6020B.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS Method Comments to determine specific QC requirements that apply. Quality control requirements are summarized in Attachment 9.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in Eurofins TestAmerica Denver Policy DV-QA-003P Quality Control Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in Eurofins TestAmerica Denver Policy DV-QA-024P QA/QC Requirements

for Federal Programs. This procedure meets all criteria for DoD QSM unless otherwise stated.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 Non-Conformance and Corrective Action System. This is in addition to the corrective actions described in the following sections.

9.2 Method Blank (MB)

For aqueous and soil samples, the method blank consists of reagent water that has been processed in the same manner as the samples. For soil samples analyzed under DoD QAPPs, the method blank consists of <1 mm glass beads that have been processed in the same manner as the samples. One method blank must be processed with each preparation batch.

Acceptance Criteria: Method blank results are acceptable if the concentration for each analyte of interest is less than ½ the reporting limit (RL). For DoD QSM the control limit is less than ½ LOQ. In the absence of project specific reporting limits, if the blank is less than 10% of the lower limit of quantitation check sample concentration, less than 10% of the regulatory limit, or less than 10% of the lowest sample concentration for each analyte in a given preparation batch, whichever is greater, then the method blank is considered acceptable.

Corrective Action: If the method blank does not meet the acceptance criteria, the source of contamination should be investigated to determine if the problem can be minimized or eliminated. Samples associated with the contaminated blank shall be reprocessed for analysis or, under the following circumstances, may be reported as qualified (qualifier flags or narrative comments):

- The same analyte was not detected in the associated samples;
- The method blank concentration is less than 1/10 of the measured concentration of any sample in the batch;
- The method blank concentration is less than 1/10 the

specified regulatory limit; or

- The analyte is a common laboratory contaminant (e.g., copper, zinc, iron, or lead) less than 2 times the RL. Note that some programs do not recognize common lab contaminants or have a more stringent criterion (e.g., DoD QSM allows common laboratory contaminants up to the RL).

If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. This anomaly must be addressed in the project narrative and the client must be notified.

9.3 Laboratory Control Sample (LCS)

The LCS consists of reagent water that is spiked with the analytes of interest at the project specific action level or, when lacking specific action levels, at approximately the mid-point of the calibration range (summarized in Attachment 10). For soil samples analyzed under DoD QAPPs, the LCS consists of <1 mm glass beads that have been spiked with the analytes of interest and processed in the same manner as the samples. One LCS must be processed for each preparation batch.

Acceptance Criteria: LCS control limits are based on three standard deviations of past laboratory results or program specific requirements. These limits must not exceed 80-120%. The control limits are maintained in TALS. For DoD QSM the laboratory must use QSM Limits for batch control if project limits are not specified.

Corrective Action: If the LCS recovery falls outside of the control limits for any analyte, that analyte is judged to be out of control. All associated samples must be reanalyzed One possible exception is a recovery for a given element above the upper control limit with no detection for the same element in the samples. If project requirements allow this exception, the data may be accepted with qualifiers, an NCM must be generated, and the failure narrated in the final report.

9.4 Matrix Spike / Matrix Spike Duplicate (MS / MSD)

The MS is prepared by taking a second aliquot of a selected sample and spiking it with the analytes of interest at the same level as the LCS (summarized in Attachment 10). An MSD is prepared by taking a third aliquot of the selected sample and spiking it with the analytes of interest at the same level as the LCS (summarized in Attachment 10). The MS and MSD are processed in the same manner as the samples. One MS/MSD pair must be processed for each preparation batch. Some programs (e.g., DoD) require that matrix spikes can be performed only on project samples, and that the samples to be used are identified on the chain of custody form. The spike concentration should be the same level as the LCS.

Acceptance Criteria: Control limits are based on historical data or project specific requirements. Historical control limits are based on three standard deviations of past laboratory results. These limits are not to exceed 75-125% recovery, and 20% relative percent difference (RPD). The control limits are maintained in TALS. For DoD QSM the laboratory must use QSM limits for batch control if project limits are not specified.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).

- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.
- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

NOTE: This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD is preferred as not all samples will contain measurable concentrations of the target analytes. Samples that have target analytes at low concentrations or non-detectable levels do not provide useful precision data. When an MS/MSD is not available, an LCS and LCSD will be used to measure precision.

9.5 Interference Check Solutions (ICSA/ICSAB)

The interference check solution is prepared with known concentrations of interfering elements so a determination may be made as to the magnitude of the interference on analytes of interest as well as a test of any software corrections. The required elements and their concentrations are listed in Attachment 5. The interference check solutions must be analyzed at the beginning of every analytical run and once every 12 hours thereafter. The results of solution "A" and solution "AB" should be monitored for possible interferences.

Acceptance Criteria: The non-spiked analytes in the A solution must be less than 2x the RL. The results for the trace elements (B portion) must be ± 20% of the expected value. In addition,

the internal standard recoveries for both the ICSA and AB must be within 70-150% for Method 6020A, 30-150% for Method 6020B and 30-120% for DoD. Some programs have control limits for the non-spiked elements in the ICSA. Please check the client specific requirements. For DoD QSM the ICSA for non-spiked elements is controlled to less than the absolute value of the LOD unless they are a verified impurity.

Corrective Action: If the ICSAB results exceed the 20% limit or the ICSA is out for DoD QSM, then the analysis sequence must be terminated. For DoD QSM if the ICSA is outside of the control limits for the non-spiked elements the sequence must also be terminated. The problem must be investigated and fixed. The ICSA and all affected samples must be re-analyzed.

NOTE: It may not be possible to obtain absolutely clean ICSA/ICSAB standards. If contamination can be confirmed by another method (e.g., ICPAES), acceptance criteria will be applied at that level and the data accepted.

9.6 Internal Standards Evaluation for Samples

The IS recovery in samples cannot fall below 70% or be above 150% of the intensity of the calibration blank for 6020A and 30-150% for 6020B. If sample IS recoveries fall outside of these criteria, a five-fold (1:4) dilution must be performed, the dilution analyzed, and the same acceptance criteria applied. For DoD QSM the internal standard for samples is controlled to 30-120%.

9.7 Serial Dilution

One serial five-fold dilution should be analyzed per preparation batch. If the analyte concentration is within the linear range of the instrument and sufficiently high (minimally, a factor of 50 times above the MDL), the serial dilution must agree to within 10% of the original analysis. If not, an interference effect is suspected, which must be described in an NCM and included in the final report narrative. Samples identified as blanks should not be used for serial dilution. For DoD QSM the serial dilution is evaluated if the parent sample concentration is greater than 50x the LOQ prior to dilution. If the acceptance criteria are not met then the parent sample is flagged “J”. Method 6020B sets the calculation level at 25x RL and the required limit at 20%.

9.8 Post-Digestion Spike Addition (PDS)

A PDS is performed for each batch. An analytical spike added to a portion of a prepared sample, or its dilution, should be recovered to within 80 - 120% of the known value. If the PDS fails to meet this criterion, matrix interference should be suspected. Typically the concentration of the PDS is 200 µg/L for each element except silver which is spiked at 50 µg/L. For DoD QSM if the parent sample concentration is less than 50x the LOQ prior to dilution then the PDS must recover

within 80-120%. If the recovery is outside of the control limits for a given element then the parent sample is flagged "J". Method 6020B allows limits of ± 25%.

9.9 Linear Range Verification (LRA/LRC)

The LDRs should be verified whenever, in the judgment of the analyst, a change in the analytical performance caused by either a change in instrument hardware or operating conditions would dictate the necessity to re-establish them. 6020B and DoD QSM require verification of linear ranges in each analytical run. As described in Section 7.2.7, a lower concentration is used for the daily check than is used for the quarterly determination.

Acceptance Criteria: The result for this standard must be within 10% of the expected value.

Corrective Action: If the Linear Range Verification standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analyst must run a standard at a lower concentration until the criteria is met or the samples cannot exceed the level of the highest calibration standard.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 Non-Conformance and Corrective Action System. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any unauthorized deviations from this procedure identified after the work has been completed must also be documented in an NCM, with a cause and corrective action described.

10.3 Instrument Maintenance

See Section 20 in the QAM

10.4 Instrument Troubleshooting

See Attachment 11

10.5 Sample Preparation

Solid and aqueous samples must be digested prior to analysis by the appropriate method (see SOPs DV-IP-0014 Acid Digestion of Aqueous Samples for Analysis by ICP-MS and DV-IP-0015 Acid Digestion of Solids).

10.6 Calibration

10.6.1 Instrument Start Up

Set up the instrument according to manufacturer's operating instructions. Allow the instrument to become thermally stable for at least 30 minutes before tuning. It is recommended that the instrument be flushed with the ICSA solution to help condition the cones and improve stability. Allow the instrument time to rinse completely before tuning the instrument.

10.6.2 Oxide\Doubly Charge Performance Check

With the sample probe in the Tune solution verify that the oxides and doubly charged ions are less the 3% by running the Tune report.

10.6.3 Instrument Tuning / Mass Calibration

Tune the instrument with a solution containing elements representing all of the mass regions of interest. The relative standard deviations must be less than 5% after running the tuning solution a minimum of 5 times. Mass calibration and resolution checks using the tuning solution must be completed at the beginning of every day. If either of the following conditions fails the instrument setup must be re-evaluated and the solution rerun.

Mass Calibration Check – The mass calibration results must be within 0.1 amu from the true value. If this criterion is not met, the mass calibration must be adjusted before running samples.

Mass Resolution Check - The resolution at 5% peak height should be approximately 0.75amu.

NOTE: Method 6020B states to use the manufacturer's instructions for the tune. Since the laboratory may perform analysis for Method 6020A on the same instrument, the same requirements are applied to both.

10.6.4 Initial Calibration

The ICP-MS is calibrated each day of operation using a blank and a single standard (see Section 7.2.3). At least three integrations are employed. The validity of the calibration is determined by the subsequent calibration verifications, which are performed at concentrations as described in the next sections.

10.6.5 Low-Level Initial Calibration Verification (LLICV/ICVL)

A low-level ICV standard at or below the reporting limit (see Section 7.2.10) is analyzed after the initial calibration for Method 6020A. This is a standard obtained from the same vendor used for calibration.

Acceptance Criteria: The ICVL recovery must be within 70-130%.

The ICVL can be reanalyzed, but two consecutive successful results must be obtained or corrective action is taken.

Corrective Action: If the ICVL results are outside of the acceptance limits, investigate the accuracy of the standards, correct as necessary, and recalibrate.

10.6.6 Mid-Level Second-Source Initial Calibration Verification (ICV)

A 40 μg/L ICV standard (see Section 7.2.4) is analyzed immediately after the initial calibration. This is a standard obtained from a different vendor than the standard used for calibration.

Acceptance Criteria: The ICV recovery must be within 90-110%. The ICV can be reanalyzed, but two consecutive successful results must be obtained or corrective action is taken.

Corrective Action: If the ICV results are outside of the acceptance limits, investigate the accuracy of the standards, correct as necessary, and recalibrate.

10.6.7 Calibration Blank

An initial calibration blank (ICB) is analyzed after the ICV. Continuing calibration blanks (CCBs) are analyzed after each continuing calibration verification. The appropriate reagent blank is used for the blanks.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Common lab contaminants such as zinc and iron must be less than the RL. In addition, the internal standard recoveries must be within 70-150% of the associated calibration blank for Method 6020A or 30-150% for Method 6020B. Client specific requirements take precedence. DoD QSM requires control of blanks to a concentration less than or equal to the LOD with the internal standard recoveries of 30-120%.

Corrective Action: If the calibration blank exceeds acceptance limits, then the possibility of instrument

contamination should be examined, particularly the possibility of carry-over from high level samples. The blank can be reanalyzed, and if successful, analysis can continue. However, samples tested after high-level samples should be retested. If the reanalysis is not successful, then the analysis should be terminated. After the problem is corrected, recalibrate and reanalyze all samples tested since the last acceptable CCB.

10.6.8 Reporting Limit (RL/CRI) Verification Standard

Because the ICP-MS calibration does not include multiple calibration levels, an independent standard is analyzed after the ICV to monitor the lab's ability to produce reliable results at RL-level concentrations. The RL verification standard (see Section 7.2.6) is analyzed after the daily ICB.

Acceptance Criteria: For Method 6020A, the results should be within 50% of the expected value. Some programs may require tighter controls. For Method 6020B and DoD QSM the control limits are 80-120%.

Corrective Action: If the RL verification fails to meet acceptance limits, data for the associated samples must be assessed. For example, if the results are high, consider blank contamination, and if the results are low, consider MDL verifications. At a minimum, sample results must be qualified in the final report. For DoD QSM, if the low-level standard does not meet the limits when spiked at the required project RL, the run sequence must be terminated.

10.6.9 Lower Limit of Quantitation Check (LLQC, LLOQ)

The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits, quarterly and on an as needed basis to demonstrate the desired detection capability. The difference between the LLQC and the RL is that this standard is carried through the entire preparation and analytical procedure. Prepare 7 aliqouts.

Acceptance Criteria: LLQC is verified when all analytes are detected within $\pm$ 35% of their true value. The RSD should be $\leq$20%

Corrective Action: If the LLQC fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be

terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.6.10 6020A Only - Low-Continuing Calibration Verification (LLCCV/CCVL) Standard

A low-level CCV standard is analyzed after every set of ten samples and at the end of the analytical sequence.

Acceptance Criteria: The CCVL recovery must be within 70-130%. In addition, the IS recovery must be within method limits. If CCVL results are not within these limits, the CCVL can be reanalyzed, but it must be successful twice in succession. If the calibration cannot be verified within these specified limits, the analysis of samples containing the affected analytes at similar concentrations cannot continue until the cause is determined and the CCVL standard successfully analyzed.

For the state of Washington the CCVL must work on the first attempt.

Corrective Action: If the CCVL fails acceptance criteria, then the analysis should be terminated. Recalibrate and reanalyze all samples tested since the last acceptable CCVL. If the associated samples are at levels greater than 10X the level of the CCVL the data may be considered acceptable but the failure must be documented with an NCM and addressed in the case narrative.

For the state of Washington the previous 10 samples must be reanalyzed. A recalibration is not necessary if the two CCVL's following the failure are successful but the samples must still be rerun.

10.6.11 Continuing Calibration Verification (CCV) Standard

A 50 μg/L CCV standard (see Section 7.2.5) is analyzed after every set of ten samples or every 2 hours, whichever is most frequent, and at the end of the analytical sequence.

Acceptance Criteria: The CCV recovery must be within 90-110%. In addition, the IS recovery must be within 70-150% for Method 6020A or 30-150% for Method 6020B. If the CCV results are not within these limits, the CCV can be reanalyzed, but it must be successful twice in succession or further corrective action must be taken.

For the state of Washington the CCV must work on the first attempt

Corrective Action: If the CCV fails acceptance criteria, then the analysis should be terminated. Recalibrate and reanalyze all samples tested since the last acceptable CCV.

For the state of Washington the previous 10 samples must be reanalyzed. A recalibration is not necessary if the two CCV's following the failure are successful but the samples must still be rerun.

10.7 Sample Analysis

10.7.1 Report the average of at least three integrations for all field and QC samples analyzed.

10.7.2 Flush the system with the rinse blank for at least 30 seconds between samples and standards during the analytical run.

10.7.3 Masses which would affect the data quality must be monitored during the analytical run to determine the potential effects of matrix on a given element. See Attachment 3 for examples.

10.7.4 Dilute and reanalyze samples that are more concentrated than the linear range for an analyte. DoD QSM requires that samples be diluted and reanalyzed if they are above the daily linear range check standard. No analyte may be reported from an analysis of a diluted sample in which the analyte concentration is less than 5 times the RL. (The sample should be diluted to the approximate midrange of the analytical curve.) See Section 9.9 for the linear range verification requirements.

10.7.5 During the course of an analytical run, the instrument may be resloped or recalibrated to correct for instrument drift. A recalibration must then be followed immediately by a new analysis of an ICV, CCV and CCB before any further samples may be analyzed.

10.7.6 The analytical run sequence should be performed as follows to meet all quality control criteria:

Instrument initialization / Warm-Up
Tune instrument
Perform mass calibration
Perform resolution check
Validate tuning criteria
Calibration blank
Calibration standard
ICV
ICB

LLICV
RL verification standard
LLQC(as needed)
ICSA
ICSAB
LRA
CCV
CCB
LLCCV (6020B)
10 Samples (which can include all sample types)
CCV
CCB
LLCCV (6020B)
Reslope
CCV
CCB
LLCCV

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate Policy CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

11.2 ICV percent recoveries are calculated according to the equation:

$$\%R = \left(\frac{\text{ICV Found Value}}{\text{ICV True Value}} \right) \times 100\%$$

11.3 CCV percent recoveries are calculated according to the equation:

$$\%R = \left(\frac{\text{CCV Found Value}}{\text{CCV True Value}} \right) \times 100\%$$

11.4 Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = \left(\frac{\text{SSR - SR}}{\text{SA}} \right) \times 100\%$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

NOTE: When the sample concentration is less than the detection limit, use SR = 0 for the purpose of calculating %R.

11.5 The relative percent difference (RPD) between sample duplicates is calculated according to the following equation:

$$RPD = \left[\frac{DU1 - DU2}{\frac{1}{2}(DU1 + DU2)} \right] \times 100$$

Where:

DU1 = Sample result

DU2 = Sample duplicate result

11.6 The final concentration for an aqueous sample is calculated as follows:

$$\text{Result } (\mu\text{g/L}) = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration from instrument readout, ppb

D = Instrument dilution factor

V1 = Final volume in liters after sample preparation

V2 = Initial volume of sample digested in liters

11.7 The concentration determined in digested solid samples when reported on a dry weight basis is as follows:

$$\text{Result } (\mu\text{g/kg}) = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration from instrument readout, ppb

D = Instrument dilution factor

V = Final volume in liters after sample preparation

W = Weight, in g, of wet sample digested

S = Percent solids/100

11.8 Sample data are reviewed by the analyst (Level 1 data review) and documented on the data review checklist (See SOP DV-QA-0020 Data Review). The data package is then submitted for level 2 review by another analyst or data reviewer. Second level review is documented on the same checklist initiated by the analyst. The data review process is explained in SOP DV-QA-0020 Data Review.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is

present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with Policy CA-Q-S-006 Detection and Quantitation Limits. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. For DoD, DOE and TX TRPP projects, an MDL verification is performed quarterly.

12.2 MDL Verification (MDLV)

Calculated MDLs from the annual studies are subject to quarterly verification by analyzing an MDLV standard.

12.2.1 Prepare an MDLV standard at 2-4 times the calculated MDL concentration.

12.2.2 Analyze the MDLV standard immediately after each MDL study and quarterly thereafter. This standard is subject to the entire preparation and analysis process.

12.2.3 The calculated MDL is verified if the MDLV standard is detected, nominally signal to noise ratio > 3, under routine instrument conditions.

12.2.4 If the first MDLV is not detected, re-prepare the MDLV standard at twice the original concentration and analyze. The lowest concentration that produces a detectable signal will then be reported as the MDL.

12.3 Instrument Detection Limit Study

Instrument detection limit (IDL) studies are conducted quarterly for each instrument and each analyte used for analysis in accordance with Policy DV-QA-014P Determination of Instrument Detection Limits.

12.3.1 Pour out seven undigested calibration blanks and run them on three non-consecutive days.

12.3.2 Calculate the standard deviation for each day. The final IDL concentration is the average of the three daily standard deviation values.

12.3.3 Method 6020B requires an initial verification of the IDL using 10 replicates in a single analytical sequence but no longer requires quarterly verification. Reverification is required after major maintenance, such as changing the detector.

12.3.4 See Policy DV-QA-014P Determination of Instrument Detection Limits for a discussion of IDL studies and evaluation of IDL results.

12.4 Linear Dynamic Range (LDR)

12.4.1 The LDR must be determined initially (i.e., at initial setup) and then every three months for each analyte used on each instrument. The linear

range is the concentration above which results cannot be reported without dilution of the sample.

12.4.2 The LDR must be determined from a linear calibration prepared in the normal manner using the normal operating procedures described in Sections 10 and 11.

12.4.3 The LDR is determined by analyzing successively higher standard concentrations of the analytes of interest. A minimum of three standards are required for the initial and on-going studies, and one of the levels must be at the upper end of the range. The calculated concentrations must be within 10% of the stated concentrations.

12.4.4 The highest standard that meets this criterion defines the maximum concentration that can be reported for sample analysis without dilutions.

12.4.5 If the instrument is adjusted in any way that may affect the LDRs, new dynamic ranges must be determined. The LDR data must be documented and kept on file.

12.5 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.5.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.5.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.5.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.5.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.5.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024 Training.

12.1 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A

new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024 Training.

13.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Environmental Health and Safety Manual, and DV-HS-001P Waste Management Plan.

14.2 The following waste streams are produce when this method is carried out:

14.2.1 **Aqueous Acidic (Metals) -** Corrosive - Waste Stream J

14.2.2 **Expired reagents and standards –** Contact the Waste Coordinator.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 6020A: *Inductively Coupled Plasma - Mass Spectrometry*, Revision 1, February 2007.

15.1.2 Method 6020B: Inductively Coupled Plasma - Mass Spectrometry, Revision 2, July 2014.

15.1.3 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.

15.1.4 Method 3020A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy, Revision 1, July 1992.

15.1.5 Method 3050B, Acid Digestion of Sediments, sludges and soils, Rev. 2, Dec. 1996.

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/20/2010

15.3 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 5, July 2013.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 6020A	Commercially available standards are purchased and verified at the laboratory rather than being prepared from the solid material. These verification records are kept on file with QA.
2	EPA 6020A	Milli-Q or Nanopure water is substituted when reagent water is called for. This water is tested to be free of contaminants by conductivity (18megOhm) and by the analysis of blanks.
3	EPA 6020A	Corrective action for a PDS failure will be limited to flagging the PDS indicating the failed analyte and the recovery rather than diluting and reanalyzing the sample.
4	EPA 6020A	Internal standard recoveries are based on the intensities of the internal standards in the most recent calibration blank rather than the intensities of the internal standards in the initial calibration standard.
5	EPA 6020A	Method 6020A states that the dilution test is applicable if the matrix sample is at least 50x the reporting limit. Eurofins TestAmerica uses the tighter limit of 50x the MDL.
6	EPA 6020B	Method 6020B states to tune the instrument according to manufacturer's instructions. Eurofins TestAmerica continues to use the tune requirements in Method 6020A in order to be able to run samples by either method under the same tune.
7	EPA 6020B	Method 6020B does not include the analysis of the ICS-AB. Eurofins TestAmerica continues to analyze this QC sample due to various program requirements.
8	EPA 6020A/B	The tuning criteria listed in method 200.8 for mass resolution is used to satisfy the requirements of method 6020A/B

17.0 Attachments

Attachment 1: Standard Reporting Limits for Water and Soil
Attachment 2: Internal Standards Gas Modes and Corresponding Metals
Attachment 3: Interference Check Sample Components and Concentrations
Attachment 4: Suggested Mass Choices
Attachment 5: Tuning Solution and P/A Solution
Attachment 6: Suggested Tuning and Response Factor Criteria
Attachment 7: Summary of Quality Control Requirements
Attachment 8: Calibration, Calibration Verification, and Spike Concentrations
Attachment 9: Troubleshooting

18.0 Revision History

This section has been added beginning with Revision 0. Only details of the last two revisions are incorporated into this SOP. Prior revisions are documented in the QA files and available upon request.

Revision 13, Dated 01 October 2021

- Added Section 10.7.5.

Revision 12, Dated 19 August 2021

- Removed Attachments 2 and 3
- Updated current Attachment 2
- Added reference to Attachment 2 in Section 4.2.1

Attachment 1

Standard Reporting Limits for Water and Soil

Element Name	Element Symbol	Water (ug/L)	Soil (ug/Kg)
Aluminum	Al	50	5,000
Antimony	Sb	2.0	200
Arsenic	As	5.0	600
Barium	Ba	1.0	200
Beryllium	Be	1.0	100
Cadmium	Cd	1.0	100
Chromium	Cr	2.0	200
Cobalt	Co	1.0	100
Copper	Cu	2.0	250
Iron	Fe	50	5,000
Lead	Pb	1.0	150
Lithium	Li	50	5,000
Manganese	Mn	1.0	250
Molybdenum	Mo	2.0	200
Nickel	Ni	2.0	150
Selenium	Se	5.0	500
Silver	Ag	5.0	100
Strontium	Sr	10	100
Thallium	Tl	1.0	100
Thorium	Th	5.0	200
Tin	Sn	10	2,500
Tungsten	W	5.0	---
Uranium	U	1.0	100
Vanadium	V	5.0	500
Zinc	Zn	10	1,000
Zirconium	Zr	0.5	---

Attachment 2
Internal Standards, Gas Modes and Corresponding Analytes

Instruments 077 and 078			
IS	**Mass**	**Gas Mode**	**Associated Analytes**
^{6}Li	6	H	Be,
Ge	72	H	Se
Ge	72	He	V, Cr, Mn, Co, Ni, Cu, Zn, As, Sr
In	115	He	Mo, Ag, Cd, Sn, Sb, Ba
Ho	165	He	W, Tl, Pb, Th, U

Attachment 3
Interference Check Sample Components and Concentrations

Interference Component	Solution A Concentration (mg/L)	Solution AB Concentration (mg/L)
Al	100.0	110.0
Ca	100.0	110.0
Fe	100.0	110.0
Mg	100.0	110.0
Na	100.0	110.0
P	100.0	100.0
K	100.0	110.0
S	100.0	100.0
C	200.0	200.0
Cl	1000.0	1000.0
Mo	2.0	2.1
Ti	2.0	2.1
As	0.0	0.1
Sb	0.0	0.1
Be	0.0	0.1
Ba	0.0	0.1
Cd	0.0	0.1
Cr	0.0	0.1
Co	0.0	0.1
Cu	0.0	0.1
Pb	0.0	0.1
Li	0.0	1.0
Mn	0.0	0.1
Ni	0.0	0.1
Nb	0.0	0.2
Pd	0.0	0.1
Pt	0.0	0.1
Se	0.0	0.1
Sr	0.0	0.1
Tl	0.0	0.1
Th	0.0	0.1
Sn	0.0	0.1
Ag	0.0	0.1
U	0.0	0.1
V	0.0	0.1
W	0.0	0.1
Zn	0.0	0.1

Attachment 4
Suggested Mass Choices

Boldface masses indicate the masses which must have the most impact on data quality and the elemental equations used to collect the data. It is strongly recommended that elements other than those of interest be monitored to indicate other potential molecular interferences which could affect the data quality.

Mass	Element of Interest
"**27**"	Aluminum
121, "**123**"	Antimony
"75"	Arsenic
138, "137", 136, **135**, 134, 132, 130	Barium
"9"	Beryllium
114, 112, "111", 110, 113, 116, 106	Cadmium
42, 43, **44**, 46, 48	Calcium
"**52**", **53**, **50**, 54	Chromium
"**59**"	Cobalt
"**63**", **65**	Copper
56, **54**, **57**, 58	Iron
"**208**", "**207**", "**206**", 204	Lead
"7"	Lithium
24, **25**, **26**	Magnesium
"**55**"	Manganese
58, "**60**", 62, **61**, 64	Nickel
93	Niobium
105	Palladium
195	Platinum
39	Potassium
80, **78**, "**82**", **76**, **77**, 74	Selenium
"**107**", **109**	Silver
23	Sodium
"88"	Strontium
"**205**", 203	Thallium
232	Thorium
192	Tungsten
"**51**", **50**	Vanadium
64, "**66**", **68**, **67**, 70	Zinc
139	Lanthanum
118	Tin
238	Uranium
35, 37	Chlorine
98, 96, 92, **97**, 94, "95"	Molybdenum
72	Germanium (IS)
165	Holmium (IS)
115	Indium (IS)
6	Lithium (6+) (IS)
45	Scandium (IS)

Attachment 5: Tuning Solution and P/A Solution

A tuning solution containing elements representing all of the mass regions of interest must be analyzed. Below is a suggested solution covering a typical mass calibration range. Instrument manufacturer recommendations should be followed for tuning solutions.

The P/A solution is used to monitor the correlation between the Pulse and Analog parts of the electron multiplier. This solution is prepared at different concentrations depending on the current instrument conditions. The parent standard concentration is shown below.

Element	Tuning Concentration (µg/L)	P/A Concentration (mg/L)
Al		5
As		20
Ba	10	5
Be	40	20
Bi		5
Cd		20
Ce	10	
Co	10	5
Cr		5
Cu		5
Ge		10
In	10	5
Ir		5
^{6}Li		5
Li	10	
Lu		5
Mg	20	10
Mn		5
Mo		10
Na		5
Ni		10
Pb	10	10
Pd		10
Rh	10	
Ru		10
Sb		10
Sc		5
Sn		10
Sr		5
Tb		2.5
Th		50
Ti		50
Tl	10	50
U	10	50
V		50
Y	10	2.5
Zn		20

Attachment 6:

Suggested Tuning and Response Factor Criteria

Minimum Response from Tuning Solution:

Be	>1,000
Mg	>2,000
Rh	>20,000
Pb	>10,000
Li	>2,000
Co	>20,000
In	>1,000
Tl	>1,000

Suggested Mass Calibration:

Be	9.0122
Mg	23.98
Rh	102.91
Pb	207.98
Li	7.016
Co	58.9332
In	114.904
Tl	204.9744

Attachment 7:

Summary of Quality Control Requirements

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
LLOQ (6020B only)	With initial setup, Quarterly and on an as needed basis	65 - 135% recovery or in-house limits	Terminate analysis; correct the problem; recalibrate.
LLICV (6020A only)	Beginning of every analytical run.	70 - 130% recovery. 6020A IS, 70-150% rec.	Terminate analysis; correct the problem; recalibrate.
ICV	Beginning of every analytical run.	90 - 110% recovery.	Terminate analysis; correct the problem; recalibrate.
ICB/CB	Immediately after each ICV	The result is < ½ RL.	Terminate analysis; correct the problem; recalibrate.
LLCCV (6020A only)	Beginning and end of run and every 10 samples OR every 2 hours, whichever is more frequent.	70 - 130% recovery. 6020A IS, 70-150% rec.	See Section 10.6.10. Reanalyze twice in succession. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCV.
CCV	Beginning and end of run and every 10 samples OR every 2 hours, whichever is more frequent.	90 - 110% recovery.	Reanalyze twice in succession. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCV.

Attachment 7: Summary of Quality Control Requirements (Continued)

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
CCB	Immediately following each CCV.	The result must be < ½ RL. .	Reanalyze once. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCB.
ICSA	Beginning and every 12 hours.	Monitor for possible interferences.	See Section 9.5
ICSAB	Immediately following each ICSA.	Monitor for possible interferences.	See Section 9.5
Method Blank	One per lot of 20 field samples or fewer.	The result must be < ½ RL. Sample results greater than 10x the blank concentration or samples for which the contaminant is < RL, do not require redigestion or reanalysis.	Re-run once. If > ½ RL, redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.
Serial Dilution	One per batch of 20 field samples or fewer.	90 - 110% recovery	See Section 9.7 for additional requirements.
Post-Digestion Spike	One per batch of 20 field samples or fewer.	80-120% recovery	See Section 9.8.
Laboratory Control Sample	One per batch of 20 field samples or fewer.	Must be within laboratory control limits	See Section 9.3
Matrix Spike	One per lot of 20 field samples or fewer.	Must be within laboratory control limits	See Section 9.6 for additional requirements.

Attachment 8
Calibration, Calibration Verification, and Spike Concentrations

Element	Initial Calibration (µg/L)	ICV (µg/L)	CCV (µg/L)	LCS (µg/L)	MS/MSD (µg/L)	Post Digestion Spike (ug/L)	CCVL (ug/L)
Aluminum	2000	800	1000	400	400	20000	50
Antimony	100	40	50	40	40	200	2
Arsenic	100	40	50	40	40	200	5
Barium	100	40	50	40	40	200	1
Beryllium	100	40	50	40	40	200	1
Cadmium	100	40	50	40	40	200	1
Chromium	100	40	50	40	40	200	2
Cobalt	100	40	50	40	40	200	1
Copper	100	40	50	40	40	200	2
Iron	2000	800	1000	400	400	20000	50
Lead	100	40	50	40	40	200	1
Lithium	200	80	100	100	100	200	10
Manganese	100	40	50	40	40	200	1
Molybdenum	100	40	50	40	40	200	2
Nickel	100	40	50	40	40	200	1.5
Selenium	100	40	50	40	40	200	5
Silver	100	40	50	40	40	50	5
Strontium	200	80	100	40	40	200	1
Thallium	100	40	50	40	40	200	1
Thorium	100	40	50	40	40	--	2
Tin	100	40	50	40	40	200	10
Tungsten	100	40	50	40	40	200	5
Uranium	100	40	50	40	40	200	1
Vanadium	100	40	50	40	40	200	5
Zinc	100	40	50	40	40	200	10
Zirconium	100	40	50	40	40	--	--

This procedure has been developed for twenty elements. Additional elements may be included in the calibration solution at the above levels. Levels may be adjusted to meet specific regulatory or client programs.

Attachment 9

ICP-MS Troubleshooting Guide

Problem	Possible Cause/ Solution
High Calibration Blanks	Inspect historical blank data to determine root cause Inspect, clean or replace torch Inspect, clean or replace pump tubing or sample tubing Inspect, clean or replace nebulizer Remake blank solution Recalibrate instrument
Instrument Drift	Make sure instrument has warmed properly Condition cones to aid stability Reslope to correct for changing cone conditions during run Stop run, clean cones and start over with a new calibration
Erratic Readings, High RSDs	Check nebulizer pressure Check sample flow around the pump, adjust tension on pump tubing to ensure smooth flow Check for clogs in the uptake tubing, nebulizer, or valve Clean or replace nebulizer
Low Sensitivity	Clean cones Adjust lens voltages Remove and clean lens, remove and clean or replace reaction cell
Bad Tune: Bad Mass Cal	Adjust lens voltages, remove and clean lens
Bad Tune: High Oxides	Inspect, clean, or replace torch, nebulizer, and spray chamber

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Title: Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series

Approvals (Signature/Date):

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1.0 Scope and Application

1.1 This Standard Operating Procedure (SOP) is applicable to the solvent extraction of organic compounds from water samples, TCLP leachates, and SPLP leachates, using a separatory funnel. This SOP based on SW-846 Method 3510C, EPA 608, EPA 610, EPA 614, AK102, NWTPH-Dx, and Oklahoma DRO method.

1.2 The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate pH and spiking mixtures are used.

1.3 This procedure does not include the concentration and cleanup steps. See SOP DV-OP-0007 Concentration and Clean-up of Organic Extracts for details concerning the concentration and cleanup of extracts.

2.0 Summary of Method

A measured volume of sample, is placed in a separatory funnel. The pH is adjusted as required for the efficient extraction of specific compounds. The organic compounds are extracted with three portions of methylene chloride. The water phase is discarded. The organic phase is dried using sodium sulfate.

3.0 Definitions

Refer to the Glossary of the Eurofins TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P Quality Control Program for definitions of general analytical and QA/QC terms.

3.1 **Extraction Holding Time**: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.2 **Preparation Batch**: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards

3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. Please reference WI-DV-0032 Guidelines for Setting up Method Comments for details on Method Comments.

3.4 **Quality Assurance Summary (QAS)**: Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in LIMS. In these situations, laboratory Project Managers describe the special requirements in a written QAS. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.5 **Aliquot**: A part that is a definite fraction of a whole; as in “take an aliquot of a sample for testing or analysis.” In the context of this SOP, “aliquot” is also used as a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

3.6 **Reagent Water** (aka ELGA water – water generated from ELGA water polishing units): Water with a resistivity of 1 Megohm-cm or greater. The Eurofins TestAmerica Denver deionized water supply meets this requirement with a resistivity of at least 10 Megohm-cm.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.3 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented in an NCM.

4.4 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them. Especially take note of the possibility of phthalate contamination from gloves. Gloves should be changed out frequently and whenever they come in contact with solvent. Glassware should be handled in a fashion that keeps gloves away from the interior and mouth of the glassware.

4.5 The decomposition of some analytes has been demonstrated under basic extraction conditions. Organochlorine pesticides may dechlorinate, phthalate esters may exchange, and phenol may react to form tannates. These reactions increase with increasing pH, and are decreased by the shorter reaction times available in Method 3510C. Method 3510C is preferred over Method 3520C for the analysis of these classes of compounds. However, the recovery of phenols is optimized by using Method 3520C and performing the initial extraction at the acid pH.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 The use of separatory funnels to extract samples using methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity. Either a face shield must be worn over safety glasses or goggles must be worn when it is performed.

5.3.2 Glass centrifuge tubes can break in the centrifuge if proper care is not taken. This can lead to a hazardous material spill and endanger employees. Do not exceed the manufacturer's recommended maximum RPM for glass containers. Normally speeds greater than 2700 rpm are not advisable.

5.3.3 The procedure calls for the use of an electric rotator. The rotator is equipped with a safety latch that does not allow the rotator to rotate even if the power switch is turned on. The separatory funnels are secured to the rotator using straps. During the procedure it will be necessary to loosen the straps in order to un-stopper the separatory funnels. Whenever the straps are loose, the safety latch must be fastened to prevent the rotator from rotating.

5.3.4 Glasswool is a carcinogen and therefore should be handled in a hood to avoid inhalation of dust.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Materials with Serious or Significant Hazard Rating

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Sodium Hydroxide	Corrosive Poison	2 mg/m3	Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, and runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and can cause burns that may result in permanent impairment of vision, even blindness with greater exposures.
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric Acid	Corrosive Carcinogen	1 mg/m3	Inhalation may cause irritation of the respiratory tract with burning pain the nose and throat, coughing, wheezing, shortness of breath, and pulmonary edema. Causes chemical burns to the respiratory tract. Inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Causes skin burns. Causes severe eye burns. May cause irreversible eye injury, blindness, permanent corneal opacification.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit			

6.0 Equipment and Supplies

NOTE: All glassware used in this procedure is cleaned following SOP DV-OP-0004 Glassware Washing of Organic Analysis Applications. In addition, the glassware is rinsed with methylene chloride immediately prior to use.

6.1 Supplies

- Separatory funnel, 2-liter with polytetrafluoroethylene (PTFE) stopcock and stopper.
- Separatory funnel, 500-mL with polytetrafluoroethylene (PTFE) stopcock and stopper.
- Separatory funnel rack and mechanical rotator.
- Balance, ≥ 1400 g capacity, accurate to ± 1 g, calibration checked daily per SOP DV-

QA-0014 Selecting and Using Balances.

- pH indicator paper, wide range.
- Class A Graduated Cylinder, sizes ranging from 50 mL to 1 L.
- Media bottles, 300 mL with Teflon-lined caps or capped with aluminum foil.
- Media bottles, 100 mL with Teflon-lined caps or capped with aluminum foil.
- Disposable pipettes, various volumes.
- Stemless glass funnel.
- Glass wool, baked at 400 °C for four hours.
- Mechanical pipette, 1 mL, positive displacement, with disposable tips, calibrated per SOP DV-QA-0008 Volumetric Verification.
- Aluminum foil.
- Paper towels.

6.2 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Reagent Water

Eurofins TestAmerica Denver has two ELGA water purification systems. The water coming from the ELGA system should be 18-18.2 Mohm-cm. The performance of the water polishing system is checked daily and recorded per SOP DV-QA-0026 DI Water Monitoring.

7.2 Methylene Chloride

Each lot of solvent is tested following SOP CA-Q-S-001 Acid and Solvent Lot Testing and Approval Program before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.3 Acids and Bases

7.3.1 1:1 Sulfuric Acid (H2SO4), *TALS Reagent ID "1:1 H2SO4"*

Place an ice water bath on a stir plate. Place a container with a magnetic stir bar in the bath. While stirring, slowly add 1 part concentrated reagent grade sulfuric acid (36N) to 1 part water from the ELGA purification system. Assign a 1 year expiration date from the date made or the vender expiration date, whichever is shorter.

7.3.2 10N Sodium Hydroxide (NaOH), *TALS Reagent ID "10N_NaOH"*

Purchased at ready-to-use concentration from commercial vendors. Assign a 1 year expiration date from the date opened or the vender expiration date, whichever is shorter.

7.3.3 1N Hydrochloric Acid (HCl), *TALS Reagent ID "1N_HCl"*

Dilute 100 mL of stock reagent grade, concentrated HCl to 1000 mL with reagent water.

7.4 Baked Sodium Sulfate, 12-60 mesh

Heat sodium sulfate in a 400 °C oven for at least four hours. Store in tightly closed container.

7.5 Baked Sodium Chloride

Bake in 400 °C oven for at least 4 hours.

7.6 Standards

Please reference SOP DV-OP-0020 Preparation, Verification, and Storage of Organic Prep Surrogates and Spike Standards and WI-DV-0009 Spike/Surrogating and Review Procedure for Organic Extractions for information regarding the surrogate and spike standards used in this procedure.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix and Method	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Water	Amber Glass	1000 mL	Cool, ≤ 6°C	7 Days	40 CFR Part 136.3
Water for Method AK 102	Amber Glass	1000 mL	Cool, ≤ 6°C and pH ≤ 2 with HCl	14 Days if properly preserved. 7 Days if un-preserved.	Method AK 102
Water for Method Oklahoma DRO	Amber Glass	1000 mL	Cool, ≤ 6°C and pH ≤ 2 with HCl	7 Days	Oklahoma Dept. of Environmental Quality
Water for Method NWTPH-DX	Amber Glass	1000 mL	Cool, ≤ 6°C and pH ≤ 2 with HCl	7 Days	NWTPH-Dx
Water for Method 8082 or 8082A	Amber Glass	1000 mL	Cool, ≤ 6°C	None [2]	SW-846 Chapter 4, Revision 4, Feb 2007
Water for Method 8081 or 8082 by Large Volume Injection	Amber Glass	250 mL	Cool, ≤ 6°C	7 Days	40 CFR Part 136.3
Water for Method 8270SIM by Large Volume Injection	Amber Glass	250 mL	Cool, ≤ 6°C	7 Days	40 CFR Part 136.3
TCLP Leachates	Glass	200 mL for 8270 100 mL for 8081 100mL for 8141	Cool, ≤ 6°C	7 Days from the start of the leach	SW-846 1311
SPLP Leachates	Glass	1000 mL	Cool, ≤ 6°C	7 Days from the start of the leach	SW-846 1312

[1] Exclusive of analysis.

[2] Some regulatory agencies do not accept SW-846 Revision 4 of Chapter 4 and will require a 1 week hold time for method 8082 and 8082A. The states of California, South Carolina, Pennsylvania, and Connecticut require a 1 week hold time.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in Eurofins TestAmerica Denver policy DV-QA-003P Quality Control Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in Eurofins TestAmerica Denver policy DV-QA-024P QA/QC Requirements for Federal Programs. This procedure meets all criteria for DoD QSM unless otherwise stated. Any deviation or exceptions from QSM requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 Non-Conformance and Corrective Action System. This is in addition to the corrective actions described in the following sections.

9.2 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P Quality Control Program for further details.

9.3 Method Blank (MB)

9.3.1 One method blank must be processed with each preparation batch. The method blank is processed and analyzed just as if it were a field sample.

9.3.2 The method blank for batches of aqueous samples for Large Volume Injection (prep method 3510C_LVI) consists of 250mL of reagent water free of any of the analyte(s) of interest.

9.3.3 The method blank for batches of aqueous samples for all other methods consists of 1 L of reagent water free of any of the analyte(s) of interest.

9.3.4 The method blank for batches of TCLP leachates for methods 8081 and 8141 consists of 100 mL of leach fluid.

9.3.5 The method blank for batches of TCLP leachates for method 8270 consists of 200 mL of leach fluid.

9.3.6 The method blank for batches of SPLP leachates consists of 1 L of leach fluid.

9.4 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

9.4.1 At least one LCS must be processed with each preparation batch. The LCS is carried through the entire analytical procedure just as if it were a sample.

9.4.2 The LCS for batches of aqueous samples for Large Volume Injection (prep method 3510C_LVI) consists of 250mL of reagent water to which the analyte(s) of interest are added at known concentrations.

9.4.3 For aqueous sample batches for all other methods, the LCS consists of 1 L of reagent water to which the analyte(s) of interest are added at known concentration.

9.4.4 For methods 8081 and 8141 TCLP leachates, the LCS consists of 100 mL of leach fluid to which the analyte(s) of interest are added at known concentration.

9.4.5 For method 8270 TCLP leachates, the LCS consists of 200 mL of leach fluid to which the analyte(s) of interest are added at known concentration.

9.4.6 For SPLP leachates, the LCS consists of 1 L of leach fluid to which the analyte(s) of interest are added at known concentration.

9.4.7 Method 608, 614, 610 requires a LCS at a 10% frequency. In other words one LCS is required for a batch of 10 or less samples. A LCSD is required for a batch of 11 or more samples.

9.4.8 Method AK102 requires LCS and a LCSD for every batch for every spike compound.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.5.1 One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

9.5.2 If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared unless Method Comments indicate otherwise. DoD requires the MS/MSD to be assigned by the client. For DoD QSM specific criteria see SOP DV-QA-024P QA/QC Requirements for Federal Programs. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

9.5.3 Method 608, 610, and 614 requires one matrix spike for every 10 samples. If the batch has more than 10 samples, then two matrix spikes must be performed. The two matrix spikes are to be performed on two different samples. If there is insufficient sample volume for matrix spikes, then a LCSD must be performed.

9.5.4 Method NWTPH-Dx requires a matrix spike and a matrix spike duplicate for every 10 samples. If insufficient sample volume is available for MS/MSD, a NCM must be written and a LCS and LCSD must be performed for every 10 samples.

9.6 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 Non-Conformance and Corrective Action System. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

10.4 Critical Procedural Considerations

10.4.1 As stated throughout this SOP, analysts must review the Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009 Spike/Surrogating and Review Procedure for Organic Extractions).

10.4.2 Analyst must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any other separatory funnel than the designated one should be cleaned or disposed of before coming into contact with the sample.

10.5 Assemble and clean the glassware immediately before use.

NOTE: Rotate glassware; do **not** use specific glassware or positions for the MB and LCS/LCSD.

10.5.1 Place a stopcock in each separatory funnel. For 1-liter extractions use a 2000 mL separatory funnel. For 250 mL, 200 mL and 100 mL extractions, use a 500 mL separatory funnel. Place a stopper for each separatory funnel on a clean sheet of aluminum foil that is marked with individual positions for each stopper. This is done to prevent cross-contamination.

NOTE: Samples logged with method 3510_LVI are for Large Volume Injection methods and require 250 mL initial volumes. Samples logged for 8270

with a TCLP pre-prep require 200mL initial volumes. Samples logged for 8081 and 8141 with a TCLP pre-prep require 100 mL initial volumes.

10.5.2 For each separatory funnel, plug a glass funnel with baked glass wool and add baked sodium sulfate. Place the funnel on a media bottle and place the media bottle below the separatory funnel.

10.5.3 Rinse each separatory funnel once with methylene chloride. Be sure that all surfaces come into contact with the solvent. Drain the methylene chloride into the media bottle through the sodium sulfate.

10.5.4 Rinse the sodium sulfate with additional methylene chloride if the first rinse did not completely saturate the sodium sulfate.

10.5.5 Allow the methylene chloride to drain completely into the media bottle. Swirl the media bottle to ensure all surfaces come into contact with the solvent. Add additional methylene chloride to the rinse if necessary.

10.5.6 Discard the methylene chloride.

10.5.7 Label each media bottle with the sample ID or batch QC ID.

10.6 Prepare LCS and Method Blank Samples

NOTE: For SW-846 methods if there is not a MS/MSD pair in the batch then perform a LCS/LCSD. Methods 608, 610, and 614 require a LCS and LCSD in batches of 11 samples or more or if there are no Matrix Spikes in batches of 10 or less.

10.6.1 For aqueous sample batches logged for Large Volume Injection, (3510_LVI), pour 250 mL of reagent water into the separatory funnels marked for the LCSs and the MB.

10.6.2 For all other aqueous sample batches, pour 1 liter of reagent water into the separatory funnels marked for the LCSs and the MB.

10.6.3 For 8270 TCLP leachates, use a 250 mL or 500 mL Class A graduated cylinder to measure out 200 mL of the appropriate leach fluid for each MB and LCS and LCSD. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.6.4 For 8081 and 8141 TCLP leachates, use a 100 mL or 250 mL Class A graduated cylinder to measure out 100 mL of the appropriate leach fluid for each MB and LCS and LCSD. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.6.5 For SPLP leachates, use a 1000 mL Class A graduated cylinder to measure out 1000 mL of the appropriate leach fluid for each MB and LCS and LCSD. Record the volume to the nearest 10 mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.7 Measure the initial sample pH of the samples.

10.7.1 Measure the initial sample pH with wide-range pH paper and record the pH on the extraction bench sheet.

10.7.2 If the sample is logged for AK102_103, Okla_DRO, or NWTPH_Dx the samples should have been field preserved. See Section 8. If the samples are not preserved, an NCM should be written.

10.8 Aliquot the samples

10.8.1 For 8270 TCLP leachates, use a 250 mL or 500 mL Class A graduated cylinder to measure out 200 mL of the leachate. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.8.2 For 8081 and 8141 TCLP leachates, use a 100 mL or 250 mL Class A graduated cylinder to measure out 100 mL of the leachate. Record the volume to the nearest mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.8.3 For SPLP leachates, use a 1 Liter Class A graduated cylinder to measure out 1000 mL of the leachate. Record the volume to the nearest 10 mL. Place the leachate bottle beside the separatory funnel so a second analyst can check that the correct leach fluid was used.

10.8.4 For water samples, it should be noted that Eurofins TestAmerica Denver routinely aliquots gravimetrically. This is done to prevent cross-contamination due to volumetric glassware and to provide a more accurate initial volume measurement. However, some clients and regulatory programs require the laboratory to aliquot samples volumetrically. The Method Comments and QASs must be read before samples are aliquotted to check for this requirement. If samples are to be aliquotted volumetrically, use Class A volumetric glassware only and proceed to Section 10.8.6

10.8.5 Weigh the bottle (250 mL amber bottles for 3510C_LVI or 1000 mL amber bottles for all other aqueous samples) and record the gross weight to the nearest gram. If there is any indication that the sample's density is not 1 g = 1 mL, then measure the density of the sample using a calibrated pipette and an analytical balance. The weight of the sample extraction will be corrected for the density later. See Section 11 for the calculation. For example, normally a 1 liter bottle weighs 500 g when empty and when filled completely can only hold 1060 mL, therefore a full bottle weighing more than 1560 g is an indication that either the sample density is greater than 1g or the sample bottle contains a lot of sediment. A wide mouth bottle should weigh no more than 1367 g. Document any sample with a density greater than 1.01 g in an NCM.

10.8.6 Inspect the samples for large amounts of sediment that may interfere with the extraction of the sample by causing excessive emulsions or clogging the stop-cock.

10.8.6.1 If the sample contains so much sediment that the entire sample volume cannot be extracted, decant the sample into the separatory funnel (or a 1 L graduated cylinder if volumetric aliquotting is required), careful not to transfer the sediment. Write a NCM to document the sediment and that it prevented the entire sample volume from being extracted and the sample container from being solvent rinsed. This is considered a deviation and must be documented in a NCM.

10.8.6.2 If the sample does not contain a significant amount of sediment, then the entire sample volume will be used in the extraction.

10.8.6.3 For the 600 method series: if there is no more than an inch of sediment in the bottom of the sample bottle, shake the sample well and determine if the sediment resettles in approximately 1 minute. If not, the density of the sediment is likely to be low enough to stay suspended and not block the sidearm.

10.8.6.4 For the 600 method series: if the density of the sediment is high and likely to cause a problem in the extraction or if there is more than an inch of sediment contact the PM so that the client's input can be obtained. Not extracting the entire sample and rinsing the bottle with the extraction solvent is a method deviation. If the client concurs that the sample can be decanted write an NCM to describe the deviation from the procedure.

10.8.7 Place the sample containers in front of the separatory funnel labeled for that sample. A second analyst should then check the labels to make sure the correct sample is being extracted. This check is documented in the Organic Extraction Checklist (WI-DV-0009 Spike/Surrogating and Review Procedure for Organic Extractions)

10.9 If volumetric aliquotting is required, transfer the entire sample into a Class A graduated cylinder and record the volume on the benchsheet. If the sample bottle contains more than 1000 mL, a 100 mL Class A graduated cylinder can be used to complete the measurement. The entire sample volume must be used. Record the volume to the nearest 10 mL. Then pour the sample into the labeled separatory funnel. Place the used graduated cylinder in front of the appropriate separatory funnel so it can be solvent rinsed later.

NOTE: A 1000 mL Class A graduated cylinder is not accurate enough to measure to the nearest 1 mL. Therefore all samples that are aliquoted using a 1000 mL Class A graduated cylinder will have the initial volume recorded to the nearest 10 mL. This accuracy is sufficient.

10.10 If volumetric aliquotting is not required, pour the sample directly into the separatory funnel. Place the empty sample container in front of the appropriate separatory funnel so it can be solvent rinsed.

10.11 Add Surrogates to All Field Samples and QC Samples

10.11.1 The standards should be allowed to come to room temperature before spiking the samples. Record the ID of the standard used on the benchsheet.

NOTE: The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch Reference work instruction WI-DV-0009 Spike/Surrogating and Review Procedure for Organic Extractions to determine the appropriate standard and the appropriate volume required.

10.11.2 Only one batch should be surrogated at a time to ensure the correct standards are used.

10.11.3 Add the appropriate volume of the appropriate working surrogate standard to the separatory funnel for each sample, MB, LCS, and MS/MSD. Record the ID of the standard used on the bench sheet. Reference work instruction WI-DV-0009 Spike/Surrogating and Review Procedure for Organic Extractions to determine the appropriate standard and the appropriate volume required.

10.12 Add Spikes to all LCS's and MS/MSDs

10.12.1 Add the appropriate volume of the appropriate working spike standard to the separatory funnels for the MS/MSD, LCS and/or LCSD samples. Record the ID of the standard used on the bench sheet. Reference work instruction WI-DV-0009 Spike/Surrogating and Review Procedure for Organic Extractions to determine the appropriate standard and the appropriate volume required.

10.13 Add approximately 6g (1 teaspoon) of NaCl to all samples and all QC samples. This is done to give the reagent water used in the MBs and LCSs some ionic strength to more closely mimic the matrix of actual water samples and to aide in the extraction of the more polar target compounds. Record the lot number of the sodium chloride on the bench sheet.

NOTE: Per the South Carolina QAS, do NOT add NaCl to South Carolina samples or associated QC. South Carolina samples should be prepared separately from other samples in order to meet this requirement.

10.14 Adjust pH of Field Samples and QC Samples

Adjust the sample pH as indicated in the chart below using a minimum amount of 1:1 sulfuric acid (or 1 M hydrochloric acid for Methods AK102, Okla_DRO and NWTPH_Dx) or 10 N sodium hydroxide, as necessary. Record the adjusted pH and the lot number of the acid or base on the bench sheet. For TCLP leachates by method 8270, usually 1 mL of 1:1 sulfuric acid is sufficient.

NOTE: TCLP Leachates may have pH of < 5. In those cases, the pH should be adjusted per the table below.

Method	Initial Extraction pH	Secondary Extraction pH
All 8270 methods *except* SIM.	1 – 2	If samples are TCLP leachates extract at 14. If samples are water extract at 11 - 12
All 8270 SIM methods	As Received	None
All 8081, 8082 and 608 methods.	5 - 9	None
All 8141 and 614 methods	5-8	None
All 8015 methods	As Received	None
All 8310 and 610 methods	As Received	None
AK102_103 Okla_DRO NWTPH_Dx	If samples are preserved between pH 1 – 2, then acidify the MB and LCS. Otherwise extract as received and document insufficient preservation in an NCM.	None

10.15 For 1 Liter samples, add 60 mL of methylene chloride to each empty sample container, unless the entire sample volume was not used. For 250 mL or smaller samples, add 30 mL of methylene chloride to each empty sample container, unless the entire sample volume was not used. Cap the container and shake gently to rinse all internal surfaces of the bottle. Pour the methylene chloride from the sample container into the appropriate separatory funnel. If a graduated cylinder was used to aliquot volumetrically, rinse the cylinder and add that rinse to the separatory funnel as well. Record the lot number of the methylene chloride on the bench sheet. If the sample contained significant sediment and the entire sample contents could not be extracted, do not rinse the empty sample container, but instead add the solvent directly to the separatory funnel. If the solvent rinse of the sample container cannot be performed, prepare a NCM.

10.16 For water samples that were aliquotted gravimetrically, reweigh the bottle and calculate the initial sample volume by subtracting the empty bottles weight from the full bottles weight, assuming a density of 1 g = 1 mL. If there is any indication that the samples density is not 1 g = 1 mL then measure the density of the sample and correct the calculated initial volume accordingly using the formula in Section 11. Document abnormal sample density in an NCM. For example, normally a 1 liter bottle when filled completely can only hold 1060 mL, therefore an initial volume greater than 1060mL is an indication that the density is not 1 g. Document any sample with a density greater than 1 g in an NCM.

10.17 If the initial volume is less than 80% of the nominal volume, the sample reporting limits and method detection limits will be elevated substantially. Document this in a NCM.

10.18 Stopper and rotate the separatory funnel for 3 minutes with periodic venting to release excess pressure. Document the extraction date and time on the benchsheet.

WARNING: Methylene chloride creates excessive pressure very rapidly! Therefore, initial venting should be done immediately after the separatory funnel has

been sealed and shaken a few seconds. Vent into hood away from people and other samples. A face shield or goggles must be worn during venting.

10.19 Allow the organic layer to separate from the water phase for at least 5 minutes or until complete visible separation has been achieved. This can take up to 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, use mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, pouring the solvent layer and emulsion back through the top of the separatory funnel (pour-back), or centrifugation. The emulsion could also be filtered through the glass funnel by adding additional sodium sulfate to remove all water in the emulsion. This technique should only be used after other techniques have failed to make complete phase separation and only after the last shake.

NOTE 1: If an emulsion forms, the analyst does not have to wait a complete 5 minutes before attempting to break the emulsion with pour-backs and centrifuge. Start employing the mechanical techniques right away to achieve phase separation.

NOTE 2: As much as 15 to 20 mL of methylene chloride is expected to dissolve in 1 L of water. Thus, solvent recovery could be as low as 35 mL from the first shake and still be acceptable. Subsequent shakes should recover at least 50 mL of solvent.

10.20 Drain the lower methylene chloride layer into the sodium sulfate filled glass funnel. Allow the methylene chloride to drain completely into the media bottle. Rinse the sodium sulfate with a small amount of methylene chloride to ensure that all compounds of interest are collected in the media bottle. Record the lot number of the sodium sulfate on the bench sheet. If the sodium sulfate becomes saturated with water, add more to the funnel or replace the existing sodium sulfate with fresh drying agent.

10.21 Repeat the extraction two more times for a total of 3 extractions. Collect all three methylene chloride extracts in the same media bottle. For the 2nd and 3rd extractions it is not necessary to wait 5 minutes to allow the solvent to separate from the water; a 3 minute wait time should be sufficient.

10.22 For the base/neutral and acid extractable method 8270, adjust the pH of the samples according to chart in Section 10.14. For 8270 TCLP leachates an excess of base is required to effectively extract pyridine, therefore at least 7 mL of base should be used to ensure the pH is 14. Then extract the sample 3 more times. For these extractions, it is not necessary to wait 5 minutes to allow the solvent to separate from the water; a 3 minute wait time should be sufficient.

NOTE: For 8270 water extractions please note that typically 2 mL of acid is needed to achieve a pH 1-2; 5 mL of base is typically required to achieve the pH of 11-12.

10.23 Cap the media bottle with a Teflon-lined cap or aluminum foil and submit for concentration and possible clean-up steps.

10.24 Dispose of the solvent-saturated water remaining in the separatory funnel in the appropriate waste container. See Section 14.

10.25 Initial weights and volumes of samples are entered into LIMS, and the transcribed data must be verified by a second person. This verification is documented on the Organic Extraction Checklists (see WI-DV-0009 Spike/Surrogating and Review Procedure for Organic Extractions).

10.26 Troubleshooting

10.26.1 If the sample appears very dark or viscous or in any way un-like water, stop and test the sample's miscibility before attempting to extract the sample by this procedure. Place a few milliliters of sample in a vial with methylene chloride. Cap and shake. If the sample is miscible in methylene chloride, the sample should be re-logged as a waste matrix with a prep method of 3580A.

10.27 Maintenance

10.27.1 Approximately every 6 months, the centrifuge should be lubricated.

10.27.2 Contact the Facilities Manager immediately if the rotator is observed to be making un-familiar noises or rotating in a "jerking" manner.

11.0 Data Analysis and Calculations

11.1 Initial Volume calculation

$$InitialVolume(mL) = \frac{FullBottle(g) - EmptyBottle(g)}{Density(g/mL)}$$

11.2 The initial data review is performed by the analyst and a second-level review is performed by the area supervisor or designee. Both reviews are documented on DV-F-0045 Organic Extraction Department Checklist. See SOP DV-QA-0020 Data Review for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-S-006 Detection and Quantitation Limits. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM or for programs which require the use of Method 8270D, Revision 5. For DoD QSM specific criteria see SOP DV-QA-024P QA/QC Requirements for Federal Programs. A blank matrix is spiked at 1-2 the laboratory RL

and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

12.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024 Training.

12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024 Training.

13.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P Waste Management Plan.

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Methylene chloride – Waste Stream B

14.2.2 Solid waste/sodium sulfate – Waste Stream D

14.2.3 Basic aqueous sample waste saturated with methylene chloride – Waste Stream X.

14.2.4 Acidic aqueous sample waste saturated with methylene chloride – Waste Stream Y.

14.2.5 Neutral aqueous sample waste saturated with methylene chloride – Waste Stream X or Waste Stream Y.

14.2.6 Expired Standards/Reagents – Contact Waste Coordinator for guidance

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005, Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

15.2 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 608, Organochlorine Pesticides and PCBs.

15.3 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 610, Polynuclear Aromatic Hydrocarbons.

15.4 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 614, Organophosphorous Pesticides.

15.5 Alaska Method AK102, “For the Determination of Diesel Range Organics”, Version 04/08/02.

15.6 Alaska Method AK103, “For the Determination of Residual Range Organics”, Version 04/08/02.

15.7 NWTPH-Dx “Semi-Volatile Petroleum Products Method for Soil and Water.

15.8 Oklahoma Department of Environmental Quality Methods 8000/8100 (Modified) Diesel Range Organics (DRO) Revision 4.1 Date 10/22/97

16.0 Modifications:

16.1 Modifications from SW-846 Method 3510C

16.1.1 Section 7.1 of the method calls for initial sample volume to be determined volumetrically either by measuring out exactly 1 liter or marking the meniscus on the sample container and later determining the volume of water required to fill the bottle back up to the mark. This SOP allows the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware.

16.1.2 Section 7.5 of the method calls for shaking the separatory funnel 1-2 minutes. This SOP calls for shaking the separatory funnel for 3 minutes.

16.1.3 Section 7.6 of the method calls for allowing the organic layer to separate from the water phase for a minimum of 10 minutes. This SOP calls for allowing the organic layer to separate from the water phase for a minimum of 5 minutes after the first extraction and a minimum of 3 minutes for subsequent extractions, up to 10 minutes if the separation is not complete.

16.1.4 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

16.1.5 The source method calls for samples to be extracted for method 8141 at the pH they are received. This procedure calls for the extraction to be performed at a pH between 5 and 8. This is done per guidelines found in Section 2 and Section 8 of SW-846 8141B.

16.2 Modifications from 40 CFR Method 608 and 610

16.2.1 Section 10.1 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.

16.2.2 Section 10.2 of the method calls for shaking the separatory funnel 1-2 minutes. This SOP calls for shaking the separatory funnel for 3 minutes.

16.2.3 Section 10.2 of the method calls for allowing the organic layer to separate from the water phase for a minimum of 10 minutes. This SOP calls for allowing the organic layer to separate from the water phase for a minimum of 5 minutes after the first extraction and a minimum of 3 minutes for subsequent extractions, up to 10 minutes if the separation is not complete.

16.2.4 Section 10.3 of the method calls for rinsing the sample collection bottle with the 60 mL methylene chloride aliquot for the second and third extraction as well as the first extraction. This SOP calls for rinsing the sample collection bottle with only the first 60-mL methylene chloride aliquot.

16.2.5 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC

samples in order to help the extraction efficiency.

16.3 Modifications from 40 CFR Method 614

16.3.1 Section 10.1 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.

16.3.2 Section 10.2 of the method calls for the extraction to be performed with at 15% v/v methylene chloride in hexane solvent. This procedure uses methylene chloride for the extraction. SOP DV-OP-0007 Concentration and Clean-up of Organic Extracts calls for the methylene chloride extract to be concentrated and exchanged to hexane.

16.3.3 Section 10.2 of the method calls for shaking the separatory funnel 1-2 minutes. This SOP calls for shaking the separatory funnel for 3 minutes.

16.3.4 Section 10.2 of the method calls for allowing the organic layer to separate from the water phase for a minimum of 10 minutes. This SOP calls for allowing the organic layer to separate from the water phase for a minimum of 5 minutes after the first extraction and a minimum of 3 minutes for subsequent extractions, up to 10 minutes if the separation is not complete.

16.3.5 Section 10.3 of the method calls for rinsing the sample collection bottle with the 60 mL solvent aliquot for the second and third extraction as well as the first extraction. This SOP calls for rinsing the sample collection bottle with only the first 60-mL methylene chloride aliquot.

16.3.6 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

16.4 Modifications from Method AK 102

16.4.1 Section 9.1.1.1 of the method calls for using no more than 1 liter of sample and to determine the volume either by measuring out exactly 1 liter or marking the meniscus on the sample container and later determining the volume of water required to fill the bottle back up to the mark. This SOP allows the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware. This SOP allows for the extraction of more than 1 L as it calls for the use of the entire sample volume.

16.4.2 Section 9.1.1.6 of the method says to allow the water and solvent layers to separate for approximately 10 minutes. This SOP calls for the allowing the organic layer to separate from the water phase for a minimum of 5 minutes after the first extraction and a minimum of 3 minutes for subsequent extractions, up to 10 minutes if the separation is not complete.

16.4.3 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

16.5 Modifications from Method NWTPH-Dx

16.5.1 The method calls for determining the initial volume of the sample my marking the meniscus on the bottle and later determining the volume of tap water required to fill the bottle back up to the mark. This SOP allows the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware.

16.5.2 The method calls for shaking the separatory funnel for one minute. This SOP calls for the separatory funnel to be shaken for at least three minutes.

16.5.3 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

16.6 Modifications from Oklahoma DRO

16.6.1 The method calls for aliquotting 800 mL to 900 mL of the sample volumetrically. This SOP calls for the initial sample volume to be determined by weight in order to achieve a more accurate initial volume and to avoid cross-contamination via glassware. This SOP allows for the extraction of more than 1 L as it calls for the use of the entire sample volume.

16.6.2 The method calls for extracting using 50 mL of solvent. This SOP calls for the extraction to be done using at least 60 mL of solvent.

16.6.3 The method calls for shaking the separatory funnel for two minutes. This SOP calls for the separatory funnel to be shaken for at least three minutes.

16.6.4 The method calls for a method blank and LCS to be analyzed every 10 samples. This SOP calls for a method blank and LCS to be analyzed every batch of 20 samples.

16.6.5 The source method does not call for the use of sodium chloride. This procedure calls for the addition of approximately 6g of sodium chloride to all samples and all QC samples in order to help the extraction efficiency.

17.0 Attachments

Table 1. Determinative Methods Using Separatory Funnel Extractions

18.0 Revision History

This section has been added beginning with Revision 0. Only details of the last two revisions are incorporated into this SOP. Prior revisions are documented in the QA files and available upon request.

- **Revision 21.0, 11 June, 2021**
  - Removed QSM versions and instead referenced SOP DV-QA-024P QA/QC Requirements for Federal Programs for information about DoD QSMs.
  - Updated language and formatting throughout.

- **Revision 20.0, 23 April, 2021**
  - Annual review
  - Updated copyright Information
  - Changed TestAmerica to Eurofins TestAmerica throughout
  - Updated language and formatting throughout
  - The weight of a full wide mouthed bottle was added to Section 10.8.5

TABLE 1.

Determinative Methods Using Separatory Funnel Extractions

Method Description	*Determinative Method*	*SOP*
Diesel Range Organics & Jet Fuels	SW-846 8015, California LUFT Method, Alaska Methods AK102 & AK103 SW-846 8015C DRO SW-846 8015D DRO NWTPH-DX	DV-GC-0027
Chlorinated Pesticides	SW-846 8081A SW-846 8081B EPA Method 608	DV-GC-0020 DV-GC-0016
Polychlorinated Biphenyls	SW-846 8082 SW-846 8082A EPA Method 608	DV-GC-0021 DV-GC-0016
Organophosphorus Pesticides	SW-846 8141A, & EPA Method 614	DV-GC-0017
Semi-volatiles by GC/MS	SW-846 8270C SW-846 8270D SW-846 8270E	DV-MS-0011 DV-MS-0012
PAH by GC/MS SIM	SW-846 8270C SW-846 8270D SW-846 8270E	DV-MS-0002

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Title: Concentration and Clean-up of Organic Extracts [SW-846, 3510C, 3540C, 3546, 3550B, 3550C, 3620C, 3630C, 3660B, 3665A, ASTM Method D7065-11, and EPA 600 Series Methods]

Approvals (Signature/Date):

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Facility Distribution No. _____ Distributed To: _____

1.0 Scope and Application

1.1 This standard operating procedure (SOP) provides instructions for the concentration, and if necessary, cleanup, of solvent extracts of organic compounds from water samples, soil samples, TCLP leachates, and SPLP leachates. This SOP is based on SW-846 Methods 3510C, 3540C, 3546, 3550B, 3550C, 3620C, 3630C, 3660B, 3665A, ASTM Method D7065-11, and EPA 600 Series methods.

1.2 The determinative methods and extraction methods used in conjunction with this procedure are listed in Attachment 1.

1.3 This procedure does not include the extraction steps. See the following SOPs for the applicable extraction procedures:

DV-OP-0006: *Extraction of Aqueous Samples by Separatory Funnel*, SW-846 3510C and EPA 600 Series

DV-OP-0010: *Soxhlet Extraction of Solid Samples*, SW-846 3540C

DV-OP-0015: *Microwave Extraction of Solid Samples*, SW-846 3546

DV-OP-0016: *Ultrasonic Extraction of Solid Samples*, SW-846 3550B and 3550C

NOTE: This SOP does not include the concentration steps of extracts for Herbicides by method 8151A or Herbicides by method 8321. See DV-OP-0011 *Extraction of Herbicides* and DV-LC-0014 *Chlorinated Phenoxyacid Herbicides by ESI/LC/MS/MS* respectively.

2.0 Summary of Method

Sample extracts are concentrated to a specific final volume using an S-EVAP, N-EVAP, or Turbo-Vap. Some methods require a solvent exchange. If necessary, various clean-up techniques are performed before the extract is sent for analysis.

3.0 Definitions

Refer to the Glossary of the Eurofins TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P *Quality Control Program* for definitions of general analytical and QA/QC terms.

3.1 **Extraction Holding Time:** The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.2 **Preparation Batch:** A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards.

3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client. See WI-DV-0032 *Guidelines for Setting up Method Comments.*

3.4 **Quality Assurance Summary (QAS):** Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the special instructions/Method Comments field in LIMS. In those situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

4.0 Interferences

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2 Visual interferences or anomalies (such as foaming, emulsions, odor, more than one layer of extract, etc.) must be documented.

4.3 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.4 Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA by 8270D_SIM_LL must be concentrated in glassware designated for that method. K-D flasks, concentrator tubes, stem-less glass funnels, and Snyder columns will be clearly marked and segregated for this purpose.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 In order to limit the emission of methylene chloride, Eurofins TestAmerica Denver uses a solvent recovery system. The system condenses and collects

methylene chloride that has been evaporated off the sample extracts while on the S-EVAP.

5.1.1.1 Each analyst must inspect the system before using it to ensure the collection tubes are in good condition, the in-process tanks are not full, and the chiller is operating correctly.

5.1.1.2 While concentrating methylene chloride or methylene chloride / acetone extracts on the S-Evap, the analyst must check the level of the solvent collected in the in-process tanks at a frequency to ensure the tank will not be overfilled. A tank will not be filled more than 90%. The analyst may use a timer set at 30 minute intervals to help remind the analyst to check the level of the solvent collected in the in-process tanks.

5.1.1.3 The solvent recovery system will never be used for the collection of ether due to the potential danger to analysts if the system were to fail during operation.

5.1.2 Glasswool is a carcinogen and therefore should be handled in a hood to avoid inhalation of dust.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit [1]	Signs and Symptoms of Exposure
Acetonitrile	Flammable Irritant Poison	40 ppm TWA	Exposure may cause cyanide poisoning resulting in reddening of the skin and eyes and pupil dilation. Effects of overexposure are often delayed due to the slow formation of cyanide ions in the body. May cause nose and throat irritation, flushing of the face, tightening of the chest. Also may cause headache, nausea, abdominal pain, convulsions, shock.
Hexane	Flammable Irritant	50 ppm TWA	Causes irritation to eyes, skin and respiratory tract. Aspiration hazard if swallowed. Can enter lungs and cause damage. May cause nervous system effects. Breathing vapors may cause drowsiness and dizziness. Causes redness and pain to the skin and eyes.

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Methanol	Flammable Irritant Poison	200 ppm TWA	Methanol evaporates at room temperature. Inhalation, ingestion and/or eye and skin contact can all possibly cause light-headedness, nausea, headache, and drowsiness. Prolonged exposure can lead to permanent blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Mercury	Corrosive Irritant Highly Toxic	0.05 mg/m^3 TWA	May be fatal if inhaled. May cause respiratory tract irritation. May be harmful if absorbed through skin. May cause skin irritation.
Methylene Chloride	Irritant Carcinogen	25 ppm TWA 125 ppm STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

NOTE 1: All glassware used in this procedure is cleaned following SOP DV-OP-0004 *Glassware Washing of Organic Analysis Applications*. In addition, the glassware is rinsed with methylene chloride immediately prior to use. Rotate glassware; do **not** use specific glassware or positions for the MB and LCS/LCSD.

NOTE 2: Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA method 8270D_SIM_LL and PAHs by method 8270C_SIM_LL must be concentrated in glassware designated for that method. K-D flasks, glass funnels, concentrator tubes, and Snyder columns will be clearly marked and segregated for this purpose.

6.1 All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.2 Kuderna-Danish (K-D) flasks.

6.3 Concentrator tubes for K-D flasks, un-graduated, approximately 10 mL.

6.4 Concentrator tubes for K-D flasks, graduated at 1mL, calibration checked before use following the steps detailed in DV-QA-0008 *Volumetric Verification*.

6.5 Snyder columns, 3-ball with ground glass joints at top and bottom

6.6 Manual, adjustable positive-displacement pipette and bottle-top re-pipettor, used to dispense 1 to 20 mL. Calibration is checked following the steps detailed in DV-QA-0008 *Volumetric Verification*.

6.7 Extract Storage Vials – variety of sizes, clear and amber

6.8 Pasteur pipettes – 6 inch and 9 inch in length.

6.9 Stem-less glass funnels

6.10 Glass wool, baked at 400°C for four hours.

6.11 Boiling Chips – contaminant free, approximately 10/40 mesh Teflon®, PTFE. For concentrating extracts to a final volume greater than 1mL.

6.12 Boiling Chips – contaminant free, carborundum #12 granules, for concentrating extracts to a 1mL final volume. These boiling chips are sufficiently small as to not add any error to the 1mL final volume.

6.13 Solvent Recovery System – includes re-circulating chiller, set no higher than 12°C, cooling condensers, Teflon® PTFE tubing and In-Process Tanks with quick-connect attachments

6.14 S-Evap, thermostat controlled water bath

6.15 N-Evap, thermostat controlled water bath with regulated nitrogen supply

6.16 **Computer Software and Hardware**

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Methylene Chloride

Each lot of solvent is tested following CA-Q-S-001 *Acid and Solvent Lot Testing and Approval Program* or before it is put into use. QA personnel post the list of approved lots at solvent storage areas. If any problems are identified, use of the solvent is suspended until further testing can be done and determines the solvent is acceptable.

7.2 Hexane

For solvents packaged in bottles, each lot of solvent is tested following CA-Q-S-001 *Acid and Solvent Lot Testing and Approval Program* before it is put into use. QA personnel post the list of approved lots at solvent storage areas. If any problems are identified, use of the solvent is suspended until further testing can be done and determines the solvent is acceptable.

7.3 Methanol, HPLC Grade

Each lot of solvent is tested following CA-Q-S-001 *Acid and Solvent Lot Testing and Approval Program* before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.4 Acetone

Each lot of solvent is tested following CA-Q-S-001 *Acid and Solvent Lot Testing and Approval Program* before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.5 Acetonitrile

Each lot of solvent is tested following CA-Q-S-001 *Acid and Solvent Lot Testing and Approval Program* before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.6 Baked Sodium Sulfate, 12-60 mesh

Heat sodium sulfate in a 400°C oven for at least four hours.

7.7 Sulfuric Acid, Concentrated

For use in PCB extract clean-up.

7.8 Florisil Solution, (FlorisilSol)

Add 900mL of hexane to a Class A graduated cylinder. Add 100 mL of Acetone to the same graduated cylinder for a final volume of 1000 mL. Pour the mixture into a 1 L amber bottle.

7.9 Florisil Cartridges,

Purchased ready to use. 1000 mg in 6 mL tube. Stored in a desiccator after opening. Restek part number 24034 or equivalent.

7.10 Anhydrous Silica Gel, 60-100 mesh, (SiGel60-100UA)

Sigma Aldrich part number 23799-1KG or equivalent

7.11 Activated Anhydrous Silica Gel, 60-100 mesh, (Active SilGel)

Bake Silica Gel from Section 7.10 above at 400°C for at least 4 hours. Store in a desiccator.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Sample extracts waiting to be concentrated are stored refrigerated at 0°C - 6°C in glass bottles or flasks and capped with Teflon-lined lids or aluminum foil. Final sample extracts are stored in glass vials with Teflon-lined lids. See Table 3 for details on storage vial types. Final concentrated extracts are stored refrigerated at 0°C - 6°C. Extracts have a holding time of 40 days from the date of extraction to the date of analysis.

8.2 All sample extracts, before or after concentration, are stored separately from standards.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in Eurofins TestAmerica Denver policy DV-QA-003P *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in Eurofins TestAmerica Denver policy DV-QA-024P *QA/QC Requirements for Federal Programs.* This procedure meets all criteria for DoD QSM unless otherwise stated. Any deviation or exceptions from QSM requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 *Non-Conformance and Corrective Action System*. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 12.0 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P *Quality Control Program* for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank for batches of aqueous samples consists of reagent water, and for batches of soil samples, consists of Ottawa sand, both of which are free of any of the analyte(s) of interest. The method blank for batches of TCLP and SPLP leachates consists of leach fluid. The method blank is processed and analyzed just as if it were a field sample.

9.5 Laboratory Control Sample (LCS)

9.5.1 At least one LCS must be processed with each preparation batch. For aqueous sample batches, the LCS consists of reagent water to which the analyte(s) of interest are added at known concentration. For soil sample batches, the LCS consists of Ottawa sand to which the analyte(s) of interest are added at a known concentration. For TCLP and SPLP leachates, the LCS consists of leach fluid to which the analyte(s) of interest are added at known concentration. The LCS is carried through the entire analytical procedure just as if it were a sample.

9.5.2 EPA Methods 608, 610, 614, and 625 require a LCS at a 10% frequency. In other words, one LCS is required for a batch of 10 or less samples. A LCSD is required for a batch of 11 or more samples.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1 One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

9.6.2 EPA Methods 608, 610, 614, and 625 require one matrix spike for every 10 samples. If the batch has more than 10 samples, then two matrix spikes must be performed. The two matrix spikes are to be performed on two different samples.

9.6.3 If insufficient sample volume is available for MS/MSD, an NCM must be written and a LCSD must be prepared.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 *Non-Conformance and Corrective Action System*. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Critical Procedural Considerations

NOTE: Rotate glassware; do **not** use specific glassware or positions for the MB and LCS/LCSD.

10.3.1 As stated throughout this SOP, analysts must review Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009 *Spike/Surrogating and Review Procedure for Organic Extractions*).

10.3.2 Analyst must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces should be cleaned or disposed of before coming into contact with the sample.

10.3.3 According to the type of sample and any cleanup procedures needed, different final solvents and volumes will be required. Refer to WI-DV-0009 *Spike/Surrogating and Review Procedure for Organic Extractions* for the appropriate final solvents and final volumes.

10.4 Refer to WI-DV-0009 *Spike/Surrogating and Review Procedure for Organic Extractions* to determine if the extract is to be concentrated by the Kuderna-Danish / N-Evap method described in Section 10.5 and 10.6, or the Turbo-Vap method described in Section 10.6.6

10.5 Concentration by the Kuderna-Danish Method (S-evap)

10.5.1 Refer to WI-DV-0009 *Spike/Surrogating and Review Procedure for Organic Extractions*. If the extract is to be concentrated to a 1 mL final volume, use a 1 mL graduated concentrator tube. For extracts that are to be concentrated to any other final volume, use an un-graduated concentrator tube.

10.5.2 Assemble the Kuderna-Danish concentrator by attaching the appropriate concentrator tube to the 500 mL K-D flask with a clip. Make sure the attachment is firm at the joint. While wearing cut-resistant gloves, tighten the joint with your fingertips and thumb. Do NOT over-tighten. Refer to Attachment 3 for configuration of the Kuderna-Danish concentrator.

NOTE: Due to the low reporting limits and the potential for contamination, the extracts that are to be analyzed for NDMA by method 8270D_SIM_LL and PAHs by method 8270C_SIM_LL must be concentrated in glassware designated for those methods. K-D flasks, concentrator tubes, and Snyder columns will be clearly marked and segregated for this purpose.

10.5.3 Rinse the apparatus with methylene chloride. Discard the rinse solvent into the appropriate waste container. Care should be taken to ensure all surfaces of the glass are coated with solvent.

10.5.4 If the extract is to be concentrated to a 1 mL final volume, add 2-3 carborundum granules to the K-D concentrator. If the extract is to be concentrated to a final volume greater than 1 mL, add 1-2 Teflon® boiling chips to each K-D concentrator.

10.5.5 If the sample extracts have not been filtered through sodium sulfate at the time of extraction, or if the sample extract have visible water, then the extracts must be dried at this point. Plug a glass funnel with baked glass wool and add approximately 1 tablespoon of baked sodium sulfate. Rinse the funnel and the sodium sulfate with methylene chloride and place it on top of the K-D. During the quantitative transfer in section 10.5.6 the extract will be filtered through the sodium sulfate.

NOTE 1: Glass wool dust is a carcinogen and therefore glass wool should only be handled in a hood to avoid inhaling any glass particles. Once covered with sodium sulfate, it can be removed from the hood.

NOTE 2: If the extract contains more water than can be easily removed by filtering through 1 tablespoon of sodium sulfate, either more sodium sulfate can be used or a solvent-rinsed separatory funnel can be used to separate the water out of the extract. A NCM should be prepared if this is necessary.

10.5.6 Quantitatively transfer the sample extract to the K-D flask. Transfer the sample label to the K-D flask. Perform a quantitative transfer of the extract by rinsing the sample extract container with methylene chloride and adding the rinse solvent to

the K-D. If the extract is being filtered through sodium sulfate, be sure to rinse the sodium sulfate well to ensure no target compounds are left on the sodium sulfate. Allow the solvent to drain from the sodium sulfate into the K-D flask then discard the sodium sulfate.

10.5.7 Turn a three-ball Snyder column upside down and rinse with methylene chloride, then rinse the bottom joint with methylene chloride. Attach the Snyder column to the top of the K-D concentrator as shown in Attachment 3.

10.5.8 Place the K-D concentrator on a s-evap water bath so that the tip of the receiver tube is submerged. The water level should not reach the joint between the concentrator tube and the K-D flask. Refer to WI-DV-0009 *Spike/Surrogating and Review Procedure for Organic Extractions* for the correct water bath temperature. Record both the observed and the corrected temperature on the benchsheet.

10.5.9 For extracts that are methylene chloride or 50/50 methylene chloride/acetone, attach the solvent recovery system tube to the top of the Snyder column. At the appropriate rate of distillation, the balls will actively chatter but the chambers should not flood.

NOTE 1: For extracts for analysis for low-level NDMA by method 8270D_SIM_LL and PAHs by 8270C_SIM_LL, the solvent recovery system will not be used to avoid possible contamination.

NOTE 2: At this time, a timer may be set for 30 minute intervals as a reminder to check the in-process solvent tanks.

10.5.10 If the method does not require a solvent exchange, skip to Section 10.5.12. If the method requires a solvent exchange, continue on to Section 10.5.11.

10.5.11 If the method requires a solvent exchange at this time, detach the solvent recovery system tube from the top of the Snyder column and add the appropriate exchange solvent through the top of the Snyder column. The exchange solvent should be added when the extract has concentrated to a level that it forms a quarter-sized pool of solvent in the bottom of the K-D. Refer to WI-DV-0009 *Spike/Surrogating and Review Procedure for Organic Extractions* for details of exchange solvents and volumes. Mark the K-D flask and sample label to indicate the exchange has been performed. There is no need to re-attach the solvent recovery system at this time as the majority of the methylene chloride has already been evaporated and collected.

10.5.12 Continue to concentrate the sample on the s-evap water bath back down to 10-15 mL, or just below the K-D and concentrator tube joint. At this point the boiling sample is just barely splashing above the top of the receiver tube.

NOTE: It is very important not to concentrate to dryness as analytes will be lost. Some of the analyses, especially for 8270 and 8015, are especially temperature sensitive and the sample should be taken off the water bath as soon as possible to avoid losing analytes. The 8081

surrogate TCMX is fairly volatile and can be lost if the extract is allowed to concentrate too low either before or after hexane exchange. If the analyst has concerns that the extract might have concentrated too low, they should notify their supervisor and/or write a NCM.

10.5.13 Remove the K-D concentrator from the water bath. Rinse the Snyder column down with a minimal amount of solvent. If the extract was exchanged, use the exchange solvent to perform the rinse, otherwise use methylene chloride.

10.5.14 Allow the extract to cool to room temperature, about 10 minutes.

10.5.15 After the extract is allowed to cool, if the level of the extract is above the level of the concentrator tube joint, add a fresh boiling chip and return the K-D concentrator to the water bath.

10.5.16 After the extract is cool, remove the Snyder column. Remove the clip holding the K-D flask and concentrator tube together. Use a Kim-wipe to dry the water off of the joint area so that water does not get into the extract. Remove the concentrator tube from the K-D flask and rinse the lower K-D flask joint into the concentrator tube with methylene chloride or the appropriate exchange solvent.

10.6 Nitrogen Evaporation (N-Evap) to Final Concentration.

10.6.1 N-evap needles should be cleaned weekly by soaking overnight in methylene chloride. This is documented in the N-evap needle log-book.

10.6.2 At the beginning of each shift, the N-evap needles should be wiped clean with a Kim-wipe soaked in methylene chloride to remove any potential contamination. If a needle comes in contact with an extract, then it needs to be cleaned before being used on the next extract.

10.6.3 Place the concentrator tube on the nitrogen evaporator. The temperature of the water bath should be at least 5°C below the boiling temperature of the solvent being evaporated (See Attachment 2). Lower the needle down to the sample so that a small dimple forms on the surface of the solvent. The stream of nitrogen should be gentle enough that it does not cause the extract to splash. Record both the observed and the actual temperature on the benchsheet.

10.6.4 During the course of the evaporation, rinse the sides of the concentrator tube with approximately 1 mL of clean solvent. The rinse should occur when the solvent gets close to the final volume. Concentrate the solvent to just below the final volume and remove from the nitrogen evaporator.

10.6.5 Transfer the extract into the appropriate vial. Refer to WI-DV-0009 *Spike/Surrogating and Review Procedure for Organic Extractions* for the appropriate final volume and correct vial.

10.6.5.1 If the extracts are to have a final volume of 1 mL, they should be in 1 mL graduated concentrator tubes. Using a Pasteur pipette, or a solvent wash bottle, add the appropriate solvent to the tube until the

extract meniscus reaches the 1 mL gradation, rinsing the sides of the concentrator tube 3 times once 1mL volume has been reached. Then using the Pasteur pipette transfer the extract to a labeled 2 mL amber glass vial.

10.6.5.2 For extracts with a final volume greater than 1mL, the vials should be calibrated using the manual, adjustable positive-displacement pipette or bottle-top re-pipettor. Document the pipette ID used on the batch record. Pipette the correct volume of clean solvent into the vial and mark the bottom of the meniscus with a thin marker. Discard the solvent. Transfer the extract into the vial using a Pasteur pipette and rinse the concentrator tube with solvent. Transfer the rinse to the vial. Bring the meniscus of the solvent up to the marked line. Cap with a Teflon-lined cap.

NOTE 1: The final concentration and volume measurement steps are critical. Use care when concentrating and make certain that the final volume measurement is accurate.

NOTE 2: Some extracts might not concentrate down to the required final volume. If the extract is very dark and viscous, or an oil layer or precipitate starts to form, a higher final volume can be used. This should be documented in an NCM.

10.6.6 After the extract has been transferred to the appropriate vial, rinse the concentrator tube with methylene chloride before washing per DV-OP-0004 *Glassware Washing of Organic Analysis Applications*. This is important to remove any residual contamination.

10.7 TurboVap Method

10.7.1 Turn on the TurboVap and adjust the water temperature to 40°C. Turn the nitrogen supply on. Record both the observed and the actual temperature on the benchsheet.

10.7.2 Switch the endpoint sensor to “Manual”.

10.7.3 Adjust the water bath level. The water level should be at least 1 inch above the extract level.

10.7.4 Turn on the nitrogen gas and adjust the gas pressure to approximately 12 psi. Lower pressure may be used if needed to prevent samples from splashing out of the TurboVap tubes.

10.7.5 Rinse the TurboVap tube with methylene chloride or the solvent the extract is in. Discard the waste.

10.7.6 Transfer the sample to the TurboVap tube. For 8141 soils extracted by soxhlet, dry the extract first by filtering through a funnel with baked sodium sulfate. Rinse the sample extract container with clean solvent and transfer to the TurboVap

tube. Do not fill the TurboVap tubes over the fill line or approximately ¾ full.

10.7.7 Place the TurboVap tube into the TurboVap and turn on nitrogen to the position the tube is in.

10.7.8 Close the lid. You should be able to see the sample extracts swirling in the tubes.

NOTE: **If the extract splashes when the nitrogen flow starts, transfer a portion of the extract back into the original extract container, or lower the gas pressure.**

10.7.9 As the extract concentrates, transfer the remainder of the extract in to the appropriate Turbovap tube. Rinse the sample container with a few milliliters of methylene chloride or appropriate solvent and transfer to the Turbovap tube.

10.7.10 During the concentration rinse the Turbovap tube walls with a few milliliters of solvent 1 or 2 times.

10.7.11 If a solvent exchange is required, concentrate to about 5 mL and add the exchange solvent. After the exchange solvent is added, swirl the extract to make sure the extract is well mixed. Concentrate back down to slightly less than the appropriate volume. Refer to Attachment 3 for details of exchange solvents and final volumes.

10.7.12 Transfer the extract into the appropriate vial.

10.7.12.1 Currently, the TurboVap is only used to concentrate extracts with final volumes greater than 1 mL. Ask the supervisor for guidance if a project requires a 1 mL final volume by TurboVap.

10.7.12.2 For extracts with a final volume greater than 1 mL, the vials should be calibrated using the manual, adjustable pipette or bottle-top re-pipettor. Document the pipette ID used on the batch record. Pipette the correct volume of clean solvent into the vial and mark the bottom of the meniscus with a thin marker. Discard the solvent. Transfer the extract to the vial using a Pasteur pipette and rinse the concentrator tube with solvent. Transfer the rinse to the vial. Bring the meniscus of the solvent up to the marked line. Cap with a Teflon-lined cap.

10.7.12.3 Rinse the Turbovap tube with methylene chloride 2-3 times before washing. Turbovap tubes are not baked. They are cleaned in accordance with DV-OP-0004 *Glassware Washing of Organic Analysis Applications*. If the Turbovap tubes need to be used again before they are dry, rinse with acetone to dry the Turbovap tube.

10.8 Cleanup Techniques

NOTE: If any sample in a batch requires a clean-up, the batch QC must also undergo the same clean-up technique.

10.8.1 Florisil Cartridge Cleanup

Florisil can be used to remove low-medium molecular weight polar hydrocarbon interfering compounds from pesticide extracts. The laboratory will use Florisil cleanups whenever water extracts have any color, whenever soil extracts have any color darker than a Post-It® Note, or whenever there is clear evidence of interferences, such as significant interfering peaks in the RT range for the target pesticide compounds or failing sample surrogate recoveries. Extracts that are to be analyzed for kepone will not be florisil cleaned, because florisil will remove kepone from the extract.

NOTE: Florisil cartridge performance checks are conducted for every lot of Florisil before use. Add 1.0 mL of the Florisil check solution described in Attachment 4 to a pre-rinsed Florisil cartridge. Following the procedure described below, load and elute the 1mL of check solution through the Florisil cartridge. Bring the final volume back down to 1.0 mL in hexane. The test sample must show 80-115 % recovery of the controlled analytes with < 5% trichlorophenol recovery, and no peaks interfering with target compounds can be detected. The non-controlled analytes will be monitored for problems, but do not have to pass the 80-115% limits. If the check fails, repeat the test. If the re-check fails, contact QA for guidance.

10.8.1.1 Clean the manifold and ports

Prior to each use, the top and underside of the manifold lid must be wiped down with hexane and a Kim-wipe to prevent any cross-contamination. The manifold ports must be left open and placed in a jar with fresh acetonitrile, in a sonication bath for a minimum of 30 minutes. The jar used in the soak and sonication of the ports must be replaced weekly to ensure it does not spread contamination. This is documented in the Organic Extraction Weekly Cleaning Logbook.

10.8.1.2 Place one Florisil cartridge into the vacuum manifold for each extract. Make sure all valves are closed.

10.8.1.3 Add approximately 6 mL of hexane to each cartridge by filling the tube.

10.8.1.4 Slowly open the valves to allow a few drops of hexane to pass through, then close the valve and allow the hexane to soak the cartridge for at least 5 minutes.

10.8.1.5 Slowly open the valves again and allow the hexane to drain through the cartridge but close the valve when the solvent level is right above the glass frit. Do not allow the cartridges to go dry. If cartridges go dry, repeat the conditioning step.

10.8.1.6 Remove the manifold top and place one clean, labeled 16 x 125 mm disposable glass test tube in each position for each of the samples. Replace the manifold top. Make sure that the solvent line from each

cartridge is placed inside the appropriate tube.

10.8.1.7 Add exactly 2.0 mL of the concentrated extract to the appropriate Florisil cartridge. Turn the valve to the on position.

10.8.1.8 Allow the extract to gravity drip through the cartridge. The flow through the cartridges should be drop-wise, not streaming.

10.8.1.9 Just before the extract level drops below the glass frit, fill the cartridge with (90:10) Florisil solution. Allow this to pass through the cartridge, then just before it falls below the glass frit again, fill the cartridge again with (90:10) Florisil solution.

10.8.1.10 Allow all of the 90:10 solution to gravity drip through the cartridges.

10.8.1.11 After visible solvent has been allowed to gravity drip through the cartridge, apply the vacuum to pull remaining solvent through cartridge, typically no more than 5 seconds.

NOTE: Do not use the vacuum to recover solvent from the cartridge before gravity drip is complete. Doing so could result in the interfering compounds that should be retained in the packing to come through into the cleaned extract.

10.8.1.12 Remove the tubes from the vacuum manifold and concentrate them back down to just below 2.0 mL on the nitrogen evaporator. Quantitatively transfer the extract to a 4mL vial that has been calibrated to hold 2.0 mL and bring the extracts up to the 2.0 mL calibration mark with hexane.

10.8.1.13 Discard the used cartridges.

10.8.2 Sulfur Removal

NOTE: This step is typically performed by the instrument analyst, as it is performed after extracts are concentrated to final volume.

Sulfur can be removed by one of three methods: mercury, copper, or tetrabutylammonium sulfite (TBA), according to laboratory preference. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and carefully draw off the sample extract with a disposable pipette, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean concentrator tube before proceeding with further sulfur cleanup.

10.8.2.1 Sulfur Removal with Elemental Mercury

NOTE: Use Mercury in a hood and sparingly in order to minimize exposure and disposal costs.

10.8.2.1.1 Transfer approximately 2 mL of sample extract into a clean

Teflon-sealed vial.

10.8.2.1.2 Add one to three drops of mercury to the extract vial and seal.

10.8.2.1.3 Shake well for 15-30 seconds. If prolonged shaking is required, use a mechanical shaker.

10.8.2.1.4 Remove the extract from the mercury using a disposable pipette and transfer to a clean vial.

10.8.2.1.5 If the mercury turns black, sulfur was present. Decant or pipette off the extract to a clean vial and repeat the procedure by adding one to three drops of fresh mercury. Do this until the mercury does not turn black.

10.8.2.1.6 If the extract is cloudy, filter the extract through a 1um disposable syringe filter.

10.8.2.1.7 Properly dispose of the mercury waste.

10.8.2.2 Sulfur Removal with Copper Powder

NOTE: This technique requires the copper powder to be very reactive, as demonstrated by a bright and shiny appearance. A pre-cleaned, activated copper may be purchased from a valid vendor. If manual preparation of reactive copper is performed, take care to remove all traces of acid in order to prevent degradation of some analytes.

10.8.2.2.1 Weigh out copper into a 20 mL VOA VIAL assuming two grams of copper needed per sample.

10.8.2.2.2 Remove oxides by treating with 10% nitric acid.

10.8.2.2.3 Rinse the copper with DI organic-free water three times to remove all traces of acid.

10.8.2.2.4 Rinse the copper with acetone and dry under a stream of nitrogen.

10.8.2.2.5 Add approximately 2 grams of the copper powder to a 2 mL vial with approximately 1ml of sample extract and shake vigorously on a mechanical shaker for at least one minute.

10.8.2.2.6 After phase separate, draw off extract and transfer to a clean vial.

10.8.3 Sulfuric Acid Cleanup

NOTE: This step is typically performed by the instrument analyst, as it is performed after extracts are concentrated to final volume.

10.8.3.1 Add 1 mL of concentrated sulfuric acid to approximately 2 mL of sample extract in a Teflon capped vial.

CAUTION: There must be no water or acetone present in the extract or the reaction may shatter the sample container.

10.8.3.2 Vortex for about 5 seconds and allow to settle. (Centrifuge if necessary)

10.8.3.3 Remove the sample extract (top layer) from the acid using a Pasteur pipette and transfer to a clean vial.

CAUTION: It is not necessary to remove all the extract since the final volume is already determined. Transferring any amount of sulfuric acid along with the extract will result in extremely rapid degradation of the chromatographic column

10.8.3.4 If the sulfuric acid layer becomes highly colored after shaking with the sample extract, transfer the hexane extract to a clean vial and repeat the cleanup procedure until color is no longer being removed by the acid, or a maximum of 5 acid cleanups.

10.8.3.5 Properly dispose of the acid waste.

10.8.4 Silica Gel Clean-up for DRO extracts

10.8.4.1 Samples requiring silica gel clean-up for the Oklahoma DRO method should follow the procedure in section 10.8.4.1, which includes the addition of a reverse surrogate. Samples requesting silica gel clean-up for other DRO methods should follow the procedure in section 10.8.4.1.2, which does not include an additional surrogate.

10.8.4.1.1 If the sample is logged for method 3630C_M, concentrate the extract and all associated QC to slightly below 1 mL on the N-Evap. Add 100uL of the “SilicaGelSurr” standard to each extract and then bring the extracts to a 1 mL final volume with methylene chloride. Transfer to the appropriate final extract vial per section 10.6. Proceed to section 10.8.4.2.

10.8.4.1.2 If the sample is not logged for method 3630C_M but silica gel clean-up is still requested, no further surrogate is added. Concentrate to 1 mL final volume normally per section 10.6. Proceed to section 10.8.4.2.

NOTE: Please note that some projects require analysis of extract that has been silica gel cleaned as well as analysis of extract that has not been cleaned. Due to the limited final volume of the extract, samples requiring analysis of both cleaned and un-cleaned extract must be extracted twice, and in separate batches with separate QC.

10.8.4.2 Add approximately 0.05 g of activated silica gel to the extract, cap, and vortex for approximately 15 seconds. Allow the silica gel to settle.

10.8.4.3 Transfer the extract to a new vial, leaving the silica gel behind. Submit for analysis.

10.9 Documentation

All observations are recorded either directly into LIMS or on the hard-copy benchsheets. Any hand-written data recorded on the hard-copy benchsheets are transferred into LIMS before extracts are delivered to the analytical group. The hard-copy benchsheets are then saved and scanned into pdf files and sent to QA for archiving.

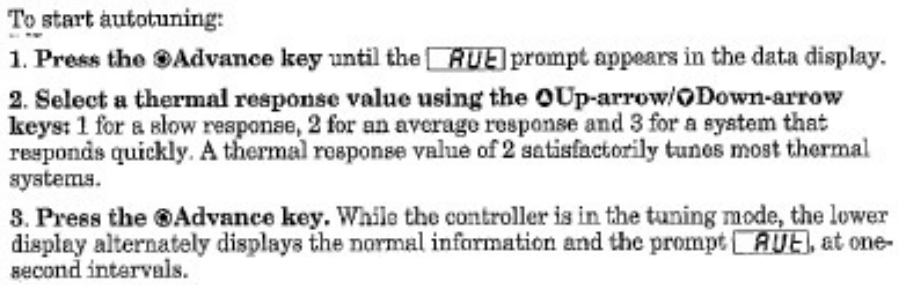
10.10 Maintenance

10.10.1 The chiller that operates the solvent recovery system should be checked periodically to ensure the water level is sufficient.

10.10.2 The SPE ports and valves used in the florisil are open and placed in a jar with fresh acetonitrile, in a sonication bath for a minimum of 30 minutes. The jar used in the soak and sonication of the ports must be replaced weekly to mitigate the risk of contamination. This is documented in the Organic Extraction Weekly Cleaning Logbook.

10.10.3 The N-Evap needles are removed once a week and soaked overnight in a jar of methylene chloride. This is documented in the Organic Extraction Weekly Cleaning Logbook.

10.10.4 The water bath used in the concentration of extracts has a thermostat that occasionally needs auto-tuned to keep the bath temperature within a narrow range. Record both the observed and the actual temperature on the benchsheet.



To start autotuning:

1. **Press the ◉Advance key** until the [AUE] prompt appears in the data display.

2. **Select a thermal response value using the ▲Up-arrow/▼Down-arrow keys:** 1 for a slow response, 2 for an average response and 3 for a system that responds quickly. A thermal response value of 2 satisfactorily tunes most thermal systems.

3. **Press the ◉Advance key.** While the controller is in the tuning mode, the lower display alternately displays the normal information and the prompt [AUE], at one-second intervals.

10.11 Troubleshooting

Unusual sample matrix may cause problems. If the extracts do not behave normally, contact a supervisor or senior analyst if you are unsure how to proceed. Document all observations and anomalies in a NCM.

11.0 Calibration

Not applicable to this procedure. See the determinative methods for calibration of the analytical instrumentation.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-S-006 *Detection and Quantitation Limits*. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024 *Training*.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024 *Training*.

13.0 Pollution Control

It is Eurofins TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health & Safety Manual, and DV-HS-001P *Waste Management Plan*.

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Methylene chloride – Waste Stream B

14.2.2 Flammable Solvents – Waste Stream C

14.2.3 1:1 MeCl2:Acetone – Waste Stream CA

14.2.4 Solid waste/sodium sulfate – Waste Stream D

14.3 Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of these materials.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, January 2005.

15.1.1 Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

15.1.2 Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

15.1.3 Method 3550C, Ultrasonic Extraction, Revision 3, February 2007.

15.1.4 Method 3540C, Soxhlet Extraction, Revision 3, December 1996.

15.1.5 Method 3546, Microwave Extraction, Revision 0, February 2006.

15.1.6 Method 3620C, Florisil Cleanup, Revision 3, February 2007.

15.1.7 Method 3660B, Sulfur Cleanup, Revision 2, December 1996.

15.1.8 Method 3660A, Sulfur Cleanup, Revision 1, July 1992.

15.1.9 Method 3665A, Sulfuric Acid/Permagante Cleanup, Revision 1, December 1996.

15.1.10 Method 3630C, Silica Gel Cleanup, Revision 3, December 1996.

15.2 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater

15.2.1 Method 608, Organochlorine Pesticides and PCBs.

15.2.2 Method 610, Polynuclear Aromatic Hydrocarbons.

15.2.3 Method 614, The Determination of Organophosphorus Pesticides in Municipal and Industrial Wastewater

15.2.4 Method 625, Base/Neutrals and Acids.

15.3 ASTM D7065-11, Standard Test Method for Determination of Nonylphenols, Bisphenol A, p-tert-Octylphenol, Nonylphenol Monoethoxylate, and Nonylphenol Diethoxylate in Environmental Waters by Gas Chromatography Mass SpectrometryMethod Modifications:

16.0 Modifications

16.1 Method SW-846 3665A calls for the clean-up to be performed using 1:1 Sulfuric Acid:H_2O. This procedure calls for the clean-up to be performed using concentrated sulfuric acid.

16.2 ASTM D7065-11 calls for the samples to be concentrated to a 0.5 mL final volume. This procedure calls for a 1 mL final volume.

16.3 Method SW-846 3620C calls for the florisil lot check to be performed using a standard containing the some pesticides at various concentrations from 5 ug/L to 50 ug/L. Per the source method, 1 mL of the standard is diluted to 2 mL (for concentrations between 2.5 ug/L and 25 ug/L) and the cleanup is then carried out and the cleaned extract concentrated to 1 mL for a final concentration of 5 ug/L to 50 ug/L. This procedure calls

for the lot check to be performed using a standard containing all the pesticides at the same concentration of 50 ug/L. 1 mL of this standard is cleaned up without prior dilution and then concentrated back down to 1 mL.

16.4 Method SW-846 3620C states that the florisil lot check passes if the pesticide recoveries are between 80% and 110% recovery. This procedure says the lot check passes if the pesticide recoveries are between 80% and 115%. This is done to match the CCV control limits.

16.5 Method SW-846 3620C states that the florisil lot check is to be performed using a standard containing the 2,4,5-Trichlorophenol at 0.1 ug/L. Per the source method, 0.5 mL of this standard is diluted to 2 mL (for a concentration of 0.025 ug/L) and the cleanup is then carried out and the cleaned extract concentrated to 1 mL for a concentration of 0.05 ug/L. This procedure calls for the lot check to be performed using a standard containing 2,4,5-trichlorophenol at 100 ug/L. 1 mL of this standard is cleaned up without prior dilution and then concentrated back down to 1 mL.

16.6 Method SW-846 3620C Section 11.1.3 states to condition the florisil cartridge with 4 mL of hexane. This procedure calls for 5 mL of hexane to be used. This is done for convenience.

16.7 Method SW-846 3630C calls for the silica gel clean-up to be performed with a column or SPE cartridge. This procedure calls for the silica gel to be added directly to the extract and mixed. The reverse surrogate used indicates if the clean-up is effective.

17.0 Attachments

Attachment 1: Determinative and Extraction Methods Used in Conjunction with this SOP.
Attachment 2: Boiling Points of Solvents
Attachment 3: Kuderna-Danish Concentrator
Attachment 4: Florisil Check Solution

18.0 Revision History

This section has been added beginning with Revision 0. Only details of the last two revisions are incorporated into this SOP. Prior revisions are documented in the QA files and available upon request.

- Revision 16, dated 23 December 2021
  - Removed all mention of Continuous Liquid-Liquid Extraction (CLLE) due to this extraction method being discontinued.
- Revision 15, dated 19 May 2021
  - Annual Review
  - Updated copyright information
  - Changed TestAmerica to Eurofins TestAmerica throughout
  - Changed section 10.5.5 to use one tablespoon of sodium sulfate instead of 1 teaspoon.
  - Added to section 10.6.5.1 to rinsing the sides of the concentrator tube 3 times once 1mL volume has been reached.
  - Updated language and formatting throughout

Attachment 1.

Determinative and Extraction Methods Used in Conjunction with this SOP

Method Description	Determinative Method	Determinative Method SOP	Extraction Method	Extraction Method SOP
Diesel Range Organics & Jet Fuels	SW-846 8015B, 8015C, 8015D, California LUFT Method, & AK102 & AK103, NW-TPH, OK DRO	DV-GC-0027	WATER: SW-846 3510C, AK102 AK103 NW-TPH OK DRO SOIL: SW-846 3550B/C SW-846 3546 AK102, AK103 NW-TPH OK DRO	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Chlorinated Pesticides	SW-846 8081A, 8081B & EPA Method 608	DV-GC-0020 DV-GC-0016	WATER: SW-846 3510C SOIL: SW-846 3550B/C SW-846 3546	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Polychlorinated Biphenyls	SW-846 8082, 8082A EPA Method 608	DV-GC-0021 DV-GC-0016	WATER: SW-846 3510C SOIL: SW-846 3550B/C SW-846 3546	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Organo-phosphorus Pesticides	SW-846 8141A, 8141B, & EPA Method 614	DV-GC-0017	WATER: SW-846 3510C SOIL: SW-846 3540C	WATER: DV-OP-0006 SOIL: DV-OP-0010
Polynuclear Aromatic Hydrocarbons	SW-846 8310 & EPA Method 610	DV-LC-0009	WATER: SW-846 3510C SOIL: SW-846 3550B/C	WATER: DV-OP-0006 SOIL: DV-OP-0016
Semi-volatiles by GC/MS	SW-846 8270C, 8270D & EPA 625	DV-MS-0011 DV-MS-0012	WATER: SW-846 3510C SOIL: SW-846 3550B/C	WATER: DV-OP-0006 or SOIL: DV-OP-0016
Low-Level Semi-Volatiles by GC/MS	SW-846 8270C	DV-MS-0011	WATER: SW-846 3510C	WATER: DV-OP-0006
Polynuclear Aromatic Hydrocarbons by GC/MS SIM	SW-846 8270C SIM	DV-MS-0002	WATER: SW-846 3510C SOIL: SW-846 3550B/C SW-846 3546	WATER: DV-OP-0006 SOIL: DV-OP-0016 or DV-OP-0015
Isotope Dilution Analysis of n-Nitrosodimethylamine by GCMS SIM using LVI	SOP	DV-MS-0015	WATER: SW-846 3510C SOIL: SW-846 3550B/C	WATER: DV-OP-0021 SOIL: DV-OP-0016

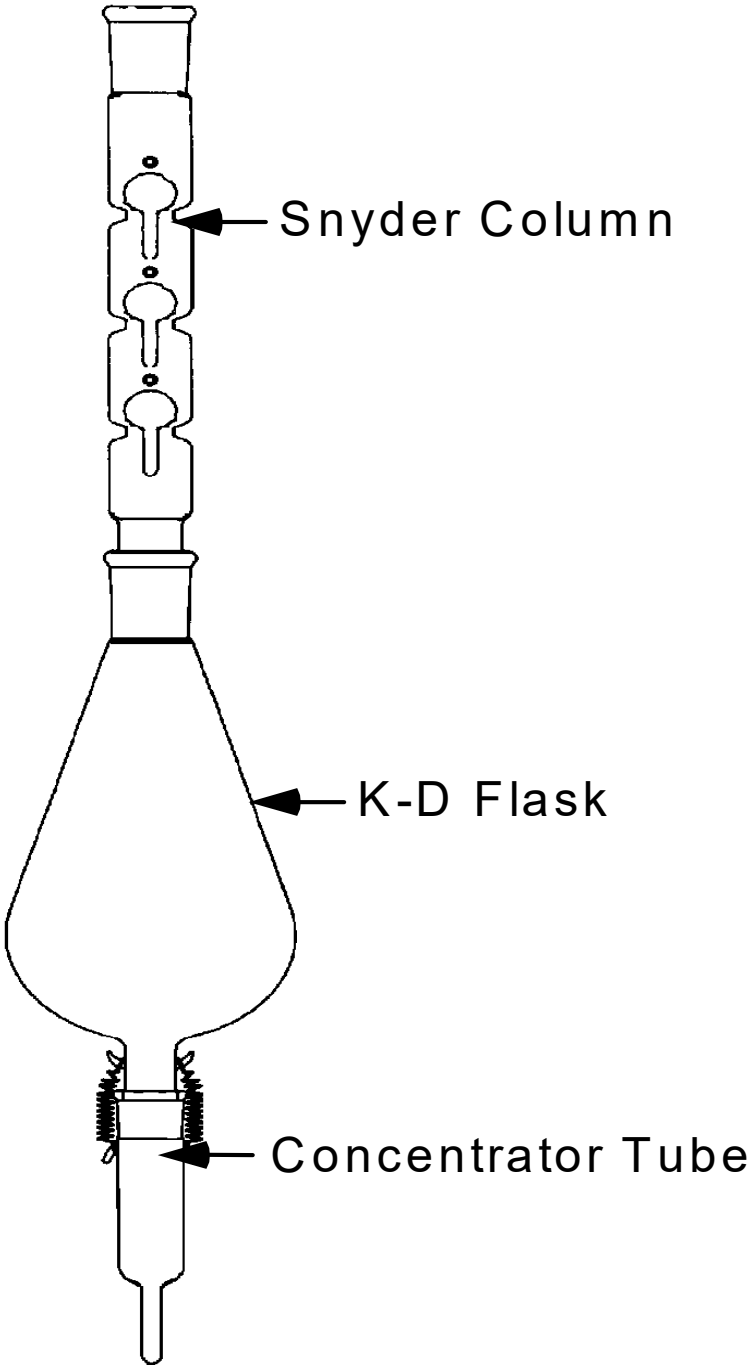
Attachment 2.

Boiling Points of Solvents

Solvent	Boiling Point (°C)
Methylene chloride	40
Acetone	56
Hexane	69
Methanol	65
Acetonitrile	82

Attachment 3.

Kuderna-Danish Concentrator



Attachment 4.

Florisil Check Solution
Prepared in Hexane

Compound	Concentration	Control
2,4,5-Trichlorophenol	0.05ug/mL	Y
Alpha-BHC	0.05ug/mL	Y
Alpha-Chlordane	0.05ug/mL	N
Aldrin	0.05ug/mL	N
Beta-BHC	0.05ug/mL	N
Dieldrin	0.05ug/mL	Y
Endosulfan I	0.05ug/mL	Y
Endosulfan II	0.05ug/mL	N
Endosulfan sulfate	0.05ug/mL	N
Endrin	0.05ug/mL	Y
Endrin Aldehyde	0.05ug/mL	N
Endrin Ketone	0.05ug/mL	N
Gamma-BHC	0.05ug/mL	Y
Gamma-Chlordane	0.05ug/mL	N
Heptachlor	0.05ug/mL	Y
Heptachlor expoxide	0.05ug/mL	N
Methoxychlor	0.05ug/mL	Y
4,4-DDD	0.05ug/mL	Y
4,4-DDE	0.05ug/mL	N
4,4-DDT	0.05ug/mL	Y
Tetrachloro-m-xylene	0.02ug/mL	Y
Decachlorobiphenyl	0.02ug/mL	Y

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Electronic Copy Only

Title: Ultrasonic Extraction of Solid Samples [SW-846 3550B & 3550C]

Approvals (Signature/Date):

Heather Fiedler — 6/11/21	Reed Pottruff — 6/11/2021
Technical Specialist — Date	Health & Safety Manager / Coordinator — Date
Maria Fayard — 6/11/21	Scott Hall — 6/11/21
Quality Assurance Officer — Date	Laboratory Director — Date

Facility Distribution No. _____ **Distributed To: _____**

1.0 Scope and Application

1.1 This SOP is applicable to the solvent extraction of organic compounds from solid samples, including wipes, using sonication (i.e., ultrasonic extraction). This SOP is based on SW-846 Method 3550B and 3550C.

1.2 The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate spiking mixtures and extraction solvents are used.

1.3 This procedure does not include the concentration and cleanup steps. See SOP DV-OP-0007 Concentration and Clean-up of Organic Extracts for those details.

2.0 Summary of Method

A measured weight of sample, typically 30 g, is mixed with anhydrous sodium sulfate to form a free flowing powder. This mixture is solvent extracted three times using an ultrasonic horn.

3.0 Definitions

Refer to the Glossary of the Eurofins TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P Quality Control Program for definitions of general analytical and QA/QC terms.

3.1 **Extraction Holding Time**: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.2 **Preparation Batch**: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards

3.3 **Method Comments:** The Method Comments are used to communicate to the bench level chemists special requirements and instructions from the client.

3.4 **Quality Assurance Summary (QAS)**: Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the Method Comments field in LIMS. In these situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally, QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.5 **Aliquot**: A part that is a definite fraction of a whole; as in “take an aliquot of a sample for testing or analysis.” In the context of this SOP, “aliquot” is also used as a verb, meaning to take all or part of a sample for preparation, extraction, and/or analysis.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 In order to extract especially wet solids, the initial sample weight might have to be reduced in order to achieve a free-flowing mixture with the sodium sulfate. This can raise the reporting limits and method detection limits.

4.3 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section of this SOP (Section 9). Specific selection of reagents may be required to avoid introduction of contaminants.

4.4 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.5 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.6 There are many sources of phthalate contamination in the laboratory. The most common of which are nitrile gloves. The analyst should never touch the inside of glassware with gloves. For the analysis of low-level phthalates by method 8270C SIM, common filter paper can introduce phthalate contamination. Therefore when samples are extracted for this analysis, the Method Comments will instruct the analyst that only glass wool can be used.

4.7 It has been observed that 8270 compounds benzoic acid, 2,4-dinitrophenol, and 4,6-dinitro-2-methylphenol will not recover well if the extract does not drain completely and quickly through the sodium sulfate. Therefore it is very important that a thorough rinse is performed – especially after the 1st sonication. Recoveries will also be improved if the filter paper and funnels used allow for quick drainage. It has been observed that Büchner funnels and glass fiber filter paper will slow drainage.

4.8 It has been observed that 8270 compound Benzidine will not recover well if the filter paper and sodium sulfate are not sufficiently rinsed. Therefore it is very important that a thorough rinse is performed – especially after the 1st sonication.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the

assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Ultrasonic disrupters can produce high intensity noise and must be used in an area with adequate noise protection. During operation, the horns will be kept in a sound enclosure inside the fume hood to protect the analyst. If a sound enclosure is not used, then hearing protection is required when within 10 feet of an operating ultrasonic disrupter and the analyst must be in the Hearing Protection Program per DV-HS-0010 Hearing Conservation Program.

5.1.2 Eye protection that satisfies ANSI Z87.1 (as described in the Environmental Health and Safety Manual), laboratory coat, and appropriate gloves must be worn while performing this procedure. Nitrile gloves shall be worn when handling solvents; latex gloves may be worn when handling samples only; and cut resistant gloves shall be worn when washing glassware.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant Poison	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hexane	Flammable	50 ppm (TWA)	Prolonged or repeated contact with skin can cause defatting and dermatitis. Contact with eyes can cause redness, tearing, and blurred vision. Exposure can cause lung irritation, chest pain, and edema, which may be fatal.
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1 Sonicator, at least 300 watts.

6.2 Sonicator horn, ¾ inch

6.3 Balance, >1400-g capacity, accurate to ± 0.1 g, calibrated daily per SOP DV-QA-0014 Selecting and Using Balances.

6.4 Beakers, 400 mL.

6.5 Media bottles, 250 mL.

6.6 Stainless steel conical funnels

6.7 Ashless cellulose filter paper

6.8 Glass wool - For the analysis of low-level phthalates by method 8270 SIM.

6.9 Pipetter with disposable 1.0-mL tips, calibrated daily per SOP DV-QA-0008 Volumetric Verification.

6.10 Aluminum foil.

6.11 Wooden tongue depressors

6.12 Metal spatulas.

6.13 Solvent dispenser pump.

6.14 Filter flask.

6.15 Vacuum pump.

6.16 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1.1 Methylene chloride – Each lot of solvent is tested following SOP CA-Q-S-001 Acid and Solvent Lot Testing and Approval Program before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.1.2 Acetone - Each lot of solvent is tested following SOP CA-Q-S-001 Acid and Solvent Lot Testing and Approval Program before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.1.3 Hexane - Each lot of solvent is tested following SOP CA-Q-S-001 Acid and Solvent Lot Testing and Approval Program before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.1.4 Baked Sodium Sulfate, 12-60 mesh - QA personnel post the list of approved lots at solvent storage areas. Heat sodium sulfate in a 400°C oven for at least four hours. Cool, covered tightly with foil, and store in tightly closed jars.

7.1.5 Baked Ottawa Sand – Heat Ottawa sand in a 400°C oven for at least four hours.

7.2 Standards

7.2.1 Please reference SOP DV-OP-0020 Preparation, Verification, and Storage of Organic Prep Surrogate and Spiked Standards for information regarding the surrogate and spike standards used in this procedure.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils for Method 8082A	Glass with Teflon-lined lids	30 grams	Cool, ≤ 6°C	None	SW-846
Wipes for Method 8082A	Glass with Teflon-lined lids	N/A	Cool, ≤ 6°C	None	SW-846
Soils for all other Methods, including 8082	Glass with Teflon-lined lids	30 grams	Cool, ≤ 6°C	14 days	SW-846
Wipes for all other Methods, including 8082	Glass with Teflon-lined lids	N/A	Cool, ≤ 6°C	14 days	SW-846

[1] Exclusive of analysis. Some regulatory agencies do not accept SW-846 Revision 4 of Chapter 4 and will require the 14 day holding time for both Methods 8082. The states of Alabama, California, Colorado, Connecticut, Nevada, New Jersey, Pennsylvania, and Rhode Island require the 14 day holding time for method 8082.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in Eurofins TestAmerica Denver policy DV-QA-003P Quality Control Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in Eurofins TestAmerica Denver policy DV-QA-024P QA/QC Requirements for Federal Programs. This procedure meets all criteria for DoD QSM unless otherwise stated. Any deviation or exceptions from QSM requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 Non-Conformance and Corrective Action System. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same

procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P Quality Control Program for further details.

9.4 Method Blank (MB)

9.4.1 One method blank must be processed with each preparation batch.

9.4.2 The method blank for batches of soil samples consists of 30 grams of baked Ottawa sand, which is free of any of the analyte(s) of interest.

9.4.3 Eurofins TestAmerica Denver typically provides clients with clean filter paper or sterile gauze to use as wipes. In these cases, the laboratory prepares wipe-matrix MBs by spiking clean filter paper or gauze (of the same type that is provided to the client) with the surrogate compounds to be used for analysis. If the client uses a different type of material for the wipes, the client should provide a clean specimen of that material to be used for the MB. If the client does not provide a blank wipe in this case, the laboratory will prepare the MBs from filter paper or gauze, from the laboratory's inventory, spiked with the surrogate compounds.

9.5 Laboratory Control Sample (LCS)

9.5.1 At least one LCS must be processed with each preparation batch. Some projects require two LCSs (LCS and LCSD) in every batch, therefore it is important to check special project instructions for each sample. Specifically, Alaska Methods AK102 and AK103 require an LCS and LCSD.

9.5.2 For soil sample batches, the LCS consists of 30 g of reagent sand to which the analyte(s) of interest are added at a known concentration.

9.5.3 LCSs for wipe-matrix samples are prepared by spiking the compounds of interest and surrogate compounds onto a piece of clean filter paper or sterile gauze. If the client uses a different type of material for the wipes, the client should provide blank wipe material to the laboratory for use in preparing the LCS. If the client does not provide blank wipe material, the laboratory will prepare LCS using clean filter paper or sterile gauze, from the laboratory's inventory, spiked with the compounds of interest and surrogate compounds.

9.5.4 The LCS is carried through the entire analytical procedure just as if it were a sample.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.6.1 One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate

(MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis. MS/MSDs are not performed on wipe samples.

9.6.2 If insufficient sample volume is available for MS/MSD, an NCM must be written. For SW-846 methods a LCS/LCSD will be required in this case with the exception of work done under the AFCEE program which allows precision to be calculated using LCSs from different batches over the duration of the project.

9.6.3 DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided a LCSD must be prepared.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e. method blank, LCS, LCSD, MS, and MSD) is spiked with surrogate compounds.

9.8 Sample Duplicates

A sample duplicate is a second aliquot of an environmental sample that is processed with the first aliquot of that sample. Sample duplicates are processed as independent samples within the same batch. The sample and duplicate results are compared to determine the effect of the sample matrix on the precision of the analytical process. As with the MS/MSD results, the sample duplicate precision results are not necessarily representative of the precision for other samples in the batch. Sample duplicates are performed when requested by the client. Sample duplicates do not count towards the 20 sample batch limit.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031 Non-Conformance and Corrective Action System. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Specific glassware or equipment positions for the MB and LCS/LCSD are not to be used.

10.4 Critical Procedural Considerations

10.4.1 As stated throughout this SOP, analysts must review the Method Comments and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see WI-DV-0009 Spike/Surrogating and Review Procedure for Organic Extractions).

10.4.2 Analysts must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any other beaker or media bottle than the designated one should be cleaned or disposed of before coming into contact with the sample. Gloves should never come into contact with the inside of beakers, media jars, or steel funnels.

10.4.3 Sodium sulfate should be kept in closed containers when not in use. It is important to close the container when not actively using the sodium sulfate.

10.5 Sonicator Tuning and Horn Inspection

10.5.1 Every week the sonicator horns are inspected for pitting and the condition is recorded in the Sonicator logbook. The degree of pitting is decided based on the horn's likeness to one of three reference images (Figure 1).

10.5.1.1 If the condition of the sonicator horn is determined to be poor. The sonicator must be removed from service until the probe is replaced.

10.5.2 If the sonicator is not self-tuning, the sonicator must be tuned once a week or whenever a new horn is installed. Tuning is documented in the sonicator maintenance log.

10.5.3 Starting at a power setting of 1, tune the sonicator so that the output is less than 20%.

10.5.4 Repeat the tune at a power setting of 5 and 10. At each power setting, tune the sonicator so that the output is less than 20%.

10.5.5 If the output is over 20%, consult your supervisor and the manufacturer's manual for troubleshooting help.

10.6 Assemble and clean the glassware immediately before use.

NOTE: Rotate glassware; do not use specific glassware or positions for the MB and LCS/LCSD.

10.6.1 Rinse 400-mL thick-walled beakers with methylene chloride.

NOTE: In order to prevent phthalate contamination, never touch the inside of a beaker with gloves on. When rinsing beakers be sure to keep gloves away from the mouth of the beaker.

10.6.2 Without gloves on, fold a 18 cm diameter cellulose filter paper in quarters.

Open the folds to create a cone. Place the filter paper in the bottom of a conical stainless steel funnel. Place the funnel on a 250-mL media bottle.

NOTE: For low-level phthalate analysis by 8270 SIM, use glass wool in place of filter paper. Be sure not to touch the glass wool with gloves. Check the Method Comments to determine if this is necessary and see Section 4.6.

10.6.3 Place approximately 1 tablespoon of baked sodium sulfate in the funnel. Rinse all surfaces of the funnel, the filter and the sodium sulfate with methylene chloride or acetone/methylene chloride (depending on the extraction solvent, see Section 10.8) so all surfaces of the funnel, filter, and sodium sulfate are rinsed.

NOTE: When preparing glassware for the extraction of wipe samples, sodium sulfate is not necessary and the solvent used in the rinse should be the solvent used in the extraction of the wipe samples. (Normally hexane for methods 8081 and 8082).

10.6.4 Allow the solvent to drain completely into the media bottle. Swirl the media bottle to ensure all surfaces come into contact with the solvent. Add additional solvent to the rinse if necessary.

10.6.5 Pour the solvent out of the media bottle over the stem of the stainless steel funnel to rinse the funnel stem.

10.6.6 Discard the solvent in the correct waste stream.

10.7 Aliquot Samples

10.7.1 If the sample is a wipe, the sonication can be performed with the wipe in its original container if the original container is large enough. Otherwise, transfer the wipe and any solvent from the original container to a clean beaker.

10.7.2 For each MB and LCS, place a clean wipe into a labeled beaker and proceed to section 10.8.

10.7.3 If the sample is a soil, mix and homogenize samples according to the instructions provided in SOP DV-QA-0023 Subsampling. Use a disposable wooden spatula or a metal spatula that has been rinsed with methylene chloride and dried with a lab tissue.

10.7.4 Break the sample aliquot up into small pieces. The aliquot must not contain particles or clumps bigger than ½ inch in diameter in order to facilitate a complete extraction.

10.7.5 Label a 400-mL beaker with the sample ID, method, and batch number.

10.7.6 Weigh 30 to 33 g of sample into the labeled beaker. Record the weight to the nearest 0.1 g directly into the LIMS or hand record the weight on

the benchsheet.

NOTE: Some clients may require the initial aliquot to be adjusted based on the percent moisture of the sample. In those cases, it might be necessary to aliquot more than 33 g of sample. If this is required, the Method Comments will state "Perform Calculation". The laboratory's LIMS (TALS) will calculate the required initial weight of wet sample needed to ensure at least 30 g of dry sample is included in the initial aliquot. In TALS, under the Batch Notes, enter a "1" in the "Perform Calculation" field. TALS will then calculate the required initial weight of wet sample needed under the "Target Amount" field in the Worksheet tab. Weigh out at least that mass of wet sample.

10.7.7 Add approximately 1 tablespoon of baked sodium sulfate to the beaker and mix well. If the sample is especially wet, more sodium sulfate will be needed to ensure the sample is free-flowing. If the sample is extremely wet, the initial sample weight might have to be reduced in order to keep the volume of sample and sodium sulfate in the beaker to a level that the horn can still thoroughly disrupt. Document in an NCM if additional sodium sulfate is added.

10.7.8 For each MB and LCS sample, weigh 30 to 33 g of baked Ottawa sand into labeled beakers. Add 1 tablespoon of baked sodium sulfate to the beaker and mix well. Record a nominal weight of 30 g in the initial volume field, but record the actual weight to the nearest 0.1 g in the notes column.

10.7.9 Cap the beaker tightly with aluminum foil.

10.7.10 Place the beaker on a cart next to the sample container so that a second analyst can check the labels. This is documented on the Organic Extraction Worksheet (See WI-DV-0009 Spike/Surrogating and Review Procedure for Organic Extractions).

10.8 Prepare a bottle with a bottle-top dispenser with the appropriate solvent.

10.8.1 Methylene Chloride is used for soil and wipe samples for the following methods:

- SW-846 8015B
- SW-846 8015C
- SW-846 8015D
- Alaska Methods AK102 and AK103
- NWTPH-Dx
- Oklahoma DRO Method

10.8.2 For soil extraction of all other methods, the solvent used is a 1:1 mixture of methylene chloride and acetone.

10.8.3 For wipe samples by method 8081 and 8082, the solvent used is hexane.

10.8.4 For wipe samples by method 8270, the solvent used is a 1:1 mixture of methylene chloride and acetone.

10.9 Add Surrogate, Spikes, and Solvent to Field Samples and all QC samples.

10.9.1 The standards should be allowed to come to room temperature before spiking the samples. Record the ID of the standard and pipettor(s) used on the benchsheet.

NOTE: The addition of spikes and surrogates to samples must be done only immediately after a second analyst has reviewed the batch. Reference work instruction WI-DV-0009 Spike/Surrogating and Review Procedure for Organic Extractions for Surrogate and spike volumes.

10.9.2 Only one batch should be surrogated at a time to ensure the correct standards are used and to ensure the solvent is added as soon as possible to the samples.

10.9.3 Ensure that the sample is free flowing before adding the surrogate standard. If the sample has become hard, gently tap the beaker to break up the solid, or pull back the foil and mix with a wooden spatula if necessary.

10.9.4 Using a calibrated pipette, add the appropriate volume of the appropriate working surrogate standard to the beaker for each field sample and method blank. Do this by punching a hole in the aluminum foil cap with the pipette tip.

10.9.5 Using a calibrated pipette, add the appropriate volume of the appropriate working spike standard to the beaker for each LCS, LCSD and MS/MSD. Do this by punching a 2nd hole in the aluminum foil cap with the pipette tip.

10.9.6 Immediately after the addition of the spike standard to the LCS, MS, & MSD sample, add approximately 100 mL of the appropriate solvent. Note that the solvent should be added as soon as possible after the addition of the spiking standards to prevent loss of the more volatile extractables. Sufficient solvent should be added so that the solvent level is at least ¾ inch above the solids.

NOTE: When hexane is used as the extraction solvent, use only enough to cover the wipe, i.e., approximately 50 mL. This will help facilitate the concentration of the extract later.

10.10 Rinse the disrupter horn with methylene chloride and wipe down with a clean laboratory tissue.

10.11 Place the bottom surface of the disrupter horn tip just below the surface of the solvent, but above the sediment layer.

10.12 Sonicate for three minutes, making sure the entire sample is agitated. The output should be set at 10 for the ¾-inch standard horn. The mode switch should be set on pulse, and the percent-duty cycle knob at 50%, for a total process time of 1:30 (1 minute 30 seconds).

10.13 Ensure the filter paper is wet before decanting and filtering occurs. Decant and filter the extract through the prepared stainless steel funnel into the media bottle. Immediately rinse the sodium sulfate in the funnel with at least 50 mL of solvent, ensuring that all sides of the filter paper have been rinsed also. **This is a critical step and must be performed as soon as the extract has drained from the funnel and must be done with at least 50 mL of solvent.**

NOTE: If proper rinsing has occurred, there should **not** be a significant yellow ring of residue (from the spike standards) around the top of the filter paper.

10.14 Repeat the extraction two more times with the appropriate solvent. Each time add sufficient solvent so that the solvent level is at least ¾ inch above the solids. If wipes are being extracted with hexane, then repeat two or more times with additional 50-mL portions of solvent.

10.15 Decant off the solvent after each sonication. After the third and final sonication, pour the entire extract into the funnel. Do not attempt to decant at this step but make every effort to recover all solvent from the beaker. If sufficient room in the media jar exists, rinse the beaker and/or the funnel with an additional 10 to 20 mL of solvent and add the rinse to the funnel.

10.16 Once the solvent has completely drained into the media bottle, dispose of the solid sample and the sodium sulfate into Waste Stream D and cap the media bottle containing the extract with aluminum foil.

10.17 Be sure to rinse the disrupter horn between samples following the procedure in Section 10.10.

10.18 If the extract contains visible solids, it will be necessary to filter the extract again. This filtration can be performed immediately before the concentration step by filtering the extract through another filter paper and funnel directly into the K-D apparatus. If the extract clogs the filter or filtration is extremely slow, the filter and funnel can be placed on a filter flask and a vacuum can be applied.

10.19 Place the extracts in a refrigerator until concentration, ensuring that the extracts in 1:1 methylene chloride:acetone are placed in a flammable rated refrigerator. Document on the benchsheet in which refrigerator the extracts are stored and the total extract count for the batch.

10.20 Handwritten notes on the benchsheet are entered into LIMS, and the transcribed data must be verified by a second person. This verification is documented on the Organic Extraction Checklist (see WI-DV-009 Spike/Surrogating and Review

Procedure for Organic Extractions).

10.21 Maintenance

10.21.1 Unless self tuning, the sonicators must be tuned once a week. See Section 10.4.

10.21.2 The probes must be inspected once a week and replaced if excessively worn.

10.22 Troubleshooting

10.22.1 If the sonicator is not working properly, (either not disrupting the soil sufficiently or over-loading) separate the converter from the horn and the horn from the probe. Always use the special wrenches to avoid damaging the parts. Clean all points of contact with either acetone or isopropyl alcohol and then re-assemble and tighten down with the wrenches.

10.22.2 If after following the steps in Section 10.22.1, the sonicator is still not working properly, try to isolate the problem by plugging the converter into a different control box. If the problem goes away, then the control box needs to be sent off for service. If the problem does not go away, proceed to Section 10.22.3.

10.22.3 If after following the steps in Sections 10.22.1 and 10.22.2 the sonicator is still not working properly, then the problem must be in the converter or the horn or probe. Switch the converter to determine if the converter needs to be sent off for repair. If the converter operates properly with a different horn and probe, then the probe needs to be replaced.

11.0 Calibration

Not applicable to this procedure.

12.0 Calculations / Data Reduction

Not Applicable.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-S-006 Detection and Quantitation Limits. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DoD/DOE QSM or for programs which require the use of Method 8270D, Revision 5. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

13.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

13.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024 Training.

13.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024 Training.

14.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method, the policies in section 13 of the Environmental Health and Safety Manual for "Waste Management and Pollution Prevention", and the Waste Management procedure, DV-HS-001P Waste Management Plan.

15.2 Waste Streams Produced By This Method

15.2.1 Methylene chloride – Waste Stream B

15.2.2 Flammable solvent – Waste Stream C

15.2.3 1:1 MeCl2:Acetone – Waste Stream CA

15.2.4 Solid waste/sodium sulfate – Waste Stream D

15.2.5 Expired Standards/Reagents – Contact Waste Coordinator for guidance

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 3550C Ultrasonic Extraction, Revision 3, February 2007.

16.2 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Method 3550B Ultrasonic Extraction, Revision 3, December 1996.

16.3 Alaska Method AK102, "For the Determination of Diesel Range Organics", Version 04/08/02.

16.4 Alaska Method AK103, "For the Determination of Residual Range Organics", Version 04/08/02.

16.5 Oklahoma Department of Environmental Quality, Methods 8000/8100 (modified) Diesel Range Organics (DRO), October 22, 1997 Rev. 4.1.

16.6 NWTPH-HCID "Hydrocarbon Identification Method for Soil and Water," Manchester Environmental Laboratory, Dept. of Ecology, State of Washington.

17.0 Method Modifications:

17.1 SW-846 Method 3550C Section 11.3.5 calls for all three extractions to be filtered a second time through the same filter. The SOP only requires each extract to be

filtered after each extraction process. The QC has shown great recovery with the use of only the filtration proceeding each extraction.

17.2 SW-846 Method 3550C Section 11.3 instructs that the surrogate and spike compounds should be added to the sample before the sample is mixed with sodium sulfate. This SOP calls for the sample to be mixed thoroughly with sodium sulfate before the surrogate and spike compounds are added. This is done per EPA Memo dated August 5, 2010 titled "Spiking (Prior To vs. After Sample Drying) Issue in SW-846 Organic Extraction Methods."

17.3 SW-846 Method 3550C calls for the use of Büchner funnels and vacuum filtration of all extracts. This SOP calls for the use of conical funnels. This was done to prevent the extract from becoming trapped in the sodium sulfate in the Büchner funnel and specifically to improve the recoveries of benzoic acid, 2,4-dinitrophenol, and 4,6-dinitro-2-methylphenol.

17.4 Oklahoma Department of Environmental Quality method calls for the aliquot not to exceed 20 g. This procedure calls for the soil aliquot to be 30 g to 33 g.

17.5 Oklahoma Department of Environmental Quality DRO method calls for solvent to be added to the sample in a 1:1 ratio (milliliters of solvent to grams of sample). This procedure calls for 100 mL of solvent to be added to 30 g of sample.

17.6 Method from the state of Washington uses a 10 g soil sample that is shaken and processed in a sonic bath. This procedure calls for the soil aliquot to be 30 g to 33 g and is processed directly with a sonicator horn.

17.7 Methods 3550B and 3550C instruct the lab to determine the dry weight of the sample. This is performed according to SOP DV-WC-0023 Percent Moisture in Soils and Wastes and is not included in this SOP.

17.8 The medium/high concentration extraction procedure described in Methods 3550B and 3550C is not addressed in this SOP.

17.9 Method AK102 and AK103 calls for samples to be extracted by soxhlet. Valid MDLs and IDOCs have been completed using this procedure, therefore method AK102 and AK103 are listed as a possible determinative methods by this procedure.

18.0 Attachments

Table 1: Determinative Methods Using Ultrasonic Extraction
Figure 1: Sonicator Horn Condition Reference Images.

19.0 Revision History

This section has been added beginning with Revision 0. Only details of the last two revisions are incorporated into this SOP. Prior revisions are documented in the QA files and available upon request.

Rev 15, dated 11 June 2021

- Annual Review
- Updated copyright information

- Changed TestAmerica to Eurofins TestAmerica throughout
- Removed QSM versions and instead referenced SOP DV-QA-024P QA/QC Requirements for Federal Programs for information about DoD QSMs.
- Updated language and formatting throughout

Rev 14, dated 04 May 2020

- Annual Technical Review

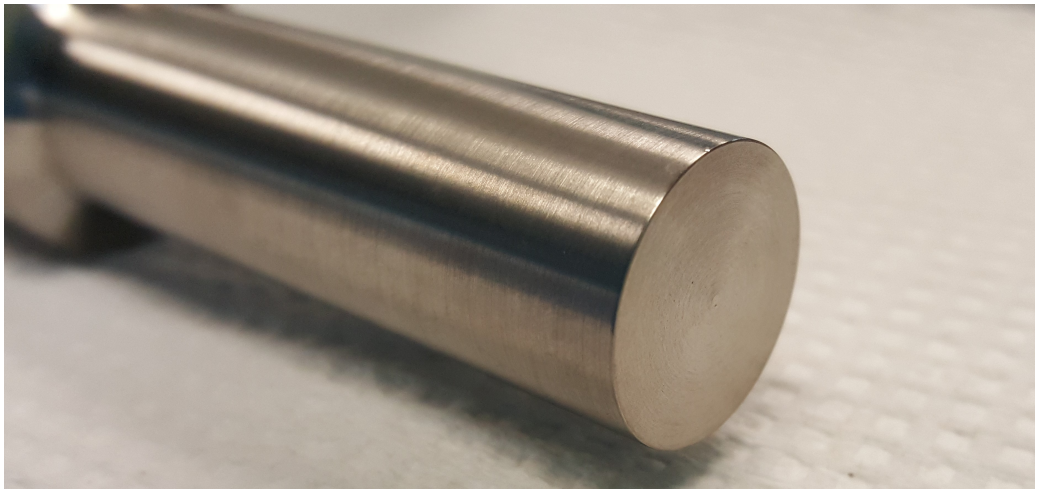
TABLE 1.

Determinative Methods Using Ultrasonic Extraction

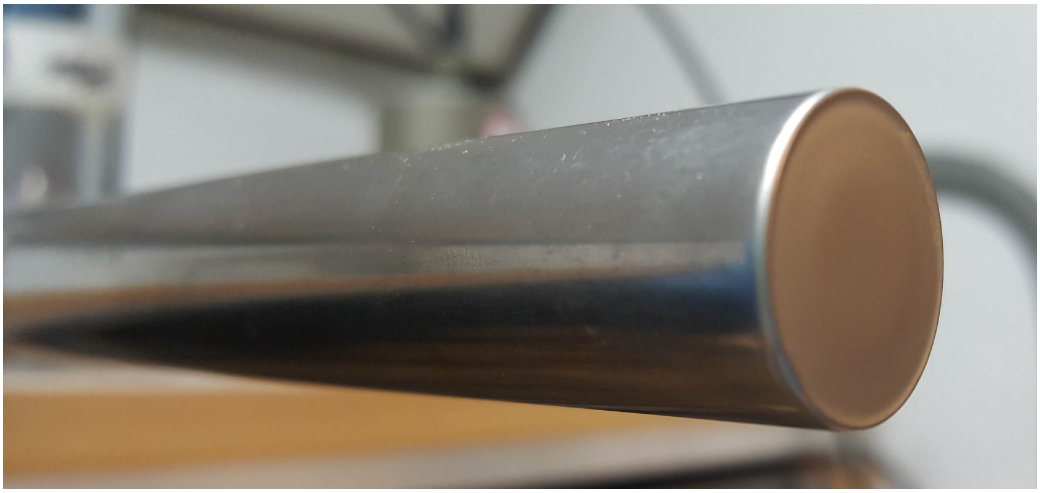
Method Description	Determinative Method	SOP
Diesel Range Organics, Jet Fuels, Motor Oil, Residual Range Organics	SW-846 8015B SW-846 8015C SW-846 8015D Alaska Methods AK102 & AK103 NWTPH-Dx Oklahoma DRO Method	DV-GC-0027
Chlorinated Pesticides	SW-846 8081A SW-846 8081B	DV-GC-0020
Polychlorinated Biphenyls	SW-846 8082 SW-846 8082A	DV-GC-0021
Polynuclear Aromatic Hydrocarbons	SW-846 8310	DV-LC-0009
Semi-volatiles by GC/MS	SW-846 8270C SW-846 8270D	DV-MS-0011 DV-MS-0012
Polynuclear Aromatic Hydrocarbons by GC/MS	SW-846 8270C SIM SW-846 8270D SIM	DV-MS-0002

Figure 1.

Sonicator Horn Condition Reference Images



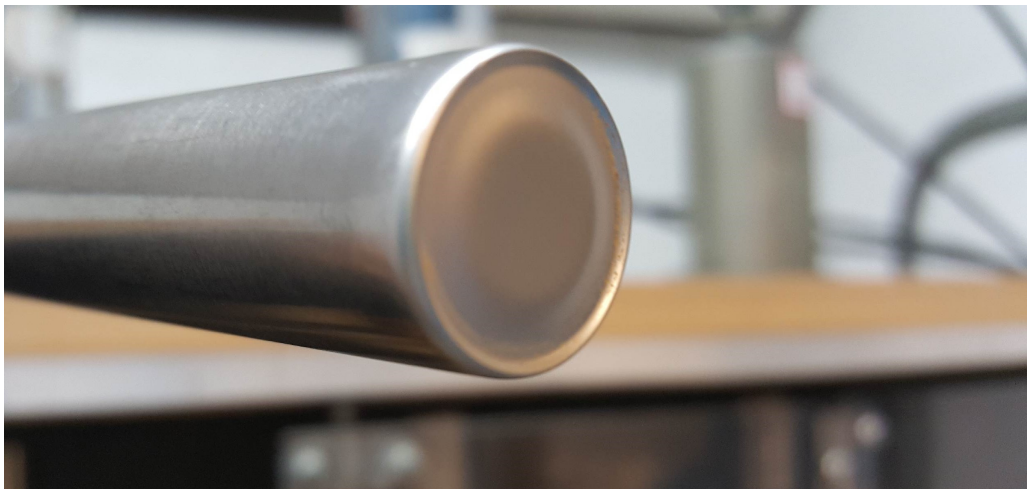
Condition: good; no further action needed.



Condition: fine; minor pitting, monitor condition.

Figure 1. (Cont.)

Sonicator Horn Condition Reference Images



Condition: poor; heavy pitting, replace horn.

ANALYTICAL METHODS

Section 10.0

Method: EPA Method TO-14A/TO-15 Volatile Organic Compounds (Standard/Quad)

Eurofins Air Toxics SOP #6 Revision 44 Effective Date: April 12, 2021 Methods Manual Summary

Description: 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 volatile organic compounds (VOCs) using EPA Method TO-14A/TO-15 protocols. An aliquot of up to 0.5 liters of air is withdrawn from the canister utilizing a volumetric loop or 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 secondary trap for further concentration and/or directly onto a GC/MS for separation and detection.

Eurofins Air Toxics maintains a suite of TO-14A/TO-15 methods, each optimized to efficiently meet the data objectives for a wide range of targeted concentration ranges. The methods, their reporting limits, and typical applications are summarized in the table below. This method summary describes TO-14A/TO-15 (Standard or Quad).

Eurofins Air Toxics Method	Base Reporting Limits	Typical Application
TO-14A/TO-15 (5&20)	5 – 20 ppbv	Soil gas and ppmv range vapor matrices
TO-14A/TO-15 (Standard or Quad)	0.5 – 5.0 ppbv	Ambient air, soil gas, and ppbv level vapor matrices
TO-15 (Extended)	0.2 – 5.0 ppbv	Ambient air and ppbv level vapor matrices
TO-14A/TO-15 (Low-level)	0.1 – 1.0 ppbv	Indoor and outdoor air
TO-14A/TO-15 SIM	0.01 – 0.5 ppbv	Indoor and outdoor air
TO-15 HSS	0.01 – 0.1 ppbv	Soil gas and other high concentration matrices

Certain compounds are not included in Eurofins Air Toxics' standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, Eurofins Air Toxics reports these non-routine compounds with partial validation. Validation may include a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification analyzed, and no method detection limit study performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Eurofins Air Toxics takes no modifications of technical significance to Method TO-15 for the "Quad" configurations. 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, the laboratory 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 TO-14A Method Modifications

Requirement	TO-14A	Eurofins Air Toxics 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 when recovery is less than 60%.
Initial Calibration	≤ 30% RSD for listed 39 VOCs	Follow TO-15 requirements of ≤ 30% RSD with 2 of Eurofins Air Toxics' 62 standard compounds allowed out to ≤ 40% RSD

The standard target analyte list, reporting limit (RL) also referred to as Limit of Quantitation, QC criteria, and QC summary can be found in Tables 2 through 6.

Table 2. Method TO-14A/TO-15 Analyte List (Quad)

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
1,1,2,2-Tetrachloroethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,1,2-Trichloroethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,1-Dichloroethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,1-Dichloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2,4-Trichlorobenzene	2.0	≤ 30%	70 – 130	70 – 130	± 25
1,2,4-Trimethylbenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dibromoethane (EDB)	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichlorobenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichloroethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,2-Dichloropropane	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,3,5-Trimethylbenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,3-Dichlorobenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,4-Dichlorobenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Benzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Bromomethane	5.0	≤ 30%	70 – 130	70 – 130	± 25

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
Carbon Tetrachloride	0.5	≤ 30%	70 – 130	70 – 130	± 25
Chlorobenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Chloroethane	2.0	≤ 30%	70 – 130	70 – 130	± 25
Chloroform	0.5	≤ 30%	70 – 130	70 – 130	± 25
Chloromethane	5.0	≤ 30%	70 – 130	70 – 130	± 25
Chlorotoluene (Benzyl Chloride)	0.5	≤ 30%	70 – 130	70 – 130	± 25
cis-1,2-Dichloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
cis-1,3-Dichloropropene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Dichloromethane (Methylene Chloride)	5.0	≤ 30%	70 – 130	70 – 130	± 25
Ethylbenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Freon 11 (Trichlorofluoromethane)	0.5	≤ 30%	70 – 130	70 – 130	± 25
Freon 113 (Trichlorotrifluoroethane)	0.5	≤ 30%	70 – 130	70 – 130	± 25
Freon 114	0.5	≤ 30%	70 – 130	70 – 130	± 25
Freon 12 (Dichlorodifluoromethane)	0.5	≤ 30%	70 – 130	70 – 130	± 25
Hexachlorobutadiene	2.0	≤ 30%	70 – 130	70 – 130	± 25
m,p-Xylene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Methyl Chloroform (1,1,1-Trichloroethane)	0.5	≤ 30%	70 – 130	70 – 130	± 25
o-Xylene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Styrene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Tetrachloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Toluene	0.5	≤ 30%	70 – 130	70 – 130	± 25
trans-1,3-Dichloropropene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Trichloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Vinyl Chloride	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,3-Butadiene	0.5	≤ 30%	70 – 130	70 – 130	± 25
1,4-Dioxane	2.0	≤ 30%	70 – 130	70 – 130	± 25
2-Butanone (Methyl Ethyl Ketone)	2.0	≤ 30%	70 – 130	70 – 130	± 25
2-Hexanone	2.0	≤ 30%	70 – 130	70 – 130	± 25
4-Ethyltoluene	0.5	≤ 30%	70 – 130	70 – 130	± 25
4-Methyl-2-Pentanone (MIBK)	0.5	≤ 30%	70 – 130	70 – 130	± 25
Acetone	5.0	≤ 30%	70 – 130	70 – 130	± 25
Bromodichloromethane	0.5	≤ 30%	70 – 130	70 – 130	± 25

Analyte	RL/LOQ (ppbv)	QC Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
Bromoform	0.5	≤ 30%	70 – 130	70 – 130	± 25
Carbon Disulfide	2.0	≤ 30%	70 – 130	70 – 130	± 25
Cyclohexane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Dibromochloromethane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Ethanol	5.0	≤ 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	2.0	≤ 30%	70 – 130	70 – 130	± 25
Methyl t-Butyl Ether (MTBE)	2.0	≤ 30%	70 – 130	70 – 130	± 25
Tetrahydrofuran	0.5	≤ 30%	70 – 130	70 – 130	± 25
trans-1,2-Dichloroethene	0.5	≤ 30%	70 – 130	70 – 130	± 25
2,2,4-Trimethylpentane	0.5	≤ 30%	70 – 130	70 – 130	± 25
Cumene	0.5	≤ 30%	70 – 130	70 – 130	± 25
Propylbenzene	0.5	≤ 30%	70 – 130	70 – 130	± 25
3-Chloroprene	2.0	≤ 30%	70 – 130	70 – 130	± 25
Naphthalene*	1.0	≤40%	60 – 140	60 – 140	± 25
TPH (Gasoline) **	50	1-Point Calibration	N/A	ICV only; 60 – 140	± 25
NMOC (Hexane/Heptane)**	10	1-Point Calibration	N/A	NA	± 25

*Due to its low vapor pressure, Naphthalene may exceed TO-15 performance requirements. The wider QC limits reflect typical performance. Although Naphthalene is not on Eurofins Air Toxics "standard" TO-15 list, it is commonly requested and included in Table 2.

**TPH and NMOC are not on Eurofins Air Toxics' "standard" TO-15 list, but are included in Table 2 due to common requests.

Table 2 is the list of Standard compounds, reporting limits and QC acceptance criteria. Each project may be customized as needed. Additional compounds and different reporting limits may be obtainable and/or achieved upon request.

Table 3. Method TO-15 Additional Analyte List (Quad)

Analyte	RL/LOQ (ppbv)	QA Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
1,2,3-Trichloropropane	0.4	≤30%	70 – 130	70 – 130	± 25
1,2,3-Trichlorobenzene	0.8	≤30%	70 – 130	70 – 130	± 25

Analyte	RL/LOQ (ppbv)	QA Acceptance Criteria			
		ICAL (%RSD)	CCV (%R)	ICV/LCS (%R)	Precision Limits (Max. RPD)
2-Chlorotoluene	0.4	≤30%	70 – 130	70 – 130	± 25
4-Isopropyltoluene (p-Cymene)	0.8	≤30%	70 – 130	70 – 130	± 25
Butane	2.0	≤30%	70 – 130	70 – 130	± 25
Butyl Benzene	0.4	≤30%	70 – 130	70 – 130	± 25
Dibromomethane	0.4	≤30%	70 – 130	70 – 130	± 25
Ethyl Acetate	2.0	≤30%	70 – 130	70 – 130	± 25
Ethyl Ether	0.8	≤30%	70 – 130	70 – 130	± 25
Freon 22 (Chlorodifluoromethane)	0.8	≤30%	70 – 130	70 – 130	± 25
Methyl Methacrylate	0.8	≤30%	70 – 130	70 – 130	± 25
n-Butanol (1-Butanol)	0.8	≤30%	70 – 130	70 – 130	± 25
Nonane	0.8	≤30%	70 – 130	70 – 130	± 25
n-Pentane	0.8	≤30%	70 – 130	70 – 130	± 25
Octane	0.4	≤30%	70 – 130	70 – 130	± 25
sec-Butylbenzene	0.8	≤30%	70 – 130	70 – 130	± 25
tert-Butyl Alcohol	2.0	≤30%	70 – 130	70 – 130	± 25
tert-Butyl Benzene	0.8	≤30%	70 – 130	70 – 130	± 25
Vinyl Acetate	0.8	≤30%	70 – 130	70 – 130	± 25
Vinyl Bromide	0.8	≤30%	70 – 130	70 – 130	± 25

Table 3 is the list of additional Method TO-15 compounds that may be requested upon request with full QC – 5-point calibration, second source calibration verification, continuing calibration verification, laboratory control spike, and method detection limit study.

Table 4. Internal Standards

Analyte	Accuracy (% R)
Bromochloromethane	60 – 140
1,4-Difluorobenzene	60 – 140
Chlorobenzene-d_5	60 – 140

Table 5. Surrogates

Analyte	Accuracy (% R)
1,2-Dichloroethane-d_4	70 – 130
Toluene-d_8	70 – 130
4-Bromofluorobenzene	70 – 130

Table 6. 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 Initial Calibration (ICAL)	Prior to sample analysis	% RSD ≤ 30 with 2 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 the time of calibration. If specified by the project, in-house generated control limits may be used.	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 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.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
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.
Laboratory Blank	After analysis of standards and prior to sample analysis, or when contamination is present.	Results less than the laboratory reporting limit	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 ±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.
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 Duplicates – Laboratory Control Spike Duplicates (LCSD)	One per analytical batch	RPD ≤25%	Narrate exceedances. If more than 5% of compound list is outside criteria or if compound has >40%RPD, investigate the cause and perform maintenance as required. If instrument maintenance is required, calibrate as needed.

APPENDIX D

LABORATORY ELAP CERTIFICATION

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CERTIFICATE OF ACCREDITATION

The ANSI National Accreditation Board

Hereby attests that

Eurofins Air Toxics
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

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

Air and Emissions		
Technology	**Method**	**Analyte**
GC/FID/dual TCD	ASTM D1945 Mod	n-Butane
GC/FID/dual TCD	ASTM D1945 Mod	Neopentane
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/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/SIM)	4-Methyl-2-pentanone (MIBK)
GC/MS	TO-15 (Full Scan/SIM)	Acetone
GC/MS	TO-15 (Full Scan/SIM)	alpha-Chlorotoluene
GC/MS	TO-15 (Full Scan/SIM)	Benzene
GC/MS	TO-15 (Full Scan/SIM)	Bromodichloromethane
GC/MS	TO-15 (Full Scan/SIM)	Bromoform

Air and Emissions		
Technology	**Method**	**Analyte**
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/SIM)	Dichlorodifluoromethane (Freon 12)
GC/MS	TO-15 (Full Scan)	Ethanol
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

Air and Emissions		
Technology	**Method**	**Analyte**
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)	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
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/SIM)	Vinyl chloride

Air and Emissions		
Technology	**Method**	**Analyte**
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
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	n-Heptane

Air and Emissions		
Technology	**Method**	**Analyte**
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	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
Preparation	**Method**	**Type**
Extraction of sorbent media	Eurofins Air Toxics SOP # 100	Solvent Extraction

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

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

EUROFINS TESTAMERICA DENVER
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ENVIRONMENTAL

Valid To: October 31, 2023 Certificate Number: 2907.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with the 2009 and 2016 TNI Environmental Testing Laboratory Standard, the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP), and the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.4 of the DoD/DOE Quality Systems Manual for Environmental Laboratories), and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP/MS, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.- Electronic Probes (pH, O_2), Oxygen Demand, Hazardous Waste Characteristics Tests, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, Total Organic Carbon, Total Organic Halide

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Metals			
Aluminum	EPA 200.7	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Antimony	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Arsenic	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Barium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Beryllium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Bismuth	----------	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Boron	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Cadmium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Calcium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D

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Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Chromium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Cobalt	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Copper	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Iron	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Lead	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Lithium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Magnesium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Manganese	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Mercury	EPA 245.1	EPA 7470A	EPA 7471A/7471B
Molybdenum	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Nickel	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Potassium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Selenium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Silica	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Silicon	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Silver	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Sodium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Strontium	EPA 200.7	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Sulfur	----------	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Thallium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Thorium	----------	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Tin	EPA 200.7	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Titanium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Tungsten	----------	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Uranium	EPA 200.8	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Vanadium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Zinc	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Zirconium	----------	EPA 6010B/6010C/6010D	----------
Nutrients			
Nitrate (as N)	EPA 300.0 By calculation	EPA 300.0 EPA 9056/9056A By Calculation/Nitrate by Calc	EPA 9056/9056A By Calculation/Nitrate by Calc

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Nitrate-Nitrite (as N)	EPA 300.0 EPA 353.2	EPA 300.0 EPA 353.2 EPA 9056/9056A	EPA 9056/9056A
Nitrite (as N)	EPA 300.0 EPA 353.2 SM 4500-NO_2 B	EPA 300.0 EPA 353.2 EPA 9056/9056A SM 4500-NO_2 B	EPA 353.2 EPA 9056/9056A
Orthophosphate (as P)	EPA 300.0	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Total Phosphorus	EPA 365.1	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Demands			
Total Organic Carbon	----------	EPA 9060/9060A	EPA 9060/9060A
Total Organic Halides	----------	EPA 9020B	----------
Wet Chemistry			
Alkalinity (Total Bicarbonate, Carbonate, and Hydroxide Alkalinity)	SM 2320B	SM 2320B	SM 2320B
Ammonia	EPA 350.1	EPA 350.1	----------
Biological Oxygen Demand	SM 5210B	SM 5210B	----------
Bromide	EPA 300.0	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Chloride	EPA 300.0 SM 4500-CL E	EPA 300.0 EPA 9056/9056A SM 4500-CL E	EPA 9056/9056A
Chemical Oxygen Demand	EPA 410.4	EPA 410.4	----------
Conductivity	----------	EPA 9050/9050A	EPA 9050/9050A
Cyanide	----------	EPA 9012A/9012B	EPA 9012A/9012B
Ferrous Iron	SM 3500Fe B, D	SM 3500Fe B, D	----------
Fluoride	EPA 300.0	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Flashpoint	----------	EPA 1010A	----------
Hexavalent Chromium	----------	EPA 7196A	EPA 7196A
Hardness, Total	SM 2340C	SM 2340C	----------
pH	SM 4500 H+B	EPA 9040B/9040C	EPA 9045C/9045D
Oil and Grease (HEM and SGT-HEM)	----------	EPA 1664A/1664B	----------
Percent Moisture	----------	----------	ASTM D2216
Perchlorate	----------	EPA 6860	EPA 6860
Phenols	----------	EPA 9066	----------
Solids, Total	----------	----------	SM 2540B
Solids, Total Suspended	SM 2540D	SM 2540D	SM 2540D
Solids, Total Dissolved	SM 2540C	SM 2540C	SM 2540C
Sulfate	EPA 300.0 SM 4500-SO4 E	EPA 300.0 EPA 9056/9056A SM 4500-SO4 E	EPA 9056/9056A
Sulfide, Total	SM 4500S2 D	EPA 9034/SM 4500S2 D	EPA 9034
Sulfide	----------	EPA 9030B	EPA 9030B

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Total Kjeldahl Nitrogen	EPA 351.2	EPA 351.2	EPA 351.2
Purgeable Organics (Volatiles)			
1,1,1,2-Tetrachloroethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1,1-Trichloroethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1,2,2-Tetrachloroethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1,2-Trichloro-1,2,2-trifluoroethane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1,2-Trichloroethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1-Dichloroethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,1-Dichloropropene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2 Dibromoethane (EDB)	EPA 624/624.1	EPA 8260B/8260C/8260D EPA 8011	EPA 8260B/8260C/8260D EPA 8011
1,2,3-Trichlorobenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2,3-Trichloropropane	EPA 624/624.1	EPA 8260B/8260C/8260D EPA 8011	EPA 8260B/8260C/8260D EPA 8011
1,2,3-Trimethylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2,4-Trichlorobenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2,4-Trimethylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Dibromo-3-chloropropane (DBCP)	EPA 624/624.1	EPA 8260B/8260C/8260D EPA 8011	EPA 8260B/8260C/8260D EPA 8011
1,2-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Dichloroethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Dichloropropane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,2-Xylene (o-Xylene)	EPA 624/624.1	EPA 8260B/8260C/8260D AK101/OK DEQ GRO	EPA 8260B/8260C/8260D AK101/OK DEQ GRO
1,3,5-Trichlorobenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,3,5-Trimethylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,3-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,3-Dichloropropane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,4-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
1,4-Dioxane	EPA 624/624.1	EPA 8260B/8260C/8260D EPA 8260B/8260C/8260D SIM	EPA 8260B/8260C/8260D EPA 8260B/8260C/8260D SIM
1-Chlorohexane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2,2-Dichloropropane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Butanone [Methyl Ethyl Ketone (MEK)]	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Chloro-1,3-butadiene (Chloroprene)	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Chloroethyl Vinyl Ether	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Chlorotoluene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Hexanone	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Nitropropane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
2-Pentanone	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
4-Chlorotoluene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
4-Isopropyltoluene (p-Cymene)	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
4-Methyl-2-pentanone (MIBK)	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Acetone	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Acetonitrile	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Acetylene	----------	RSK-175	----------
Acetylene Ethane	----------	RSK-175	----------
Acrolein	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Acrylonitrile	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Allyl Chloride (3-Chloro-1-propene)	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Benzene	EPA 624/624.1	EPA 8260B/8260C/8260D AK101/OK DEQ GRO	EPA 8260B/8260C/8260D AK101/OK DEQ GRO
Bromobenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Bromochloromethane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Bromodichloromethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Bromoform	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Bromomethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Butadiene	----------	EPA 8260B/8260C/8260D SIM	EPA 8260B/8260C/8260D SIM
Carbon Disulfide	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Carbon Tetrachloride	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Chlorobenzene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Chloroethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Chloroform	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Chloromethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
cis-1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
cis-1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
cis-1,4-Dichloro-2-butene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Cyclohexane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Cyclohexanone	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Dibromochloromethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Dibromomethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Dichlorodifluoromethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Dichlorofluoromethane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Diethyl Ether (Ethyl Ether)	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Di-isopropylether (Isopropyl ether)	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Ethane	----------	RSK-175	----------
Ethanol	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Ethyl Acetate	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Ethyl Benzene	EPA 624/624.1	EPA 8260B/8260C/8260D AK101/OK DEQ GRO	EPA 8260B/8260C/8260D AK101/OK DEQ GRO
Ethyl Methacrylate	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Ethyl Tert-Butyl Ether	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Ethylene (Ethene)	----------	RSK-175	----------
Gas Range Organics (GRO)	----------	EPA 8015B/8015C/8015D/ AK101/OK DEQ GRO/NWTPH-Gx	EPA 8015B/8015C/8015D/ AK101/OK DEQ GRO/NWTPH-Gx

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Hexachlorobutadiene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Hexane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Iodomethane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Isobutyl alcohol (2-Methyl-1-propanol)	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Isopropyl Alcohol	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Isopropylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
m+p-Xylene	EPA 624/624.1	EPA 8260B/8260C/8260D AK101/OK DEQ GRO	EPA 8260B/8260C/8260D AK101/ K DEQ GRO
Methacrylonitrile	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Methane	----------	RSK-175	----------
Methyl Acetate	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Methyl Cyclohexane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Methyl Methacrylate	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Methyl Tert-Butyl Ether (MtBE)	EPA 624/624.1	EPA 8260B/8260C/8260D OK DEQ GRO	EPA 8260B/8260C/8260D OK DEQ GRO
Methylene Chloride	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Naphthalene	EPA 624/624.1	EPA 8260B/8260C/8260D OK DEQ GRO	EPA 8260B/8260C/8260D OK DEQ GRO
n-Butyl Alcohol (n-Butanol)	----------	EPA 8260B/8260C/8260D EPA 8015B/8015C	EPA 8260B/8260C/8260D EPA 8015B/8015C
n-Butylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
n-Propylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Pentachloroethane	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Propionitrile	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
sec-Butylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Styrene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
tert-Butyl Alcohol (2-Methyl-2-propanol)	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
tert-Butylbenzene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Tetrachloroethene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Tetrahydrofuran	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Toluene	EPA 624/624.1	EPA 8260B/8260C/8260D AK101/OK DEQ GRO	EPA 8260B/8260C/8260D AK101/OK DEQ GRO
Total Petroleum Hydrocarbons (TPH)	EPA 1664A/1664B	EPA 1664A/1664B	----------
trans-1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
trans-1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
trans-1,4-Dichloro-2-butene	----------	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Trichloroethene	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Trichlorofluoromethane	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Vinyl Acetate	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Vinyl Chloride	EPA 624/624.1	EPA 8260B/8260C/8260D	EPA 8260B/8260C/8260D
Xylenes, Total	EPA 624/624.1	EPA 8260B/8260C/8260D AK101/OK DEQ GRO	EPA 8260B/8260C/8260D AK101/OK DEQ GRO
Extractable Organics (Semivolatiles)			
1,1-Biphenyl	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
1,2,4,5-Tetrachlorobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,2,4-Trichlorobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,2-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,2-Diphenylhydrazine (Azobenzene)	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,3,5-Trinitrobenzene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,3-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,3-Dinitrobenzene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,4-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,4-Dinitrobenzene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,4-Dioxane	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1,4-Naphthoquinone	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1-Chloronaphthalene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
1-Methylnaphthalene	----------	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
1-Naphthylamine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,2-oxybis(1-chloropropane) [bis (2-Chloroisopropyl) Ether]	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,3,4,6-Tetrachlorophenol	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,4,5-Trichlorophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,4,6-Tribromophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,4,6-Trichlorophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,4-Dichlorophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,4-Dimethylphenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,4-Dinitrophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,4-Dinitrotoluene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,6-Dichlorophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2,6-Dinitrotoluene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Acetylaminofluorene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Chloronaphthalene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Chlorophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-methyl-4,6-Dinitrophenol (Dinoseb)	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Methylnaphthalene	----------	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
2-Methylphenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Naphthylamine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Nitroaniline	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Nitrophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-Picoline	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
2-sec-butyl-4,6-Dinitrophenol	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
3,3'-Dichlorobenzidine	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
3,3-Dimethylbenzidine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
3+4-Methylphenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
3-Methylcholanthrene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
3-Nitroaniline	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
4,6-Dinitro-2-methylphenol	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
4-Aminobiphenyl	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
4-Bromophenyl phenyl ether	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
4-chloro-3-Methylphenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
4-Chloroanilene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
4-Chlorophenyl phenyl ether	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
4-Nitroaniline	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
4-Nitrophenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
5-nitro-o-Toluidine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
7,12-Dimethylbenz(a)anthracene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Acenaphthene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Acenaphthylene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Acetophenone	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Alachlor	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
alpha-, alpha-Dimethylphenethylamine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Aniline	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Anthracene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Aramite	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Atrazine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Azobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Benzaldehyde	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Benzidine	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Benzo(a)anthracene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Benzo(a)pyrene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Benzo(b)fluoranthene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Benzo(ghi)perylene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Benzo(k)fluoranthene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Benzoic Acid	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Benzyl Alcohol	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
bis (2-Chloroethoxy) Methane	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
bis (2-Chloroethyl) Ether	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
bis (2-Ethylhexyl) Phthalate	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
butyl Benzyl Phthalate	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Caprolactam	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Carbazole	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Chlorobenzilate	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Chrysene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Cresols	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Diallate	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Dibenzo (a,h) anthracene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Dibenzofuran	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Diesel Range Organics (DRO)	----------	EPA 8015B/8015C/8015D AK102/8015D/OK DEQ DRO/NWTPH-Dx	EPA 8015B/8015C/8015D AK102/8015D/OK DEQ DRO/NWTPH-Dx
Diethyl Phthalate	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Dimethoate	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Dimethyl Phthalate	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
di-n-butyl Phthalate	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
di-n-octyl Phthalate	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
woDiphenylamine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Disulfoton	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Ethyl Methanesulfonate	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Famphur	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Fluorene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Fluoroanthene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Hexachlorobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Hexachlorobutadiene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Hexachlorocyclopentadiene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Hexachloroethane	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Hexachlorophene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Hexachloropropene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Indeno (1,2,3-cd) pyrene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Isodrin	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Isophorone	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Isosafrole	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Methapyrilene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Methyl Methane Sulfonate	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Motor Oil (Residual Range Organics)	----------	EPA 8015B/8015C/8015D AK103/OK DEQ RRO	EPA 8015B/ 8015C/8015D AK103/ OK DEQ RRO
Naphthalene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Nitrobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Nitroquinoline-1-oxide (4-Nitroquinoline-1-oxide)	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosodiethylamine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosodimethylamine	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosodi-n-butylamine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosodi-n-propylamine	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosodiphenylamine	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosomethylethylamine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosomorpholine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
N-Nitrosopiperidine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
N-Nitrosopyrrolidine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
o,o,o-triethyl Phosphorothioate	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
o-Toluidine	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Parathion, ethyl	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Parathion, methyl	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
p-Dimethylaminoazobenzene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Pentachlorobenzene	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Pentachloroethane	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Pentachloronitobenzene	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Pentachlorophenol	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8321A/8321B	EPA 8270C/8270D/8270E EPA 8321A/8321B
Phenacetin	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Phenanthrene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Phenol	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Phorate	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
p-Phenylene Diamine	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Pronamide	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Pyrene	EPA 625/625.1	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM	EPA 8270C/8270D/8270E EPA 8270C/8270D/8270E SIM
Pyridine	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Safrole	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Sulfotepp	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Thionazin	----------	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Tributyl phosphate	EPA 625/625.1	EPA 8270C/8270D/8270E	EPA 8270C/8270D/8270E
Pesticides/Herbicides/PCBs			
2,4,5-T	----------	EPA 8151A	EPA 8151A
2,4,5-TP	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4-D	----------	EPA 8151A	EPA 8151A
2,4-DB	----------	EPA 8321A/8321B	EPA 8321A/8321B
4,4'-DDD	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDE	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDT	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Aldrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
alpha-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
alpha-Chlordane (cis-Chlordane)	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Atrazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Azinophos ethyl	----------	EPA 8141A/8141B	EPA 8141A/8141B
Azinophos methyl	----------	EPA 8141A/8141B	EPA 8141A/8141B
beta-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Bolstar	----------	EPA 8141A/8141B	EPA 8141A/8141B
Chlordane (technical)	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Chloropyrifos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Coumaphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dalapon	----------	EPA 8151A EPA 8321A/8321B	EPA 8151A EPA 8321A/8321B

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
delta-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Demeton, total	----------	EPA 8141A/8141B	EPA 8141A/8141B
Demeton-O	----------	EPA 8141A/8141B	EPA 8141A/8141B
Demeton-S	----------	EPA 8141A/8141B	EPA 8141A/8141B
Diazinon	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dicamba	----------	EPA 8151A EPA 8321A/8321B	EPA 8151A EPA 8321A/8321B
Dichloroprop	----------	EPA 8151A EPA 8321A/8321B	EPA 8151A EPA 8321A/8321B
Dichlorovos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dieldrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Dimethoate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dinoseb (2-methyl-4,6-Dinitrophenol)	----------	EPA 8151A EPA 8321A/8321B	EPA 8321A/8321B
Disulfoton	----------	EPA 8141A/8141B	EPA 8141A/8141B
Endonsulfan sulfate	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endosulfan I	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endosulfan II	EPA 608/608.3	EPA 8081A /8081B	EPA 8081A/8081B
Endrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endrin aldehyde	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endrin ketone	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
EPN	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ethoprop	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ethyl Parathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Famphur	----------	EPA 8141A/8141B	EPA 8141A/8141B
Fensulfothion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Fenthion	----------	EPA 8141A/8141B	EPA 8141A/8141B
gamma-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
gamma-Chlordane (trans-Chlordane)	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Heptachlor	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Heptachlor epoxide	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Hexachlorobenzene	----------	EPA 8081A/8081B	EPA 8081A/8081B
Malathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
MCPA	----------	EPA 8151A EPA 8321A/8321B	EPA 8151A EPA 8321A/8321B
MCPP	----------	EPA 8151A EPA 8321A/8321B	EPA 8151A EPA 8321A/8321B
Merphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Methoxychlor	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Methyl parathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Mevinphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Naled	----------	EPA 8141A/8141B	EPA 8141A/8141B
o,o,o-Triethylphos Phorothioate	----------	EPA 8141A/8141B	EPA 8141A/8141B
PCB-1016 (Arochlor)	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1221	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1232	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
PCB-1242	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1248	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1254	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1260	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1262	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1268	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
Pentachlorophenol	----------	EPA 8151A	EPA 8151A
Phorate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Phosmet	----------	EPA 8141A/8141B	EPA 8141A/8141B
Picrolam	----------	EPA 8151A	EPA 8151A
Propazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ronnel	----------	EPA 8141A/8141B	EPA 8141A/8141B
Simazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Stirophos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Sulfotepp	----------	EPA 8141A/8141B	EPA 8141A/8141B
Thionazin	----------	EPA 8141A/8141B	EPA 8141A/8141B
Tokuthion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Total PCBs	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
Toxaphene	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Trichloronate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Explosives			
1,3,5-Trinitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
1,3-Dinitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,4,6-Trinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
3,5-Dinitroaniline	----------	EPA 8330B EPA 8321A/8321B	EPA 8330B EPA 8321A/8321B
2,4-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,4-Diamino-6-nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,6-Dinitroltoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,6-Diamino-4-nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2-amino-4,6-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
3-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
4-amino-2,6-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
4-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Nitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Nitroglycerin	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Nitroguanidine	----------	EPA 8321A/8321B	EPA 8321A/8321B
HMX (octahydro-1,3,5,7-tetrabitro-1,3,5,7-Tetrazocine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Pentaerythritoltetranitrate (PETN)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Picric acid	----------	EPA 8330A/8330B	EPA 8330A/8330B
RDX (hexahydro-1,3,5-trinitro-1,3,5-Triazine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Tetryl (methyl 2,4,6-Trinitrophenylnitramine	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
DNX (Hexahydro-1,3-dinitroso-5-nitro-1,3,5-triazine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
MNX (Hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
TNX (hexahydro-1,3,5-trinitroso-1,3,5-triazine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Triaminotrinitrobenzene (TATB)	----------	EPA 8330B EPA 8321A/8321B	EPA 8330B EPA 8321A/8321B
Explosives LC/MS/MS			
1,3,5-Trinitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
1,3-Dinitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4,6-Trinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
3,5-Dinitroaniline	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,6-Dinitroltoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2-Amino-4,6-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
3-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
4-Amino-2,6-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
4-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
DNX (hexahydro-1,3-dinitroso-5-nitro-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
MNX (hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Nitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
Nitroglycerin	----------	EPA 8321A/8321B	EPA 8321A/8321B
Nitroguanidine	----------	EPA 8321A/8321B	EPA 8321A/8321B
HMX (octahydro-1,3,5,7-tetrabitro-1,3,5,7-Tetrazocine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Pentaerythritoltetranitrate (PETN)	----------	EPA 8321A/8321B	EPA 8321A/8321B
RDX (hexahydro-1,3,5-trinitro-1,3,5-Triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Tetryl (methyl 2,4,6-Trinitrophenylnitramine	----------	EPA 8321A/8321B	EPA 8321A/8321B
TNX (hexahydro-1,3,5-trinitroso-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Tris(o-cresyl)phosphate	----------	EPA 8321A/8321B	EPA 8321A/8321B
Triaminotrinitrobenzene (TATB)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Chemical Warfare Agents			
Thiodiglycol (2,2'-Thiodiethanol)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Hazardous Waste Characteristics			
Conductivity	SM 2510B	EPA 9050A	EPA 9050A
Corrosivity	SM 4500 H+B	EPA 9040B/9040C	EPA 9045C/9045D
Paint filter liquids test	----------	EPA 9095A	EPA 9095A
Synthetic Precipitation Leaching Procedure (SPLP)	----------	EPA 1312	EPA 1312
Toxicity Characteristic Leaching Procedure	----------	EPA 1311	EPA 1311
California Waste Extraction Test	----------	CA WET	CA WET
Turbidity	EPA 180.1	----------	----------
Organic Prep Methods			
Continuous liquid-liquid extraction	----------	EPA 3520C	----------
Microwave extraction	----------	----------	EPA 3546
Separatory funnel liquid-liquid extraction	----------	EPA 3510C	----------
Solid phase extraction	----------	EPA 3535A	----------
Soxhlet extraction	----------	----------	EPA 3540C
Ultrasonic extraction	----------	----------	EPA 3550B/3550C
Volatiles purge and trap	----------	EPA 5030B	EPA 5030A EPA 5035/5035A
Waste dilution	----------	EPA 3580A	EPA 3580A
Organic Cleanup Procedures			
Florisil Cleanup	----------	EPA 3620B	EPA 3620B
Florisil Cleanup	----------	EPA 3620C	EPA 3620C
Sulfur Cleanup	----------	EPA 3660A/EPA 3660B	EPA 3660A/EPA 3660B
Sulfuric Acid/Permanganate Cleanup	----------	EPA 3665A	EPA 3665A
Metals Digestion			
Acid Digestion for Total Metals	----------	EPA 3010A	----------
Acid Digestion for Total Metals	----------	EPA 3020A	----------
Acid Digestion of Sediments, Sludges and Soils	----------	----------	EPA 3050B
Acid Digestion Total Recoverable or Dissolved Metals	----------	EPA 3005A	----------

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program), accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

WYOMING STORAGE TANK PROGRAM

Parameter/Analyte	**Method(s)**
Metals	
Cadmium	EPA 6010C/6010D
Chromium	EPA 6010C/6010D
Lead	EPA 6010C/6010D
Wet Chemistry	
Hexavalent chromium	EPA 7196A
Pureable Organics (Volatiles)	
tert-Amyl Methyl Ether	EPA 8260B/8260C
Benzene	EPA 8260B/8260C
tert-Butyl alcohol (2-Methyl-2-propanol)	EPA 8260B/8260C
1,2-Dichloroethane	EPA 8260B/8260C
Di-isopropylether	EPA 8260B/8260C
Ethyl benzene	EPA 8260B/8260C
Ethyl tert-butyl ether	EPA 8260B/8260C
Gas Range Organics (GRO)	EPA 8015B/8015C/8015D
Methyl tert-butyl ether (MTBE)	EPA 8260B/8260C
Naphthalene	EPA 8260B/8260C
Toluene	EPA 8260B/8260C
Xylenes, total	EPA 8260B/8260C
1,2-Xylene	EPA 8260B/8260C
m+p-Xylene	EPA 8260B/8260C
Extractable Organics (Semivolatiles)	
Diesel Range Organics (DRO)	EPA 8015B/8015C/8015D (WY: C10-C32)
Organic Prep Methods	
Volatiles Purge and Trap	EPA 5030B (water) /5030A (solids)

Accredited Laboratory

A2LA has accredited

EUROFINS TESTAMERICA DENVER

Arvada, CO

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2017, the 2009 and 2016 TNI Environmental Testing Laboratory Standard, the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP), and the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.4 of the DoD/DOE Quality System Manual for Environmental Laboratories (QSM), accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

Presented this 1st day of December 2021.

Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 2907.01
Valid to October 31, 2023

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.