DDMT Uniform Federal Policy-Quality Assurance Project Plan, Revision 1

Environmental Restoration Support at Former Defense Depot Memphis, Tennessee

Contract W90FYQ-09-D-0005, Task Order CK04

March 2018

Table of Contents

Tables

Figures

Appendices

Acronyms and Abbreviations

1,2-DCE	1,2-dichloroethene
AOC	area of concern
AS/SVE	air sparging with soil vapor extraction
ASTM	American Society for Testing and Materials
BEC	BRAC Environmental Coordinator
BFB	4-bromofluoro-benzene
bgs	below ground surface
BRAC	Base Realignment and Closure
CA	corrective action
CCB	continuing calibration blank
CCV	continuing calibration verification
cDCE	cis-1,2-dichloroethene
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CF	chloroform
CFR	Code of Federal Regulations
CoC	chain-of-custody
COR	Contracting Officer's Representative
CT	carbon tetrachloride
CTL	CT Laboratories, Inc.
CVOC	chlorinated volatile organic compound
CWM	chemical warfare material
CY	cubic yard
DCA	1,2-dichloroethane
DDMT	Defense Depot Memphis, Tennessee
DO	dissolved oxygen
DoD	Department of Defense
DQCR	daily quality control report
DQO	data quality objective
DSA	Diane Short & Associates
EBT	enhanced bioremediation treatment
EICP	extracted ion current profile
ELAP	Environmental Laboratory Accreditation Program
ET&D	excavation, transportation, and disposal
FFA	Federal Facilities Agreement
FFS	Focused Feasibility Study
ft	feet/foot
FSVE	Fluvial Soil Vapor Extraction
FTL	Field Team Leader
GC/MS	gas chromatography/mass spectrometry
GPS	global positioning system
HAZWOPER	Hazardous Waste Operations and Emergency Response
HCl	hydrochloric acid
HDR	HDR, Inc.
HNO_3	nitric acid
HSWA	Hazardous and Solid Waste Amendment
IAQ	Intermediate Aquifer

ICAL	initial calibration
ICB	initial calibration blank
ICS	interference check solutions
ICV	initial calibration verification
IDW	investigative derived waste
IRACR	Interim Remedial Action Completion Report
IS	internal standard
IW	injection well
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LDR	linear dynamic range
LIMS	Laboratory Information Management System
LLICV	low-level calibration check standard
LOD	limit of detection
LOQ	limit of quantitation
LTM	long-term monitoring
LUC	land use control
MAQ	Memphis Aquifer
MCL	maximum contaminant level
MD	matrix duplicate
MDL	method detection limit
mg/L	milligrams per liter
MI	Main Installation
MIP	membrane interface probe
mL	milliliter
MLGW	Memphis Light Gas & Water
MNA	monitored natural attenuation
MS	matrix spike
MSA	method of standard additions
MSD	matrix spike duplicate
ORP	oxidation-reduction potential
OSHA	Occupational Safety and Health Administration
OU	operable unit
PCB	polychlorinated biphenyl
PCE	tetrachloroethene
PCP	pentachlorophenol
PDS	post-digestion spike
PID	photo-ionization detector
PM	Project Manager
POC	point of contact
ppbv	parts per billion by volume
PRB	permeable reactive barrier
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
QSM	Quality Systems Manual

RA	remedial action
RAO	remedial action objective
RCRA	Resource Conservation and Recovery Act
RF	response factor
RL	reporting limit
ROD	Record of Decision
RPD	relative percent difference
RPM	Remedial Project Manager
RRT	relative retention time
RT	retention time
SOP	Standard Operating Procedure
SPCC	system performance check compound
SRI	Supplemental Remedial Investigation
SVE	soil vapor extraction
SVOC	semi-volatile organic compound
SWMU	Solid Waste Management Unit
TC	target concentration
TCL	Target Compound List
TCE	trichloroethene
TDEC	Tennessee Department of Environment & Conservation
TeCA	1,1,2,2-tetrachloroethane
TM	Technical Manager
TSA	technical systems audit
TTA	target treatment area
UC	Upper Claiborne
UFP-QAPP	Uniform Federal Policy – Quality Assurance Project Plan
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
VC	vinyl chloride
VMP	vapor monitoring point
VOC	volatile organic compound
ZVI	zero valent iron
µg/L	micrograms per liter
ºC	degrees Celsius

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QAPP Worksheet #1 & 2: Title and Approval Page
(UFP-QAPP Manual Section 2.1)
(EPA 2106-G-05 Section 2.2.1)

Project Name	Environmental Restoration Support, Former Defense Depot Memphis, Tennessee (DDMT)
Site Location	Memphis, Shelby County, Tennessee
Contract Number	W90FYQ-09-D-0005
Task Order	CK04

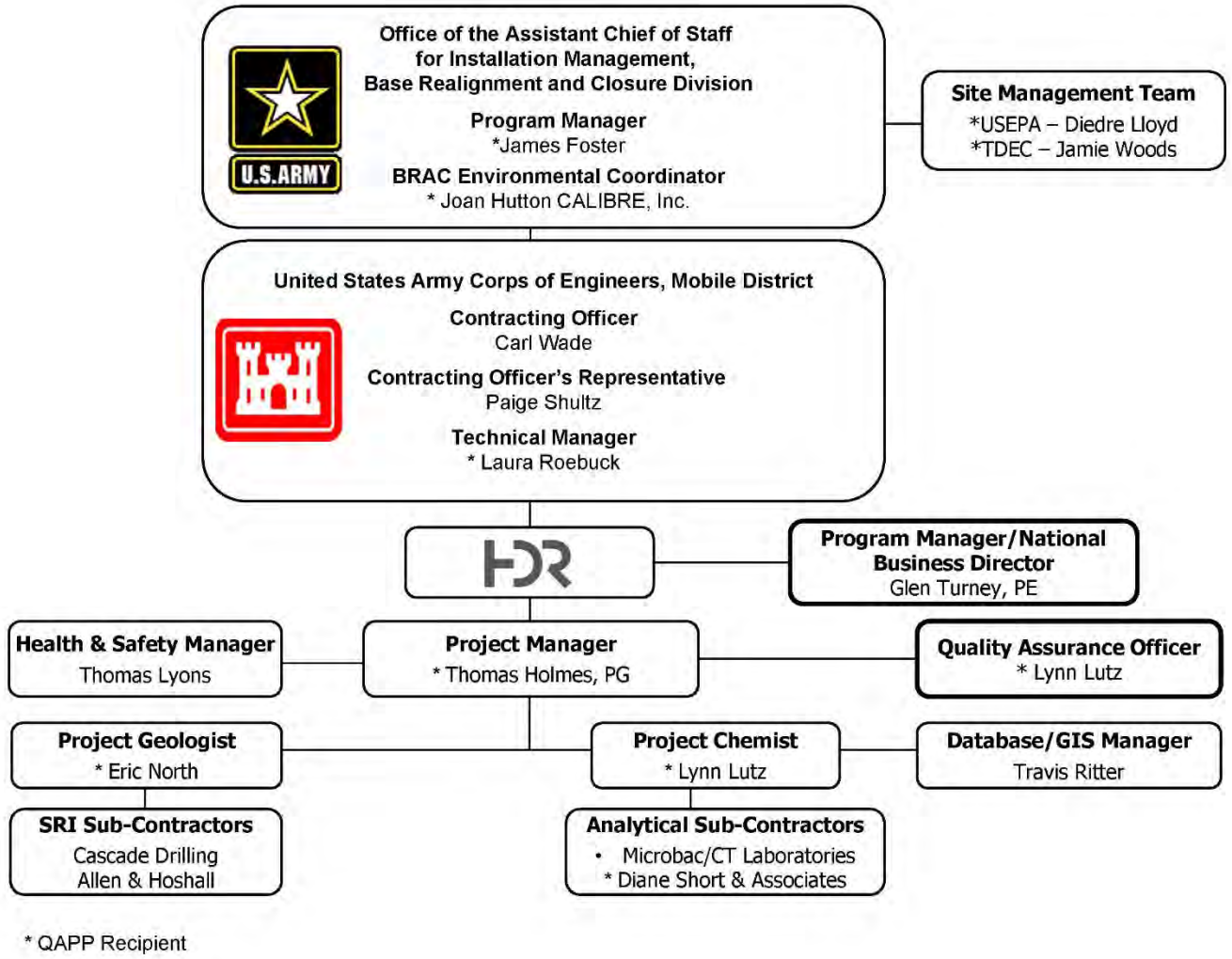
Approvals:

Role	Title	Organization	Name
Lead Organization	Program Manager	United States Army, Assistant Chief of Staff for Installation Management, Base Realignment and Closure (BRAC) Division	James Foster
Lead Organization	BRAC Environmental Coordinator (BEC)	CALIBRE, Inc.	Joan Hutton
Stakeholder Agency	Contracting Officer's Representative (COR)	United States Army Corps of Engineers (USACE), Mobile District	Paige Shultz
Stakeholder Agency	Technical Manager (TM)	USACE - Mobile	Laura Roebuck
Federal Regulatory Agency	Remedial Project Manager (RPM)	United States Environmental Protection Agency (USEPA), Region 4	Diedre Lloyd
State Regulatory Agency	RPM	Tennessee Department of Environment & Conservation (TDEC)	Jamie Woods
Plan Preparation and Implementation	Project Manager (PM)	HDR	Tom Holmes
Plan Preparation and Implementation	Quality Assurance (QA) Officer / Project Chemist	HDR	Lynn Lutz
Plan Preparation and Implementation	Project Geologist	HDR	Eric North

Previous Plans and Reports Relevant to Project:

Title	Date	Author
Record of Decision for Interim Remedial Action of the Groundwater at Dunn Field (OU-1) at the Defense Distribution Depot Memphis. Prepared for U.S. Army Corps of Engineers, Huntsville Division.	April 1996	CH2M HILL
Main Installation Record of Decision. Prepared for the U.S. Army Engineering and Support Center, Huntsville, Alabama.	February 2001	CH2M HILL
Dunn Field Record of Decision. Prepared for U.S. Army Corps of Engineers, Huntsville Division.	March 2004	CH2M HILL
Dunn Field Disposal Sites Remedial Action Completion Report, Revision 1. Prepared for U.S. Air Force Center for Environmental Excellence.	July 2006	MACTEC
Dunn Field Record of Decision Amendment, Revision 3. Prepared for Air Force Center for Engineering and the Environment.	January 2009	e^2M
Source Areas Interim Remedial Action Completion Report, Revision 1. Prepared for Air Force Center for Engineering and the Environment.	September 2009	HDR|e^2M
Main Installation Interim Remedial Action Completion Report, Revision 1. Prepared for Air Force Center for Engineering and the Environment.	February 2010	HDR|e^2M
Preliminary Close Out Report, Defense Depot Memphis Tennessee.	February 2010	USEPA
Dunn Field Interim Remedial Action 2009 Operations and Closure Report. Prepared for Air Force Center for Engineering and the Environment.	December 2010	HDR
Off Depot Groundwater Interim Remedial Action Completion Report, Revision 1. Prepared for Air Force Center for Engineering and the Environment.	July 2011	HDR
Third Five-Year Review, Revision 1. Prepared for the United States Army Corps of Engineers, Tulsa District.	November 2012	HDR
Remedial Action Operations and Long-Term Monitoring Quality Assurance Project Plan, Revision 2. Prepared for the United States Army Corps of Engineers, Tulsa District.	March 2016	HDR
Off Depot Air Sparge-Soil Vapor Extraction System Annual Operations Report, Year Five, Revision 1. Prepared for the United States Army Corps of Engineers, Tulsa District.	August 2016	HDR
Supplemental Remedial Investigation Phase 2 Work Plan, Revision 1. Prepared for the United States Army Corps of Engineers, Mobile District.	August 2016	HDR
Annual Long-Term Monitoring Report-2016, Revision 0. Prepared for the United States Army Corps of Engineers, Mobile District	April 2017	HDR

QAPP Worksheet #3 & 5: Project Organization and QAPP Distribution
(UFP-QAPP Manual Section 2.3 and 2.4)
(EPA 2106-G-05 Section 2.2.3 and 2.2.4)



* QAPP Recipient

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Laura Roebuck	USACE-SAM	TM	laura.w.roebuck@usace.army.mil	251-690-3480	
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Jamie Woods	TDEC	RPM	jamie.woods@tn.gov	901-371-3041	
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QAPP Worksheet #4, 7 & 8: Personnel Qualifications and Sign-off Sheet
(UFP-QAPP Manual Sections 2.3.2 – 2.3.4)
(EPA 2106-G-05 Section 2.2.1 and 2.2.7)

ORGANIZATION: HDR

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Tom Holmes	PM	MS Geophysics 40 years	Registered Professional Geologist, Georgia, United States	
Lynn Lutz	QA Officer/ Project Chemist	BA Chemistry 34 years	Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER), 40-hour; First Aid/CPR certification	
Eric North	Project Geologist	BS Geology 18 years	Registered Professional Geologist, Texas, OSHA HAZWOPER 40-Hour trained, 8-Hour OSHA Site Supervisor trained, CPR/first aid certification, 10 Hour OSHA Construction Safety Certification, Department of Transportation Packaging and Shipping Certification, Excavation and Trenching Competent Person Certification	
Travis Ritter	Project DB/ GIS Manager	MS Environmental Science 16 years	OSHA HAZWOPER, 40-hour; OSHA 510 Construction Industry Health and Safety	

ORGANIZATION: Microbac

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Michelle Taylor	Laboratory PM	-- (3 years college) 6 years	--	

ORGANIZATION: CTL

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Eric Korthals	Laboratory PM	BS/MS Biology 33 years	--	

ORGANIZATION: ALS Environmental

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Kate Kaneko	Laboratory PM	BA Chemistry 28 years	Member, American Chemical Society	

ORGANIZATION: DSA

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Diane Short	Data Validation PM	MS Chemistry/ Molecular Genetics 42 years	Provisional Auditor ISO 9000/ 2000 and ISO 14001	

**Signatures indicate personnel have read and agree to implement this Quality Assurance Project Plan (QAPP) as written.*

QAPP Worksheet #6: Communication Pathways
(UFP-QAPP Manual Section 2.4.2)
(EPA 2106-G-05 Section 2.2.4)

Communication Driver	Responsible Entity	Name	Contact Information	Procedure (timing, pathway, etc.)
Contract Execution/ Document Review	USACE TM	Laura Roebuck	251-690-3480	Email/verbal communication with HDR PM. Quality assurance (QA) supervision for contract activities
Regulatory Interface	BEC	Joan Hutton	770-317-4323	Communicates with USEPA/TDEC as needed and submits project documents for regulatory review. Monitors compliance with the Federal Facilities Agreement (FFA) (USEPA, 1995).
USEPA Oversight	USEPA RPM	Diedre Lloyd	404-562-8855	Reviews and comments on project documents, participation in monthly Site Management Team calls to discuss recent project activities, document review and project schedule, upcoming documents and issue resolution.
TDEC Oversight	TDEC RPM	Jamie Woods	901-371-3041	Reviews and comments on project documents, participation in monthly Site Management Team calls to discuss recent project activities, document review and project schedule, upcoming documents and issue resolution.
Technical Direction	BEC	Joan Hutton	770-317-4323	Reviews project documents and represents the BRAC PM.
Manage all Task Order activities	HDR PM	Tom Holmes	404-295-3279	Monthly progress reports provided to the USACE TM. Notify USACE TM and BEC of field-related problems by phone or email by close of business the day of the event if possible and no later than noon Central Time the following day.
Field Decisions	HDR PM	Tom Holmes	404-295-3279	Notify BEC of planned changes to field activities due to site conditions or other factors for discussion with USEPA and TDEC RPMs and regulatory buy-in.
Manage Field Tasks	HDR Project Geologist	Eric North	512-775-7848	Supervises HDR field activities. Communicates with HDR PM and Project Chemist. Provides daily quality control reports (DQCRs) and notification of any work problems to the HDR PM.
QAPP changes in the field	HDR QA Officer	Lynn Lutz	303-754-4266	Manage and implement in-field QAPP changes. Notify PM of QAPP changes.
Reporting Lab Data Quality Issues	Laboratory PM	M. Taylor-Microbac K. Kaneko-ALS	740-373-4071 805-526-7161*2089	Notifies the HDR Project Chemist regarding laboratory data quality issues including corrective actions (CAs) and data usability.
Field CAs	HDR Project Geologist/Field Team Leader (FTL)	Eric North	512-775-7848	CAs will be issued in writing by the FTL to the HDR PM for review and submittal to USACE TM and BEC.

Communication Driver	Responsible Entity	Name	Contact Information	Procedure (timing, pathway, etc.)
Analytical CAs	HDR Project Chemist	Lynn Lutz	303-754-4266	Coordinates laboratory analyses, reviews deliverables, determines the need for CA on analytical issues and notifies the HDR PM. Provides the data validation report and releases data to the HDR PM.
Stop Work Authority	All Site Workers	-	-	All site workers can issue a stop work order for issues that present immediate and imminent danger. The HDR PM and Health and Safety Officer will be consulted verbally after the Stop Work and then with a follow-up report per the Site Safety and Health Plan.
QAPP Changes	HDR QA Officer / HDR PM	Lynn Lutz/ Tom Holmes	303-754-4266 404-395-3279	Manage and implement QAPP changes. Provide revisions to all QAPP recipients via email and hard copy, as applicable.

QAPP Worksheet #9: Project Planning Session Summary
(UFP-QAPP Manual Section 2.5.1)
(EPA 2106-G-05 Section 2.2.5)

No planning sessions were held based on the status of environmental restoration at DDMT.

The selected remedies for DDMT were described in the Main Installation Record of Decision (CH2MHILL, 2001), the Dunn Field Record of Decision (CH2MHILL, 2004) and the Dunn Field Record of Decision Amendment (e^2M, 2009a). The contaminants of concern are metals, petroleum hydrocarbons and volatile organic compounds in soil and chlorinated volatile organic compounds in groundwater.

All selected remedies have been implemented. Construction, operation and performance monitoring for the remedies were documented in Interim Remedial Action Completion Reports (IRACRs), which were reviewed and approved by the USEPA and TDEC.

The implemented remedies have either met cleanup standards or are making progress toward the standards, except for groundwater contamination on the Main Installation (MI). Enhanced bioremediation was implemented on the MI in 2006 to 2009 and 2012 to 2014. While concentrations of groundwater contaminants were reduced, the reductions were not sufficient to meet the cleanup standards. A Supplemental Remedial Investigation (SRI) is currently being performed, and a Focused Feasibility Study (FFS) will be performed upon completion of the Investigation to develop a remedial strategy to achieve cleanup standards throughout the MI. The supplemental investigation includes document review to examine the basis for the selected remedy and field investigation to improve the site hydrogeological model and delineation of contaminant plumes and to evaluate potential off-site impacts to groundwater. SRI Phase 1 was completed in 2015 and Phase 2 in April 2017; the report for Phases 1 and 2 is in preparation. Further investigation, SRI Phases 3 and 4, is planned in late 2017 and 2018 to include installation of additional monitoring wells, an update to the baseline human health risk assessment for groundwater impacts, additional groundwater modeling and a vapor intrusion study. All activities are being performed with review and concurrence from USEPA and TDEC. Further remedial action (RA) will be conducted after the FFS is completed and the selected remedy has been confirmed or revised.

This generic QAPP has been prepared for guidance of sampling and analysis for RA monitoring, long-term monitoring of groundwater and investigations of soil, soil vapor and groundwater. Project-specific QAPPs will be prepared for all Site Investigations.

QAPP Worksheet #10: Conceptual Site Model
(UFP-QAPP Manual Section 2.5.2)
(EPA 2106-G-05 Section 2.2.5)

Background Information

DDMT is located in southeastern Memphis, Shelby County, Tennessee approximately 5 miles east of the Mississippi River and just northeast of Interstate 240 (Figure 1). DDMT originated as a military facility in the early 1940s to provide stock control, material storage, and maintenance services for the U.S. Army. The facility covered approximately 632 acres consisting of the MI and Dunn Field. In 1995, DDMT was placed on the list of Department of Defense (DoD) facilities to be closed under BRAC. Storage and distribution activities continued until DDMT closed in September 1997.

In 1990, USEPA Region 4 and TDEC issued the Depot a Resource Conservation and Recovery Act (RCRA) Part B permit for the storage of hazardous waste (No. TN 4210020570). The Hazardous and Solid Waste Amendment (HSWA) portion of the permit issued by USEPA included requirements for the identification and, if necessary, CA of Solid Waste Management Units (SWMUs) and Areas of Concern (AOCs). The RCRA Part B permit for hazardous waste storage was terminated in October 1998 because the storage unit was not constructed. The HSWA portion of the permit for CA was terminated in January 2005, with all CA activities directed to be performed under Comprehensive Environmental Response, Compensation and Liability Act (CERCLA).

Subsequent to issuing the RCRA permit, USEPA prepared a final Hazard Ranking System Scoring Package for the facility. In October 1992, USEPA added the Depot to the National Priorities List (57 Federal Register 47180 No. 199). In March 1995, USEPA, TDEC, and the Depot entered into the FFA (USEPA, 1995) under CERCLA, Section 120, and RCRA, Sections 3008(h) and 3004(u) and (v). The FFA outlines the process for investigation and cleanup of the Depot sites under CERCLA. The parties agreed that investigation and cleanup of releases from the sites (including formerly identified SWMUs/AOCs) would satisfy any RCRA CA obligation.

During FFA development, the Depot was divided into four Operable Units (OUs): Dunn Field, OU 1; Southwest Quadrant MI, OU 2; Southeastern Watershed and Golf Course, OU 3; and North-Central Area MI, OU 4.

Topography

DDMT is located in the Gulf Coastal Plain approximately three miles east of the bluffs at the edge of the Mississippi Alluvial Plain. Ground surface at the MI is nearly level with elevations generally from 290 to 305 feet (ft); the highest point is at 312 ft along Dunn Avenue near the northwest MI and the lowest point is at 267 ft below the earthen dam for Lake Danielson on the golf course in the southeast MI.

There are no naturally flowing streams or creeks on DDMT. Site drainage occurs by overland flow via swales, ditches, concrete-lined channels, and a storm drainage system, which directs storm water into a series of storm drains for transport to discharge points around the perimeter. DDMT is generally level with or above surrounding terrain, so it receives little runoff from adjacent areas.

Geology and Hydrogeology

The geologic units of interest at DDMT are (from youngest to oldest): loess, including surface soil; fluvial deposits; Jackson Formation/Upper Claiborne Group (Jackson/Upper Claiborne); and Memphis Sand.

The loess consists of wind-blown and deposited brown to reddish-brown, low-plasticity clayey silt to silty clay. The loess deposits are about 20 to 30 ft thick and are continuous throughout the DDMT.

The fluvial (terrace) deposits at DDMT consist of two general layers. The upper layer is silty, sandy clay to clayey sand and ranges from about 0 to 30 ft thick. The lower layer is composed of interlayered sand, sandy gravel, and gravelly sand, and ranges from 30 to 100 ft thick. The uppermost aquifer is the unconfined Fluvial Aquifer, consisting of saturated sands and gravelly sands in the lower portion of the deposits. The saturated thickness ranges from 0 ft (dry) to approximately 70 ft, and is controlled by the uppermost clay configuration in the Upper Claiborne. The groundwater in the Fluvial Aquifer is not a drinking water source for area residents; however, the current Tennessee groundwater classification at DDMT is General Use (TDEC Chapter 1200-04-03).

The Jackson/Upper Claiborne forms the upper confining unit for the Memphis Aquifer (MAQ) on a regional basis and separates the Fluvial Aquifer from the MAQ at DDMT. The Upper Claiborne Group includes the Cockfield and Cook Formations, and the individual formations of the Jackson/Upper Claiborne consist of clays, silts, and sands deposited in lenses or individual beds that are not areally extensive. The Jackson Formation is reported to be absent in the area of DDMT. The Cockfield Formation consists of inter-fingering fine sand, silt, clay, and local lenses of lignite. The Cook Mountain Formation consists primarily of clay with varying amounts of fine sand and is reported to be the most persistent clay layer in the Jackson/Upper Claiborne confining unit. The Intermediate Aquifer (IAQ) is locally developed in the Upper Claiborne sands.

The Memphis Sand primarily consists of thick bedded, white to brown or gray, very fine grained to gravelly, partly argillaceous and micaceous sand. The Memphis Sand ranges from 500 to 890 ft in thickness, and begins at a depth below ground surface (bgs) of approximately 120 to 300 ft. The MAQ is a regional deep, confined aquifer and is the primary source of water for the City of Memphis. Memphis Light Gas & Water (MLGW) extracts groundwater from several well fields in the Memphis area, which has created a regional cone of depression in the potentiometric surface, with steeper local cones of depression at each well field. The Allen Well Field is the closest well field to DDMT and is located approximately 2 miles west of Dunn Field.

Hydraulic connections (windows) between the Fluvial Aquifer and the MAQ are present in the Memphis area where the Jackson/Upper Claiborne confining unit is thin or absent. Downward leakage from the Fluvial Aquifer is also widespread; areas where the confining unit is thin or absent and in the vicinity of MLGW well fields are particularly susceptible to leakage. A window has been identified in the northwestern MI. Sands in the fluvial deposits and in the Upper Claiborne were observed to be in contact at several well borings located in the central MI and act as a single water table aquifer in that area. The Upper Claiborne sand continues into the window, increases in thickness to the northwest and provides a connection to the Memphis Sand.

Site Contaminants

The MI contained approximately 567 acres with open storage areas, warehouses, former military family housing, and outdoor recreational areas. Types of past activities that could result in the presence of hazardous materials in environmental media at the MI include repackaging hazardous substance for storage or shipment, pesticide application, painting and sandblasting, vehicle maintenance, and hazardous material handling/storage. Other historical activities in open and enclosed storage areas included storing transformers with polychlorinated biphenyls (PCBs), storing and using pesticides/herbicides, and treating wood products with pentachlorophenol (PCP). These industrial activities resulted in the presence of metals, pesticides, and other less frequently detected chemicals in surface soil, surface water, and sediment, and chlorinated volatile organic compounds (CVOCs) in groundwater at the MI.

Dunn Field, which is located across Dunn Avenue from the north-northwest portion of the MI, contained approximately 65 acres and includes former mineral storage and waste disposal areas. Historical records indicate that chemical warfare material (CWM), chlorinated lime, super tropical bleach, and calcium hypochlorite, food stocks, paints/thinners, petroleum/oil/lubricants, acids, herbicides, mixed chemicals, and medical waste were reportedly destroyed or buried in pits and trenches at the Dunn Field disposal sites.

Known or suspected contaminants or classes of contaminants;

Soil contamination at the MI was only found in near-surface soils and consisted primarily of metals, PCBs, semi-volatile organic compounds (SVOCs), and a pesticide, dieldrin. PCP-contaminated soils in the wood treatment area were remediated prior to the final remedial investigation for the MI. Groundwater contaminants were limited to CVOCs primarily tetrachloroethene (PCE) and trichloroethene (TCE); carbon tetrachloride (CT) and chloroform (CF) were in the southeast MI, in addition to PCE and TCE . CVOCs were not detected at high concentrations in soil samples on the MI.

Soil contamination at Dunn Field was generally limited to CVOCs, primarily 1,1,2,2-tetrachloroethane (TeCA); TCE; PCE; 1,2-dichloroethane (DCA); total 1,2-dichloroethene (1,2-DCE); CT; CF; and vinyl chloride (VC). The CVOCs detected in soil samples were also detected most frequently in groundwater sampling events. TCE and TeCA were detected at the highest concentrations in soil and groundwater samples.

Primary release mechanism;

Release of contaminants to soil on the MI was associated with site operations and maintenance, and the release of CVOCs to groundwater is believed to be due to multiple, small spills and leaks.

Release of contamination to soil and groundwater on Dunn Field was due to waste disposal activities.

Secondary contaminant migration;

Secondary contaminant migration was limited to CVOCs on the MI and Dunn Field. Following release through spills or waste disposal, contaminants were held in the loess because of limited

permeability and adsorption to clay and organic matter in the fine-grained soils. CVOCs migrated into the fluvial soils beneath the loess with infiltration of rainwater and then to groundwater.

Removal and Remedial Actions

Main Installation

Pre-ROD Removal Actions

The following actions were taken on the MI prior to the ROD. The locations are shown on Figure 2.

- Approximately 602 cubic yards (CY) of surface and subsurface soil contaminated by PCP was removed from the dip vat area (Building 737) in 1985.
- Hazardous materials and petroleum/oil/lubricants from damaged drums were reclaimed and repackaged at Building 873 in 1985. Approximately 800 55-gallon drums were recouped in this open storage area and then returned to their original location for storage and distribution.
- Approximately 5,000 tons (3,700 CY) of dieldrin-contaminated surface soil in the Housing Area was removed in 1998. The Housing Area is an exception to the overall, light-industrial land use for the MI and remediation levels met residential standards in this area.
- Approximately 530 tons (400 CY) of surface soil contaminated by PCBs around the cafeteria (Building 274) was removed in 1998.
- Approximately 980 CY of surface and subsurface soil from near Buildings 1084, 1085, 1087, 1088, 1089 and 1090 was removed in 2000 because of elevated levels of metals and polyaromatic hydrocarbons.

Record of Decision

The *Memphis Depot Main Installation Record of Decision* (MI ROD) (CH2M HILL, 2001) was approved in 2001 and contained the following components:

- Excavation, transport and off-site disposal of lead-contaminated surface soil.
- Deed restrictions and land use controls (LUCs).
- Enhanced bioremediation treatment (EBT) of CVOCs in the most contaminated part of the groundwater plume.
- Long-term groundwater monitoring.

The area of lead contamination in soil near Building 949 (approximately 300 CY) was excavated and disposed off-site prior to final execution of the ROD. The action was taken to accommodate the economic redevelopment of the site and noted as a significant change in the ROD.

The MI ROD stated EBT would target the most contaminated areas and untreated parts of the groundwater plume would degrade under natural attenuation; the MI RD used groundwater concentrations of 100 micrograms per liter (µg/L) for PCE and TCE to delineate the areas in the southwest and southeast MI, target treatment area (TTA)-1 and TTA-2, respectively.

Remedial Action and Long-Term Monitoring

EBT was conducted through injection of sodium lactate solution to the fluvial aquifer and performance monitoring in TTA-1 and TTA-2 from August 2006 through March 2009. LUCs were implemented through deed restrictions, zoning regulations, a Notice of Land Use Restrictions

recorded in January 2005, and annual inspections since 2005. The *Main Installation Interim Remedial Action Completion Report, Revision 1* (MI IRACR) (HDR|e^{2}M, 2010), including an 'operating properly and successfully' determination, was approved by USEPA in March 2010. Although EBT did not achieve the goal of reducing concentrations below maximum contaminant levels (MCLs), additional field investigation, groundwater modeling and trend analysis indicated that additional RA was not necessary. Additional monitoring wells installed in the IAQ and the upper portion of the MAQ supported the groundwater model results.

Additional EBT was performed in an expanded area from November 2012 through November 2014 following rebound in CVOC concentrations in in the EBT areas. Wells used for injections and performance monitoring during the additional EBT are shown on Figure 3. The *Main Installation Year Four Enhanced Bioremediation Treatment Report* (HDR 2015) was approved by USEPA and TDEC in May 2015. While CVOC concentrations were reduced during the additional EBT, the RA was not sufficient to meet the remedial action objectives (RAOs) for the MI.

Long-term monitoring (LTM) has been performed at the MI since 2004. MI LTM wells are shown on Figure 4. As noted in the figure legend, the well symbols indicate the aquifer (Fluvial, Intermediate or Memphis) and the wells are color coded by area (Background, B-835, North-Central, South-Central, West-Central, Southeast, TTA-1 North, TTA-1 South, TTA-2 and Window). The *Annual Long-Term Monitoring Report-2016 Revision 0* (HDR, 2017) included the following summary for the MI:

- Fluvial Aquifer groundwater elevation contours on the MI suggest a sink in the south-central MI with leakage to the IAQ. Groundwater flow appears to be onto the MI from all sides with flow off the MI through vertical leakage at the window in the northwest MI and the suggested sink in the south-central MI (Figure 5).
- Groundwater flow in the upper IAQ at the northwestern MI is indicated to be to the north (Figure 6), while flow in the MAQ is to the southwest based on only a few wells (Figure 7). The central section of the Allen Well Field is located to the west-northwest of the MI, which suggests the gradient should be to the west. The northerly gradient in the upper IAQ may be due to discontinuous sand units in that area.
- Primary CVOC concentrations exceeded the MCL in 86 of 138 MI LTM wells in 2016. CVOC concentrations in a number of LTM wells showed no impact from EBT indicating the areal extent of RA will need to be expanded to reduce concentrations below the MCLs throughout the MI. Concentrations of PCE and TCE are most commonly detected above the MCL in MI LTM wells; concentrations and isopleths are shown on Figures 8 and 9 for the Fluvial Aquifer and on Figures 10 and 11 for the IAQ.
- cis-1,2-dichloroethene (cDCE) and VC concentrations and reduced PCE and TCE concentrations indicate reductive dechlorination is still active near some wells in areas where EBT-1 or EBT-2 was conducted. Other wells within the EBT areas have rebounding concentrations of PCE and/or TCE.
- Migration of CVOCs onto the MI will also need to be addressed in planning additional RA. Off-site impacts have been confirmed at TTA-1 North by off-site well MW-269 and are likely at the North-Central plume based on the location of MW-263 at the property boundary. Other areas may be identified through the SRI.

- CVOCs in the Fluvial Aquifer have migrated vertically into the IAQ through the window in the northwestern area of the MI. CVOC concentration remain below the MCL in the two wells screened in the Memphis Sand, MW-254 and MW-255 (Figure 4).

Due to the number of LTM wells exceeding MCLs and CVOC concentrations at wells located outside areas impacted by previous EBT, the Army is re-evaluating the selected remedy for the MI. SRI and FFS are currently being performed to develop a remedial strategy to achieve RAOs throughout the MI. The initial SRI tasks including review of MI documents and regional studies, and the Phase 1 field investigation were completed in 2015. The SRI Phase 1 Summary Report (HDR, 2016) was approved by USEPA and TDEC in April and May 2016. An additional investigation, SRI Phase 2, is ongoing, as noted in Worksheet #9.

Dunn Field

Pre-ROD Removal Actions and Interim Remedial Action

The following actions were taken on Dunn Field prior to the final ROD. The locations are shown on Figure 12.

- The *Record of Decision for Interim Remedial Action of the Groundwater at Dunn Field (OU-1)* (CH2M HILL, 1996) was approved in April 1996 to prevent further contaminant plume migration and reduce contaminant mass in groundwater. Recovery wells in the Fluvial Aquifer were installed along the western and northern boundary of Dunn Field, operated from 1998 to 2009 and removed 918 pounds of total volatile organic compounds (VOCs), including 369 pounds of TCE. The system was shut down due to reduced CVOC concentrations in groundwater following implementation of the Source Areas RA. The final year of IRA groundwater monitoring and closure activities were described in the *Dunn Field Groundwater Interim Remedial Action 2009 Operations and Closure Report* (HDR, 2010).
- A non-time critical removal action was conducted to reduce or eliminate the potential risk posed by CWM wastes at Sites 1, 24-A, and 24-B. The removal action was completed in March 2001 and documented in the *Final Chemical Warfare Materiel Investigation/Removal Action Report* (UXB International, Inc., 2001). Approximately 914 CY of soil contaminated with mustard degradation by-products, and 19 CY of mustard-contaminated soil were excavated, transported, and disposed offsite. Twenty-nine bomb casings were recovered from Site 24-A.
- A non-time critical removal action to address lead contaminated surface soil at a former pistol range in the Northeast Open Area; the action was completed in March 2003 and documented in *Removal Action at Former Pistol Range, Site 60* (Jacobs Federal Programs, 2003). Approximately 930 CY of lead contaminated surface soil were excavated, transported, and disposed off-site at an approved, permitted landfill.

Record of Decision and ROD Amendment

The *Memphis Depot Dunn Field Record of Decision* (Dunn Field ROD) (CH2M HILL, 2004) was approved in April 2004 and contained the following components of the selected remedy:

- Excavation, transportation, and disposal (ET&D) of soil and material within disposal sites
- Soil vapor extraction (SVE) in subsurface soils
- Zero valent iron (ZVI) injection in the most contaminated part of the groundwater plume on Dunn Field, and a permeable reactive barrier (PRB) in the off-site groundwater plume

- Monitored natural attenuation (MNA) and LTM of groundwater
- Deed restrictions and LUCs.

The *Dunn Field Record of Decision Amendment, Revision 3* (ROD Amendment) (e^2M, 2009a) was approved in March 2009. The fundamental change in the ROD Amendment was the use of air sparging with soil vapor extraction (AS/SVE) instead of a PRB for the Off Depot groundwater plume. The ROD Amendment also revised the criteria for extent of the AS/SVE system and clarified the treatment objective. The AS/SVE system was selected to cross the core of the plume near the downgradient end and to reduce the individual CVOC concentrations in groundwater to 50 µg/L or less.

Remedial Action and Long-Term Monitoring

Three RAs were performed to implement the selected remedies for Dunn Field: Disposal Sites RA (ET&D); Source Areas RA (SVE, ZVI injections and LUCs); and Off-Depot Groundwater RA (AS/SVE, MNA, and LTM). The locations are shown on Figure 13.

The Disposal Sites RA included excavation and off-site disposal of soil and waste material from five sites and was completed in 2006. The *Disposal Site Remedial Action Completion Report* (MACTEC, 2006) was approved in August 2006.

The Source Areas RA included SVE in the vadose zone and injection of ZVI in groundwater. The Fluvial SVE (FSVE) in fluvial soils (30 to 70 ft bgs) was operated from July 2007 to July 2012 and removed approximately 4,000 pounds of VOCs. Thermal SVE, or in situ thermal desorption, was performed in the loess (0 to 30 ft bgs) from May to December 2008 and removed approximately 12,500 pounds of VOCs. ZVI injection was not required due to success of SVE in reducing groundwater impacts. Excavation and off-site disposal of soil and waste material in two additional areas were also conducted in the Source Areas RA. The *Source Areas Interim Remedial Action Completion Report, Revision 1* (Source Areas IRACR) (HDR|e^2M 2009) was approved by USEPA and TDEC in November 2009.

The FSVE system was shut down in July 2012 after meeting soil remediation goals. The final year of FSVE operations and monitoring is described in *Dunn Field Source Areas Fluvial Soil Vapor Extraction System Annual Operations Report, Year Five, Revision 0* (HDR, 2012).

The Off Depot RA included installation of an AS/SVE system and implementation of LUCs on Dunn Field. The AS/SVE system with 90 AS points and 12 SVE wells began operation in December 2009. LUCs were implemented through deed restrictions, zoning regulations, a Notice of Land Use Restrictions recorded in June 2009, and annual inspections since 2009. The *Dunn Field Off Depot Groundwater Interim Remedial Action Completion Report, Revision 1* (Off Depot IRACR) (HDR 2011) was approved by USEPA in August 2011 and by TDEC in November 2011.

The AS/SVE system was installed to reduce individual CVOC concentrations in the treatment area below 50 µg/L and to continue operation until the upgradient concentrations of individual CVOCs in the Dunn Field plume do not exceed 50 µg/L. The AS/SVE system has removed approximately 84 pounds of VOCs from startup in December 2009 through December 2015. Since April 2012, only TCE in one LTM well in the AS/SVE area, MW-159, has exceeded 50 µg/L. The impact of the Source Areas and Off Depot RAs on the groundwater plume originating at Dunn Field is shown on Figure 14.

LTM has been performed in the Dunn Field/Off Depot Area since 2010. Dunn Field LTM wells are shown on Figure 15. As noted in the figure legend, the well symbols indicate the aquifer (Fluvial, Intermediate or Memphis) and the wells are color coded by area (Background, DF North, DF West and Off Depot). The *Annual Long-Term Monitoring Report-2016 Revision 0* (HDR, 2017) included the following summary for Dunn Field:

- Fluvial Aquifer groundwater flow on Dunn Field is to the west toward a trough approximately 1,200 ft west of Dunn Field, where flow diverges to the north or south (Figure 5).
- TCE is the most common groundwater contaminant on Dunn Field and the Off Depot area; concentrations and isopleths from October 2016 are shown on Figure 16.
- The Dunn Field North plume along the northern boundary of Dunn Field (Figure 16) is considered to result from a suspected, off-site source(s) upgradient of Dunn Field. Eight wells in the plume exceeded MCLs for PCE, TCE and/or DCE in 2016. A membrane interface probe (MIP) survey was conducted in the northeast corner of Dunn Field in March 2017 to investigate the area as a potential source for elevated CVOC concentrations in groundwater. Preliminary results indicate CVOCs are not present above soil remediation goals and do not represent a source area for groundwater contamination; the report is in preparation.
- Smaller isolated plumes with CVOC concentrations above MCLs or target concentrations (TCs) are located in the Dunn Field West area at MW-87, and in the Off Depot area at MW-144/MW-190 and at MW-159/MW-246.
    - CVOC concentrations at MW-87 in October 2016 slightly exceeded the MCL for TCE (Figure 16) and exceeded TCs for CF, TeCA and TCA. MW-87 is the only Dunn Field West well with increased CVOC concentrations since the FSVE system was shutdown in 2012. The concentrations are considered to result from residual contamination in the vadose zone.
    - In 2016, CVOC concentrations exceeded the TC for TeCA at MW-144, MW-159, MW-190 and MW-246 and the MCL for TCE at MW-144, MW-159 and MW-190 (Figure 16). The exceedances of TCs and MCLs at four wells in 2016 were a decrease from seven wells in 2015. The concentrations are considered to represent residual groundwater contaminant migration from the Dunn Field Source areas. The concentrations are decreasing over time as uncontaminated water flows through the area and residual contaminated water passes through the AS/SVE treatment area.
- Only two wells in the DF West and Off Depot areas had CVOCs at concentrations above the active treatment objective of 50 µg/L, TCE (171 µg/L) at MW-159 and CF (70.1 µg/L) at MW-87.
    - Additional AS wells are planned to reduce CVOC concentrations near MW-159. Five wells will be installed and incorporated in AS/SVE system operations in accordance with the *Off Depot Air Sparge Well Installation Work Plan, Dunn Field* (Trinity, 2016) approved by TDEC and USEPA. Well installation will occur upon completion of an access agreement with MLGW.
    - Based on vadose zone modeling results and TCE only slightly exceeding the MCL, no additional RA is currently planned at MW-87.

Potential Receptors and Exposure Pathways

Exposure pathways for soil contamination are not a concern because soil remediation goals were met for removal and RAs at DDMT and LUCs are in place for residual contaminant concentrations that do not allow unlimited use and unrestricted exposure.

The groundwater in the Fluvial Aquifer is not a drinking water source for area residents; however, the groundwater is classified as General Use by TDEC.

At the MI, groundwater in the Fluvial Aquifer flows on to the site from all sides and discharges through vertical leakage at the window in the northwest MI and the suggested sink in the south-central MI. Groundwater contaminants have been detected above MCLs within the window and could potentially impact the MAQ which provides drinking water for the Memphis area. MLGW wells within the Allen Well Field are located 0.5 to 2 miles from the MI.

There are low concentrations DCE and CF, below MCLs, in IAQ wells at Dunn Field. However, there are no observed gaps in the clay at the base of the Fluvial Aquifer in that area, and the reported CVOCs are not the most common contaminants in the Dunn Field plume.

Potential receptors and exposure pathways for groundwater contamination at DDMT are 1) through impacts to the MAQ affecting residential and commercial/industrial users and 2) vapor intrusion affecting workers at the MI or residents in the Off Depot area. Neither of these exposure pathways are complete. Groundwater contaminants have not been detected above MCLs in the MAQ wells (MW-254 and MW-255) on the MI, which are approximately 0.75 mile from the nearest well (115A) in the Allen Well Field, and there has been no observed vertical migration from Dunn Field or the Off Depot area. Vapor sampling in shallow soils, which was described in the Off Depot IRACR, indicated no impacts to residents in that area. A vapor intrusion study at the MI will be conducted in 2017 to evaluate impacts to commercial workers.

Land Use Considerations

All 567acres on the MI have been transferred through public benefit and economic development conveyances. Approximately 41 acres at Dunn Field have been transferred through a public benefit conveyance and a competitive public sale. The remaining 24 acres along the western and northern side of Dunn Field are still held by the Army and are to be transferred through a competitive public sale when remedial activities are completed.

LUCs implemented for the MI and Dunn Field prevent residential use, except in the former housing area on the MI, and drilling of groundwater supply wells and production or consumptive use of groundwater. The MI is primarily used for warehousing and logistics in the Memphis Depot Business Park and for operations at Barnhart Crane and Rigging. Dunn Field is undeveloped.

Current interpretation of Nature and Extent of Contamination

There is no remaining soil contamination at concentrations requiring RA at DDMT.

Groundwater contamination above MCLs is documented through semiannual sampling in accordance with the LTM plans for the MI and Dunn Field. Although several CVOCs have been

detected above MCLs, the CVOCs detected most frequently above MCLs are PCE and TCE at the MI and TCE at Dunn Field. Recent maps from the October 2016 LTM event showing concentrations at individual wells and isopleths, and groundwater elevation contours are presented on Figures 8 to 11 and 16.

Data Gaps and Uncertainties and Remedial Actions

MI

There are several individual plumes with PCE, TCE and/or CT as the primary contaminants; cDCE, VC and CF are also present, as either source contaminants or products of reductive dechlorination from EBT. The following data gaps were identified in the SRI Phase 1 Summary Report: (HDR, 2016):

- The upgradient extent and potential source areas have not been determined for all plumes.
- The influence of on-site and off-site sources on groundwater has not been defined. Migration of groundwater contaminants from off-site sources have been confirmed in one area (TTA-1N) and is suspected in another area (North-Central); a potential new plume is indicated by analytical results at MW-270.
- The groundwater flow in the Fluvial Aquifer and the potential for vertical migration through a suspected sink have not been clearly established.

These data gaps need to be addressed to complete a re-evaluation of the selected remedy. Additional phases of the SRI are planned and will be conducted in accordance with site-specific work plans.

Dunn Field

The selected remedy components for Dunn Field have made significant progress in reducing CVOC concentrations in groundwater within the plume originating from Dunn Field. Additional AS wells to be installed in 2017 are expected to result in the treatment objective being achieved within approximately one year following their inclusion in AS/SVE operations.

The suspected, off-site source(s) of CVOCs in monitoring wells upgradient of the northeast corner of Dunn Field has not been identified. Continued plume migration on to Dunn Field could prevent cleanup objectives from being achieved.

A MIP survey was conducted in the northeast corner of Dunn Field to evaluate the potential for on-site soil contamination impacting groundwater in that area. As stated above, preliminary results of the MIP survey indicate CVOCs are not present above soil remediation goals.

QAPP Worksheet #11: Project Data Quality Objectives
(UFP-QAPP Manual Section 2.6.1)
(EPA 2106-G-05 Section 2.2.6)

The Data Quality Objectives (DQOs) were developed using a systematic planning process. The DQOs document the decisions that need to be made and the level of data quality needed to ensure that those decisions are based on sound scientific data. The DQOs for environmental restoration at DDMT are developed based on USEPA's 7-step DQO process (USEPA, 2006).

Step 1: State the Problem
An updated, generic QAPP is needed for ongoing sampling and analysis during operations monitoring and LTM and to aid development of project-specific QAPPs for planned investigations for soil, groundwater and vapor.

Step 2: Identify the Goal of the Study
Monitor progress toward RAOs through sampling and analysis of groundwater and vapor and provide guidance for additional investigations.

RAOs for the MI and Dunn Field include reducing contaminant concentrations in groundwater below MCLs (and TCs at Dunn Field). Concentrations above or below the MCL will be used to halt, expand or revise RAs. The upgradient and downgradient extent of groundwater plumes must be defined to MCLs to identify potential source areas and receptors.

Step 3: Identify Information Inputs
Data required to answer the study questions include:

- Ground water elevations and VOC concentrations from LTM wells are used to evaluate groundwater flow direction (on to or away from DDMT, within or across lithological units) and areas where MCLs are exceeded and trends in CVOC concentrations. The LTM wells for the MI and Dunn Field are listed on Tables 1 and 2. Sample frequencies are updated in the annual LTM reports; the 2018 sample frequencies are also listed on Tables 1 and 2.
- VOC concentrations in vapor samples from the AS/SVE system are used to evaluate CVOC mass removal from groundwater and to monitor CVOC concentrations in vapor effluent with respect to *de minimus* discharge limits.
- Additional groundwater investigation will provide soil lithology from boring logs, well installation records, ground water elevations and VOC concentrations to address Conceptual Site Model data gaps. The data will be used with LTM data to determine groundwater flow direction (on to or away from DDMT, within or across lithological units), areas where MCLs are exceeded and trends in CVOC concentrations, intermingling of groundwater plumes, potential source areas and receptors.
- Additional vapor intrusion studies will provide soil lithology from boring logs and VOC concentrations in soil vapor, and potentially indoor air, to evaluate impacts from existing soil and groundwater contamination.

Step 4: Define the Boundaries of the Study
The study area is defined by the former DDMT property boundaries for the MI and Dunn Field and the extent of groundwater plumes migrating onto or away from the property (Figures 8 to 11 and 16).

The groundwater contaminants at DDMT are CVOCs, but sample analyses include all Target Compound List (TCL) VOCs to identify degradation products and potential off-site impacts. Groundwater analytical data have been collected at DDMT since 1993 and are maintained in a database for use in trend analyses and other review. LTM will continue until groundwater RAOs are met.

Step 5: Develop the Analytic Approach
Samples will be analyzed for TCL VOCs by Method 8260 for soil and groundwater and by Method TO15 for vapor samples. Parameters of interest are CVOCs: TeCA; TCE; PCE; DCA; cDCE; DCE; CT; CF; and VC. Additional analyses will be identified in project-specific QAPPs as needed.

Groundwater results above the MCL, or TCs at Dunn Field, will be used to determine if continued RA is necessary. LTM will continue until results are below MCLs at locations impacted by past operations at DDMT. Individual well locations can be abandoned when results are below MCLs and the data is not required for evaluation of other wells.

AS/SVE vapor results will be compared to results of samples collected since operations began in 2009 to evaluate effectiveness of remedial operations. Shut down of the AS/SVE system will be determined based on groundwater analyses.

Other analytical approaches may be identified in project-specific QAPPs.

Step 6: Specify Performance or Acceptance Criteria
Performance criteria for analytical data are presented in WS#12. Acceptance criteria will be based on compliance with the task descriptions in WS#17 and the applicable Standard Operating Procedures (SOPs).

Step 7: Develop the Plan for Obtaining Data
The specific tasks to be performed under this site-wide QAPP are groundwater sampling for LTM and vapor sampling for AS/SVE system operations.

LTM Is conducted using 138 wells on or adjacent to the MI and 85 wells on or adjacent to Dunn Field, which includes the Off Depot area. The only existing wells not included in LTM are 30 4-inch diameter injection wells (IWs) installed for EBT; the IWs are within 50 ft of 2-inch diameter monitoring wells included in LTM. LTM wells are assigned biennial, annual or semiannual sample frequencies. Semiannual LTM events include visual well assessments, water level measurements and sample collection per the assigned frequencies.

Vapor sampling includes bi-monthly photo-ionization detector (PID) measurements at SVE wells and the vapor effluent discharge, quarterly PID and vacuum measurements at vapor monitoring points (VMPs), and quarterly laboratory analysis of a vapor sample from the vapor effluent discharge.

The project tasks and schedule are listed on WS#14 and sampling design and rationale are described further on WS#17.

QAPP Worksheet #12: Measurement Performance Criteria
(UFP-QAPP Manual Section 2.6.2)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Groundwater
Analytical Group[1]: VOCs
Concentration Level: Low/Medium

Data Quality Indicators	QC Sample and / or Activity Used to Assess Measurement Performance	Measurement Performance Criteria
Precision-overall	Field duplicate	≤ 20% Relative Percent Difference (RPD)
Precision-overall	Matrix Spike/Matrix Spike Duplicate (MS/MSD)	≤ 20% RPD
Precision-lab	Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	±20% RPD
Accuracy/bias	Surrogate spike recoveries	Quality Systems Manual (QSM) v.5.1 App. C Table 24 (or lab if not in QSM) control limits
Accuracy/bias	MS/MSD recoveries	QSM v.5.1 App. C Table 24 (or lab if not in QSM) control limits
Accuracy/bias	LCS/LCSD recoveries	QSM v.5.1 App. C Table 24 (or lab if not in QSM) control limits
Accuracy/bias-contamination	Method blanks	No analyte detected at ≥1/2 reporting limit (RL) or > 10% sample concentration or regulatory limit
Accuracy/bias-contamination	Equipment blanks, ambient blanks, trip blanks	No analyte detected at ≥ RL

Matrix: Investigation Derived Waste (IDW) Water
Analytical Group[1]: Metals (Cu, Zn)
Concentration Level: Low

Data Quality Indicators	QC Sample and / or Activity Used to Assess Measurement Performance	Measurement Performance Criteria
Precision-overall	Field duplicate RPDs	≤ 20% RPD
Precision-overall	MS/MSD	≤ 20% RPD
Precision-lab	LCS/LCSD	≤ 20% RPD
Precision-lab	Lab duplicate	≤ 20% RPD
Accuracy/bias	MS/MSD recoveries	QSM v.5.1 App. C Table 4 control limits: Copper 86-114% Zinc 87-115%
Accuracy/bias	LCS/LCSD recoveries	QSM v.5.1 App. C Table 4 control limits: Copper 86-114% Zinc 87-115%
Accuracy/bias	Dilution	Five-fold dilution must agree within ± 10% of the original measurement.
Accuracy/bias	Post-digestion spike (PDS)	Recovery within 80-120%.
Accuracy/bias-contamination	Method blanks	No analytes detected > ½ limit of quantitation (LOQ) or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.
Accuracy/bias-contamination	Equipment blanks	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.
Accuracy/bias-contamination	Initial and continuing calibration blank (ICB / CCB)	No analytes detected > limit of detection (LOD).

Matrix: Air (soil vapor)
Analytical Group or Method: VOCs
Concentration Level: Low

Data Quality Indicators	QC Sample and / or Activity Used to Assess Measurement Performance	Measurement Performance Criteria
Precision-overall	Field duplicate RPDs	<30% RPD
Precision-lab	LCS/LCSD RPDs	<30% RPD
Accuracy/bias	Surrogate spike recoveries	70-130% recovery
Accuracy/bias	LCS/LCSD recoveries	QSM v.5.1 App. C Table 43 control limits
Accuracy/bias-contamination	Method blanks	No analyte detected at ≥1/2 RL or > 10% sample concentration or regulatory limit

QAPP Worksheet #13: Secondary Data Uses and Limitations
(UFP-QAPP Manual Section 2.7)
(EPA 2106-G-05 Chapter 3: QAPP Elements for Evaluating Existing Data)

Source	Data Type	Data Uses for Current Project
National Weather Service http://w2.weather.gov/climate/index.php?wfo=meg	Meteorological - Historical monthly rainfall and long-term averages	Review of groundwater level changes
U.S. Army Environmental Hygiene Agency, *Geohydrologic Study, Defense Depot Memphis Tennessee*. January 1983.	Site history, soil borings and sampling activities.	Background information, cross-sections.
O.H. Materials Company. *Summary Report, On-Site Remedial Activities at the Defense Depot Memphis*. February 1986.	RA summary and confirmation sampling	Background information.
A.T. Kearney, Inc. *RCRA Facilities Assessment Report*. 1990	Environmental site descriptions	Background information.
Law Environmental. *Defense Depot Memphis Tennessee Remedial Investigation Final Report*. August 1990.	Site history, soil borings and sampling activities.	Background information, cross-sections.
Woodward-Clyde. *Environmental Baseline Survey Report, Defense Depot Memphis Tennessee*. November 1996.	Site history, soil borings and sampling activities.	Background information, cross-sections.
OHM/IT Remediation Services, Inc. *Post Removal Report: Contaminated Soil Remediation Family Housing Area, Memphis Depot, Tennessee, Volumes I and II.* March 1999.	RA summary and confirmation sampling	Background information.
CH2M HILL. *Memphis Depot Main Installation Remedial Investigation Report - Volumes I through IV.* January 2000.	Site history, soil borings and sampling activities.	Background information, cross-sections.
CH2M HILL. *Memphis Depot – Main Installation Groundwater Feasibility Study Report - Final*. July 2000.	Groundwater modeling and attenuation study	Background information.
Jacobs-Sverdrup Inc. *Remediation Report, Removal Action in Parcels 35 and 28 (Old Paint Shop and Maintenance Area), Former Defense Distribution Depot, Memphis.* September 2000.	RA summary and confirmation sampling	Background information.
CH2M HILL, 2002. *Evaluation of Soil and Groundwater Data Collected from Long-Term Operational Areas (LTOAs), Main Installation, Memphis Depot*. Prepared for U.S Army Engineering and Support Center Huntsville. July 2002.	Site history, soil borings and sampling activities.	Background information, cross-sections.

Source	Data Type	Data Uses for Current Project
Jacobs Engineering Group. *Decontamination Report and Certification for Closure of Permitted Container Storage Facility (Building T-308).* November 2001.	RA summary and confirmation sampling	Background information.
UXB International. *Final Chemical Warfare Material Investigations/ Removal Report*. 2001.	Dunn Field CWM investigation and removal	Background information.
CH2M HILL. *Memphis Depot Dunn Field Remedial Investigation Report* - Volumes I through III. July 2002.	Site history, soil borings and sampling activities.	Background information, cross-sections.
Jacobs Federal Programs. *Remediation Report, Removal Action at Building 949, Former Defense Distribution Depot, Memphis*. February 2002	RA summary and confirmation sampling	Background information.
MACTEC. *Early Implementation of Selected Remedy Interim Remedial Action Completion Report, Revision 1.* September 2005	RA and monitoring results in Off Depot area	Background information.
HDR|e²M. *Main Installation Source Area Evaluation, Revision 0*. March, 2008.	Summary of site history and sampling activities.	Background information.
HDR|e²M. *Main Installation Source Area Investigation, Revision 0*. February, 2009.	Site assessment and sampling activities.	Background information.

QAPP Worksheet #14/16: Project Tasks & Schedule
(UFP-QAPP Manual Section 2.8.2)
(EPA 2106-G-05 Section 2.2.4)

The tasks and schedule for LTM and AS/SVE monitoring and submittal of deliverables for regulatory agency review are shown below. Additional investigations, including SRI Phase 3 and 4 and the MI vapor intrusion study, will be described in project-specific QAPPs.

Activity	Responsible party	Planned start date	Planned completion date	Deliverable(s)	Deliverable due date
Spring LTM Event	HDR	1 April	30 April	Semiannual LTM Summary Report, Rev. 0	60 days after completion
Autumn LTM Event	HDR	1 October	31 October	Annual LTM Report, Rev.0	90 days after completion
Annual AS/SVE Monitoring *Weekly operations* *Bi-monthly PID Readings* *Quarterly VMP readings and effluent sample*	Trinity	1 May	30 April	Semiannual LTM Summary Report, Rev. 0 Annual LTM Report, Rev.0	60 days after completion

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits
(UFP-QAPP Manual Section 2.6.2.3)
(EPA 2106-G-05 Section 2.2.6)

Matrix: Dunn Field/Off Depot Groundwater
Analytical Method: VOCs
Concentration level: Low/Medium

Analyte	Project Action Limit (MCL or TC)	Project Quantitation Limit Goal	Achievable Laboratory Limits[2]	
			MDLs	RLs
	(µg/L)	*(µg/L)*	*(µg/L)*	*(µg/L)*
1,1,2,2-Tetrachloroethane	2.2	1	0.2	0.5
1,1,2-Trichloroethane	1.9	1	0.25	1
1,1-Dichloroethene	7	1	0.5	1
1,2-Dichloroethane	5	1	0.25	0.5
Carbon tetrachloride	3	1	0.25	1
Chloroform	12	1	0.125	0.3
Tetrachloroethene	2.5	1	0.25	1
Trichloroethene	5	1	0.25	1
Vinyl chloride	2	1	0.25	1
cis-1,2-Dichloroethene	35	1	0.25	1
trans-1,2-Dichloroethene	50	1	0.25	1

[1] Analytical method detection limits (MDLs) and RLs are those documented in validated methods.
[2] Achievable MDLs and RLs are limits that an individual laboratory can achieve when performing a specific analytical method.
[3] Project Action Limits for groundwater on Dunn Field are the lowest of the USEPA MCLs (2016) or the groundwater TC from the Dunn Field Record of Decision (March 2004).

Matrix: Main Installation Groundwater
Analytical Method: VOCs
Concentration level: Low/Medium

Analyte	Project Action Limit (MCL)	Project Quantitation Limit Goal	Achievable Laboratory Limits[2]	
			MDLs	RLs
	(µg/L)	*(µg/L)*	*(µg/L)*	*(µg/L)*
Carbon tetrachloride	5	1	0.25	1
Chloroform	80	1	0.125	0.3
Tetrachloroethene	5	1	0.25	1
Trichloroethene	5	1	0.25	1
Vinyl chloride	2	1	0.25	1
cis-1,2-Dichloroethene	70	1	0.25	1
trans-1,2-Dichloroethene	100	1	0.25	1

[1] Analytical MDLs and RLs are those documented in validated methods.
[2] Achievable MDLs and RLs are limits that an individual laboratory can achieve when performing a specific analytical method.
Project Action Limits for groundwater on the MI are the current (2016) USEPA MCLs.

Matrix: Wastewater from Groundwater Sampling and SVE Condensate
Analytical Method: Metals
Concentration level: Low/Medium

Analyte	Project Action Limit	Project Quantitation Limit Goal	Achievable Laboratory Limits[2]	
			MDLs	RLs
	(mg/L)	*(mg/L)*	*(mg/L)*	*(mg/L)*
Copper	0.032	0.02	0.005	0.02
Zinc	0.521	0.02	0.005	0.02

Project Action Limits for wastewater from groundwater sampling and SVE condensate that will be discharged to the storm water drain on Dunn Field are the TDEC Water Pollution Control Division acute water quality criteria for Fish and Aquatic Life protection.

Matrix: Air (soil vapor)
Analytical Method: VOCs
Concentration level: Low/Medium

Analyte	Project Action Limit (Vapor RG)	Project Quantitation Limit Goal	Achievable Laboratory Limits[2]	
			MDLs	RLs
	(ppbv)	*(ppbv)*	*(ppbv)*	*(ppbv)*
1,1,2,2-Tetrachloroethane	0.55	0.4	0.023	0.15
1,1,2-Trichloroethane	2.03	0.5	0.029	0.18
1,1-Dichloroethene	29.03	0.5	0.043	0.25
1,2-Dichloroethane	0.64	0.4	0.047	0.25
Carbon Tetrachloride	14.22	0.5	0.025	0.16
Chloroform	32.63	0.5	0.039	0.20
cis-1,2-Dichloroethene	39.52	0.5	0.045	0.25
Methylene Chloride	2.85	0.5	0.043	0.29
Tetrachloroethene	0.99	0.5	0.021	0.15
trans-1,2-Dichloroethene	133.5	0.5	0.043	0.25
Trichloroethene	2.06	0.5	0.028	0.19
Vinyl Chloride	14.77	0.5	0.074	0.39

[1] Analytical MDLs and RLs are those documented in validated methods.
[2] Achievable MDLs and RLs are limits that an individual laboratory can achieve when performing a specific analytical method.
Project Action Limits are fluvial vapor remediation goals from the Dunn Field Record of Decision (March 2004).

QAPP Worksheet #17: Sampling Design and Rationale
(UFP-QAPP Manual Section 3.1.1)
(EPA 2106-G-05 Section 2.3.1)

The sampling activities will be conducted in accordance with the MI and Dunn Field LTM plans as modified in the latest annual report, the *Off Depot Groundwater Remedial Action Work Plan* (e²M, 2009b) as modified in the latest annual report, and the field SOPs listed in Worksheet 21. The number of samples and the analytical parameters are summarized in Worksheet 18. Samples to be collected consist of groundwater samples, effluent vapor samples and samples of wastewater generated by AS/SVE condensate and LTM purge water.

Physical Boundaries for the Area

The sampling area is defined by the former DDMT property boundaries for the MI and Dunn Field and groundwater plumes migrating onto or away from the property; the extent is approximated by the LTM wells shown on Figures 4 and 15. The area may be expanded as monitoring and additional investigations proceed.

Basis for the Placement and Number of Sample Locations

Groundwater Monitoring

LTM activities consist of monitoring designated LTM wells at the MI and Dunn Field during sampling events in April and October. The monitoring events include well assessments and a water level measurement sweep prior to sampling, sample collection using passive diffusion bags (PDBs) at most wells, and sample collection using low-flow sampling/ bailers where necessary with water quality measurements for well stabilization.

The LTM network consists of 138 wells on the MI and 85 wells on Dunn Field LTM. The MI and Dunn Field LTM wells were divided by aquifer: Fluvial, IAQ/Upper Claiborne (UC) or MAQ. In addition, the wells are grouped by area or plume. The areas for the MI LTM wells are: TTA-1 North, TTA-1 South, TTA-2, West-Central, Building 835, North-Central, South-Central, Southeast MI and Background. The areas for the Dunn LTM wells are: DF North, DF West, Off Depot and Background.

The well locations were installed for previous site investigations or performance monitoring of RAs. The wells are maintained for use in LTM unless the wells are damaged and cannot be repaired or CVOC concentrations are below MCLs and the well is not required for plume delineation. The LTM wells are listed on Tables 1 and 2; the horizontal coordinates are based on Tennessee State Plane coordinates1927 North American Datum and the elevations are based on the North American Vertical Datum of 1988. The locations are shown on Figures 4 and 15.

Sample frequencies are updated in the annual LTM reports, based on the following criteria:

- New wells will be sampled semiannually over two years prior to a frequency being determined.
- Wells with stable concentrations below the MCL in the eight most recent samples will be sampled biennially.

- Wells with stable concentrations approximately two times the MCL or less will be sampled annually.
- All other wells will be sampled semiannually.
- Exceptions to the criteria will be made as appropriate:
    - The MI MAQ wells have not exceeded MCLs but will continue to be sampled annually due to increased concentrations in upgradient wells.
    - The DF North wells exceeding an MCL will continue to be sampled annually until the need for remedial action is determined.
    - Off Depot wells adjacent to the AS/SVE system will be sampled more frequently than required by the new criteria in order to monitor AS/SVE performance.

The sample frequencies for MI and Dunn Field LTM wells in 2018 are semiannual (105), annual (69) and biennial (58).The 2018 sample frequencies for all wells are also listed on Tables 1 and 2; sample frequencies by aquifer are:

- MI
    - 114 Fluvial Aquifer - semiannual (75), annual (22) and biennial (17);
    - 30 IAQ/UC - semiannual (14), annual (11) and biennial (5); and
    - 3 MAQ - semiannual (1) and annual (2).
- Dunn Field
    - 80 Fluvial Aquifer - semiannual (15), annual (32) and biennial (33);
    - 4 IAQ/UC - annual (2) and biennial (2); and
    - 1 MAQ - biennial.

AS/SVE Vapor Samples

Weekly system inspections are performed to monitor and maintain system operations. Flow rates and vacuum pressures in SVE wells and system effluent and the AS flow rate are recorded biweekly when the AS manifold is open and monthly when the manifold is closed. Maintenance is performed per equipment requirement and as required when problems are identified.

AS/SVE system monitoring consists of vacuum measurements at VMPs; PID readings at the system effluent, SVE wells and VMPs; and laboratory analysis of vapor samples from the system effluent. PID readings at the SVE well manifold and the system effluent are collected during months of full AS/SVE operation. PID readings and vacuum measurements at VMPs, and effluent vapor samples are collected quarterly during full operation.

The AS/SVE System is shown on Figure 17.

IDW Water Samples

Investigation-derived waste (IDW) water is generated during groundwater sampling and AS/SVE operations. IDW water is generated during groundwater sampling from well development, purging

prior to sampling and excess water in PDBs, and during AS/SVE operations from condensate from the AS compressor and SVE wells. Groundwater sampling IDW water is collected in 5-gallon buckets with lids and added to a 3,000-gallon storage tank on Dunn Field. Condensate from AS/SVE operations is stored in a 505-gallon polyethylene tank outside the SVE building. Once the exterior tank nears capacity, water is pumped to a trailer-mounted transfer tank and transferred to the storage tank on Dunn Field for analysis prior to discharge.

The IDW water stored on Dunn Field is discharged to a storm water inlet on Dunn Field per agreement with TDEC Division of Water Pollution Control. The discharges have the following concentration limits: copper <0.032 milligrams per liter (mg/L) and zinc <0.521 mg/L. When the storage tank nears capacity, grab samples of wastewater are collected for analysis of copper and zinc; although not required by TDEC, the samples are also analyzed for VOCs. Field quality control samples are not considered necessary for IDW water samples. The wastewater is passed through a bag filter to remove sediment prior to sampling and discharge; during sampling, the filter discharge is captured and returned to the storage tank. Letter reports with analytical results are submitted to TDEC and USEPA following each discharge.

QAPP Worksheet #18: Sampling Locations and Methods/SOP Requirements Table
(UFP-QAPP Manual Section 3.1.1 and 3.1.2)
(EPA 2106-G-05 Section 2.3.1 and 2.3.2)

Sampling Location	Number of Locations	Matrix	Depth (feet)	Analytical Group	Concentration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]	Rationale for Sampling Location
MI Fluvial Aquifer - Background	17	GW	73-141	VOCs	Low	147 field, 15 duplicate	SOP 4	Background
MI Fluvial Aquifer - Plume	97	GW	79-148		Low-moderate			Within Plumes - Offsite Sources
MI IAQ	30	GW	115-208		Low-moderate			Vertical migration
MI MAQ	3	GW	245-306		Low			Vertical migration
DF Fluvial Aquifer - Background	12	GW	26-86	VOCs	Low	85 field, 9 duplicate	SOP 4	Background
DF Fluvial Aquifer - North	15	GW	65-103		Low-moderate			Offsite Plume
DF Fluvial Aquifer - West	19	GW	70-92		Low-moderate			Within plumes
DF Fluvial Aquifer – Off Depot	34	GW	68-111		Low-moderate			Within plumes
DF IAQ	3	GW	88-184		Low			Vertical migration
DF MAQ	1	GW	275		Low			Vertical migration
AS/SVE	1	V	35-76	VOCs	Low	1 field	SOP 5	Effluent

[1] *Specify the appropriate letter or number from the Project Sampling SOP References table (Worksheet #21).*

GW: Groundwater

V: Air (Soil Vapor)

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times
(UFP-QAPP Manual Section 3.1.2.2)
(EPA 2106-G-05 Section 2.3.2)

Laboratory (Name, sample receipt address, point of contact [POC], e-mail, and phone numbers): Microbac Laboratories, 158 Starlite Drive, Marietta, Ohio 45750, Michelle Taylor, michelle.taylor@microbac.com, 800-373-4071
Required accreditations/certifications: DoD Environmental Laboratory Accreditation Program (ELAP)
Back-up Laboratory: CTL, 1230 Lange Court, Baraboo, WI 53913-3109, Eric Korthals, ekorthals@ctlaboratories.com, 608-356-2760
Sample Delivery Method: FedEx next day air

Analyte Group	Matrix	Method/ SOP Reference	Accreditation Expiration Date	Containers (number, size & type per sample)	Preservation Requirements	Maximum Holding Time (preparation/ analysis)	Data Package Turnaround
VOCs	Groundwater	USEPA SW846 5030B/8260B (L-1)	12/31/2018	3 40-milliliter (mL) VOC vials w/ Teflon®-lined septa; no headspace	Chill ≤ 6 degrees Celsius (°C), hydrochloric acid (HCl) to pH<2	14 Days (preserved) 7days (unpreserved)	3 weeks for Level 3; 4 weeks for Level 4
VOCs	IDW Water	USEPA SW846 5030/8260B (L-1)	12/31/2018	3 40-mL VOC vials w/ Teflon®-lined septa; no headspace	Chill ≤ 6°C, HCl to pH<2	14 Days (preserved) 7 days (unpreserved	1 week for Level 2
Metals	IDW Water	USEPA SW846 6010 (L-5)	12/31/2018	250-1000 mL plastic bottle	nitric acid (HNO_3) to pH < 2	180 days	3 weeks for Level 3; 4 weeks for Level 4

Laboratory (Name, sample receipt address, POC, e-mail, and phone numbers): ALS Environmental, 2655 Park Center Drive, Simi Valley, CA 93065, Kate Kaneko, kate.kaneko@alsglobal.com, 805-526-7161*2089
Required accreditations/certifications: DoD ELAP
Back-up Laboratory: NA
Sample Delivery Method: FedEx ground

Analyte Group	Matrix	Method/ SOP Reference	Accreditation Expiration Date	Containers (number, size & type per sample)	Preservation Requirements	Maximum Holding Time (preparation/ analysis)	Data Package Turnaround
VOCs	Air (Soil Vapor)	USEPA TO15 (L-6)	2/28/2018	6-liter Summa™ canister	None	30 days	3 weeks for Level 3; 4 weeks for Level 4

QAPP Worksheet #20: Field QC Summary
(UFP-QAPP Section 3.1.1 and 3.1.2)
(EPA 2106-G-05 Section 2.3.5)

Matrix	Analyte/ Analytical Group	Field Samples	Field Duplicates	Matrix Spikes	Matrix Spike Duplicates	Field Blanks	Equipment Blanks	Trip Blanks	Other	Total # analyses
Groundwater[1]	VOCs	175	18	9	9	0	3	8	0	222
Air[2] (Soil Vapor)	VOCs	4	1	0	0	0	0	0	0	5
IDW Water	VOCs	1	0	0	0	0	0	0	0	1
IDW Water	Metals	1	0	0	0	0	0	0	0	1

Note:

[1)] Groundwater samples are collected at 232 locations assigned semiannual, annual or biennial frequency. The number of samples per semiannual event varies from approximately 139 to 175. Annual and biennial sampling is conducted in April for Dunn Field and in October for the MI.

[2)] Air samples are collected from one location (AS/SVE effluent) quarterly. A duplicate sample is collected annually.

[3)] IDW water samples are collected as necessary, usually annually; no field quality control (QC) samples are collected.

QAPP Worksheet #21: Field SOPs
(UFP-QAPP Manual Section 3.1.2)
(EPA 2106-G-05 Section 2.3.2)

Method/ SOP Reference	Title, Revision Number and Date	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)
SOP 1	General Procedures for Field Personnel, Rev. 1, November 2014	HDR	Log books, first aid kit, personal protective equipment	N
SOP 2	Drilling and Soil Sampling, Rev. 2, February 2015	HDR	Drilling equipment, sample jars, disposable scoops	N
SOP 3	Well Installation, Development and Abandonment, Rev. 1, November 2014	HDR	Well construction materials, cement/bentonite grout, concrete	N
SOP 4	Groundwater Sample Collection, Rev. 1, November 2014	HDR	YSI6920 or similar multi-probe device with flow-through cell, non-dedicated bladder pumps, disposable Teflon bailers, passive diffusion bags	N
SOP 5	Vapor Sample Collection, Rev. 2, April 2015	HDR	Summa canisters, flow controllers	N
SOP 7	Sample Control and Documentation, Rev. 2, April 2015	HDR	Sampling log book, DQCR forms, digital camera, chain-of-custody (CoC) forms	N
SOP 8	Sample Packing and Shipping, Rev. 2, October 2016	HDR	Sample bottles, bubble wrap, ice, zip lock bags, coolers, tape, custody seals	N
SOP 9	Sampling Equipment Decontamination, Rev. 1, November 2014	HDR	American Society for Testing and Materials (ASTM) Type II water (supplied by lab) or distilled water, pesticide-grade methanol, Alconox detergent, brushes	N
SOP 11	Field Sampling Technical Systems Audit, Revision 3, November 2017	HDR	Field audit checklist (Attachment 11-1 to the SOP), pen with waterproof ink, computer with Microsoft Word, other field SOPs	N
L-8	Standard Operating Procedure Sample Receiving and Login, Revision 19, October 2016	Microbac	Thermometers, hood, pH strips, IR temperature guns, disposable pipets, Geiger counter, disposable gloves, laptop or notebook computer equipped for bar code reading	N

Method/ SOP Reference	Title, Revision Number and Date	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)
L-10	Sample Receiving, Acceptance and Log-In, Rev. 16, April 2016	ALS	CoCs, scanner with PDF function, computer with Laboratory Information Management System (LIMS) software	N
Technical Bulletin	GeoExplorer 6000 Series Quick Start Guide	Trimble	Geo-XH	N

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection
(UFP-QAPP Manual Section 3.1.2.4)
(EPA 2106-G-05 Section 2.3.6)

Field Equipment	Activity	SOP Reference	Title or position of responsible person	Frequency	Acceptance Criteria	Corrective Action
GeoExplorer 6000	Field Global Positioning System (GPS) location of sample points	Technical Bulletin	FTL	Per Trimble manual	Meet manufacturer's specifications	Re-try. Send to manufacturer for repair
YSI 650MDS	Water quality measurement (pH, oxygen reduction potential [ORP], dissolved oxygen [DO], conductivity, temperature)	HDR SOP 4	FTL	Daily during use and when readings are inconsistent	Meet manufacturer's specifications	Replace components and re-try. Send to manufacturer for repair
Horiba U-2000	Water quality measurement (pH, ORP, DO, conductivity, temperature, turbidity)	HDR SOP 4	FTL	Daily during use and when readings are inconsistent	Meet manufacturer's specifications	Replace components and re-try. Send to manufacturer for repair
Lamotte 2020e	Water quality measurement (turbidity)	HDR SOP 4	FTL	Daily during use and when readings are inconsistent	Meet manufacturer's specifications	Replace components and re-try. Send to manufacturer for repair
RAE PGM-7600 PID	VOC screening	HDR SOP 4	FTL	Daily during use and when readings are inconsistent	Meet manufacturer's specifications	Re-try. Send to manufacturer for repair
Heron Dipper-T	Water level measurement	HDR SOP 4	FTL	Daily during use	Meet manufacturer's specifications	Re-try. Send to manufacturer for repair
Solinst 101	Water level measurement	HDR SOP 4	FTL	Daily during use	Meet manufacturer's specifications	Re-try. Ssend to manufacturer for repair
Geotech PRO Pumpbox and Geocontrol 2	Groundwater purge and sample	HDR SOP 4	FTL	Daily during use	Meet manufacturer's specifications and project requirements	Send to manufacturer for repair
Thomas TG-180HST	Clear vapor tubing	HDR SOP 5	FTL	Daily during use	Meet manufacturer's specifications and project requirements	Send to manufacturer for repair
Thomas 107CDC20	Vapor purge and sample	HDR SOP 5	FTL	Daily during use	Meet manufacturer's specifications and project requirements	Send to manufacturer for repair

QAPP Worksheet #23: Analytical SOPs
(UFP-QAPP Manual Section 3.2.1)
(EPA 2106-G-05 Section 2.3.4)

SOP #	Title, Revision Number and Date	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	Modified for Project Work? (Y/N)
SOP 4	Groundwater Sample Collection, Revision 1, November 2014	Screening	Field water quality (pH, conductivity, DO, ORP, temperature, turbidity)	Field multimeter (YSI, Horiba, Lamotte))	N
L-1	Analysis of Volatile Organic Analytes by Methods 8260A and 8260B, Revision 25, January 2017 (MSV01)	Definitive	VOCs in Water	Hewlett-Packard [HP] 6890 GC equipped with HP 5973 mass spectrometer HP Enviroquant software	N
L-5	Perkin Elmer Optima 4300 Inductively Coupled Plasma Atomic Emission Spectroscopy SW846 6010 / EPA 200.7, Revision 9, September 2016 (ME600G)	Definitive	Metals in Water	Perkin Elmer Optima 4300 equipped with a CETAC ASXpress-520 Autosampler Argon gas supply (liquid) Dell Pentium 4 computer with Microsoft Windows 2000 Professional and Perkin Elmer WinLab32 ICP Continuous Software Version 4.0.0.0303 ESI Microflow PFA-ST3-84 Nebulizer	N
L-6	Standard Operating Procedure for Determination of Volatile Organic Compounds in air Samples Collected in Specially Prepared Canisters and Gas Collection Bags by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 23, April 2016 (VOA-TO15)	Definitive	VOCs in Air	GC: HP 5890 Series II Plus, HP 6890 Series, HP 6890A Series, Agilent 6890N Series MS: HP 5972 Series, HP 5973 Series, Agilent 5973, Agilent 5973N, Agilent 5973*inert*, Agilent 5975B*inert*, Agilent 5975C*inert* Data System: IBM-compatible PC with Windows 95/98/NT/XP (Microsoft Office EXCEL version 2003 or newer) and Hewlett Packard Chemstation software including EnviroQuant with Extracted Ion Current Profile (EICP), National Institute of Standards and Technology library (2002 version or newer) or equivalent	N

SOP #	Title, Revision Number and Date	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	Modified for Project Work? (Y/N)
L-7	Microwave Digestion – Aqueous, SW846 3015A, Revision 19, December 2016 (ME407)	Definitive	Metals in Water	Mars Xpress microwave unit, must provide programmable power with a minimum of 574W and can be programmed to within ± 10W of required power Beckman GS-6 or equivalent centrifuge Analytical balance, 600 g capacity	N
L-8	Standard Operating Procedure Sample Receiving and Login, Revision 19, October 2016 (LOGIN01)	Not Applicable	All	PDA, Laptop or notebook computer (equipped for bar coding) IR Temperature Guns Thermometers pH paper	N

QAPP Worksheet #24: Analytical Instrument Calibration
(UFP-QAPP Manual Section 3.2.2)
(EPA 2106-G-05 Section 2.3.6)

Instrument	Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/MS for VOCs in Water SW-846 8260B	Mass spectral ion intensities with 4-bromofluoro-benzene (BFB)	Every 12 hours prior to ICAL, ICV or CCV	Mass – Ion Abundance Criteria 50 – 15-40% of mass 95 75 – 30-60% of mass 95 95 – base peak, 100% relative abundance 96 – 5-9% of mass 95 173 – <2% of mass 174 174 – >50% of mass 95 175 – 5-9% of mass 174 176 - >95% and <101% of mass 174 177 - 5-9% of mass 176 (Established criteria in Table 4 of SW-846 8260B)	Retune instrument and repeat BFB check. Flagging criteria are not appropriate.	Analyst	L-1
	Initial multipoint calibration for all analytes (initial calibration [ICAL]) (minimum five standards)	When second source calibration or continuing calibration is out of control or when system conditions have been altered.	1. Average response factor (RF) for System Performance Check Compounds (SPCCs): ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. RSD for RFs for Calibration Check Compounds: ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression– coefficient of determination r2 ≥ 0.99 (6 points shall be used for second order, 7 points shall be used for third order).	Evaluate cause; repeat calibration; or qualify data and discuss in narrative. See SOP section 13.7 for additional CA. Flagging criteria are not appropriate.	Analyst	L-1

Instrument	Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/MS for VOCs in Water SW-846 8260B (continued)	Continuing Calibration verification (CCV)	Once per each 12 hours, prior to sample analysis (criteria for these checks must be met prior to sample analysis)	1. Average Relative Response Factor for SPCCs : ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. %Difference/Drift for all target compounds and surrogates: VOCs and SVOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	1) Evaluate system and take CA. 2) Rerun calibration check. 3) If still out, prepare new calibration curve for any analyte not meeting criteria. 4) Reinject any samples analyzed after criteria were exceeded. 5) Qualify the data. Criteria for these checks must be met prior to sample analysis. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Analyst	L-1
	Retention time (RT) window position establishment for each analyte and surrogate	Once per ICAL	Position shall be set using the midpoint standard of the ICAL curve.	NA	Analyst	L-1
ICP for Metals in Water SW-846 6010	ICAL for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 ≥ 0.99. Minimum one high standard and a calibration blank.	Correct problem, then repeat ICAL. No samples shall be analyzed until ICAL has passed.	Analyst	L-5
	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL. No samples shall be analyzed until calibration has been verified with a second source.	Analyst	L-5

Instrument	Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
ICP for Metals in Water SW-846 6010 (cont'd)	Low-level Calibration Check Standard (LLICV)	Daily.	All reported analytes within ± 20% of true value. Low level calibration check standard should be less than or equal to the LOQ.	Correct problem and repeat ICAL. No samples shall be analyzed without a valid LLICV.	Analyst	L-5
	CCV	After every 10 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	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, take CA(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. Results may not be reported without a valid CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.	Analyst	L-5
GC/MS for VOCs in Air TO-15	Mass spectral ion intensities with BFB	Every 24 hours prior to ICAL, ICV or CCV	<u>Mass – Ion Abundance Criteria</u> 50 – 8-40% of mass 95 75 – 30-66% of mass 95 95 – base peak, 100% relative abundance 96 – 5-9% of mass 95 173 – <2% of mass 174 174 – 50-120% of mass 95 175 – 4-9% of mass 174 176 – 93-101% of mass 174 177 - 5-9% of mass 176 (Established criteria in Table 3 of TO-15)	Retune instrument and repeat BFB check. Flagging criteria are not appropriate.	Analyst	L-6

Instrument	Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/MS for VOCs in Air TO-15 (cont'd.)	Initial multipoint calibration for all analytes (ICAL) (minimum five standards)	ICAL prior to sample analysis	%RSD for all analytes < 30% with at most 2 exceptions up to 40%. Relative retention time (RRT) for each target compound at each calibration level must be within 0.06RRT units of the mean RRT for the compound. Internal Standard (IS): the area response at each calibration level must be within 40% of the mean area response over the ICAL range. RT shift for each of the ISs at each calibration level must be within 20 seconds of the mean RT over the ICAL range for each IS.	Inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other CAs to meet the ICAL technical acceptance criteria. Flagging criteria are not appropriate.	Analyst	L-6
	CCV	Daily, before sample analysis unless ICAL performed on same day and every 24 hours of analysis time	All analytes within ± 30% of expected value.	Inspect the system for problems. It may be necessary to clean the ion source, change the column, or take other CAs to meet the continuing calibration technical acceptance criteria. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Analyst	L-6

[1]*Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).*

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection
(UFP-QAPP Manual Section 3.2.3)
(EPA 2106-G-05 Section 2.3.6)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
GC/MS for VOCs in Water	Clean mass spectrometer, change/check helium, change trap, clip column, change purge and trap ferrules, bake out column	GC/MS	Purge lines, purge flow, trap, ion source, column	Prior to calibration check and/or as necessary	Acceptable ICAL or CCV	Correct problem and repeat ICAL or CCV	Analyst / Supervisor	MSV01 (L-1)
ICP for metals in water	Clean torch and nebulizer when needed.	ICP	Check torch and nebulizer every day.	Daily	Acceptable instrument performance	Fix issues as necessary.	Analyst	ME600E (L-5)
	Change tubing.		Inspect tubing	When it loses pliability and is worn.	Acceptable performance	Fix issues as necessary.	Analyst	
	Drain compressor daily.		Drain compressor daily.	Daily	Acceptable performance	Fix issues as necessary.	Analyst	
	The instruments are under service contracts so that every year a service representative will perform a systems check.		The instruments are under service contracts so that every year a service representative will perform a systems check.	Yearly	Acceptable performance	Fix issues as necessary.	Service representative	
	The water in the recirculator/cooler must be changed yearly.		The water in the recirculator/cooler must be changed yearly.	Yearly	Acceptable performance	Fix issues as necessary.	Analyst	

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
GC/MS for VOCs in Air	Concentrating Trap	GC/MS	NA	Routine maintenance includes periodic solvent cleaning	Acceptable performance	Fix issues as necessary.	Analyst	VOA-TO15 Rev.18 (L-6)
	Column Performance			Monitored by observing both peak shapes and column bleed.	Acceptable performance	Fix issues as necessary.	Analyst	
	Vacuum System			Every six months, including changing the pump oil and checking the molecular sieve in the back-streaming trap.	Acceptable performance	Fix issues as necessary.	Analyst	

[1]*Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).*

QAPP Worksheet #26 & 27: Sample Handling, Custody, and Disposal
(UFP-QAPP Manual Section 3.3)
(EPA 2106-G-05 Section 2.3.3)

Sampling Organization: HDR
Laboratory: Microbac or ALS
Method of sample delivery (shipper/carrier): FedEx
Number of days from reporting until sample disposal: 60

Activity	Organization and title or position of person responsible for the activity	SOP Reference
SAMPLE COLLECTION, PACKAGING, AND SHIPMENT		
Sample Collection	HDR Field Team	HDR SOP 4, HDR SOP 5
Sample Packaging	HDR Field Team	HDR SOP 8
Coordination of Shipment	HDR Field Team and Project Chemist, Laboratory (Microbac, ALS) PM	HDR SOP 8
Type of Shipment/Carrier: Cooler (groundwater in vials/bottles), shipped via FedEx, next morning delivery. Cardboard carton (air sample in Summa™ canister), shipped via FedEx Ground.	HDR Field Team	HDR SOP 8
SAMPLE RECEIPT AND ANALYSIS		
Sample Receipt	Laboratory (Microbac, ALS) sample custodian	Microbac SOP L-8, ALS SOP L-10
Sample Custody and Storage	Laboratory (Microbac, ALS) sample custodian	Microbac SOP L-8, ALS SOP L-10
Sample Preparation	Laboratory (Microbac, ALS) sample preparation chemist or analyst	Microbac SOPs L-1, L-7, ALS SOP L-6
Sample Determinative Analysis	Laboratory (Microbac, ALS) sample analyst	Microbac SOPs L-1, L-5, ALS SOP L-6

Activity	Organization and title or position of person responsible for the activity	SOP Reference
SAMPLE ARCHIVING		
Field Sample Storage (No. of days from sample collection): 60 days from data package report	Laboratory (Microbac, ALS) sample custodian.	Microbac SOP L-8, ALS SOP L-10
Sample Extract/ Digestate Storage (No. of days from extraction/digestion): 60 days from data package report	Laboratory (Microbac, ALS) sample custodian	Microbac SOP L-8, ALS SOP L-10
Biological Sample Storage (No. of days from sample collection): NA	N/A	N/A
SAMPLE DISPOSAL		
Number of Days from Analysis: 60 days from data package report	Laboratory (Microbac, ALS) sample custodian	Microbac SOP L-8, ALS SOP L-10

QAPP Worksheet #28: Analytical Quality Control and Corrective Action
(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)
(EPA 2106-G-05 Section 2.3.5)

Matrix: Groundwater
Analytical Group: VOCs by GC/MS
Analytical Method/SOP: SW-846 8260B / L-1

QC SAMPLE	Number/ Frequency	Method / SOP Acceptance Limits	Corrective Action	Title/ Position Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Second-source calibration verification	One each time a five-point calibration is performed	Compounds within ± 20% expected value	Reanalyze ICV. Upon second failure, repeat ICAL.	Analyst	Accuracy/ Bias	Compounds within ± 20% expected value. Flagging criteria are not appropriate.
Evaluation of RRT	Each sample.	RRT of the analyte within ± 0.06 RRT units of ICAL	Correct problem then reanalyze all samples analyzed since the last RT check. Lab may update RTs based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the lab must take appropriate CAs as required by the method and rerun the ICAL to reestablish the RTs.	Analyst	--	RRT of the analyte within ± 0.06 RRT units of ICAL. Flagging criteria are not appropriate.

QC SAMPLE	Number/ Frequency	Method / SOP Acceptance Limits	Corrective Action	Title/ Position Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
IS –RT and area response checked from daily calibration check	Every field sample, standard, and QC sample.	RT ± 30 seconds and EICP area within -50% to +100% of *the mid-point standard in the ICAL* for each IS compound.	1) Inspect MS and GC for malfunctions. 2) Take appropriate CAs. 3) Reanalyze samples analyzed while system was malfunctioning. 4) If sample exceeds criteria, reanalyze sample. If still out, report both analyses and document CA.	Analyst	Accuracy	RT ± 30 seconds and EICP area within -50% to +100% of *the mid-point standard in the ICAL* for each IS compound. Apply Q-flag to analytes associated with the non-compliant IS.
Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a second source QC check sample	Once per analyst	Analyte-specific limits as per laboratory historical limits.	1) Recalculate results. 2) Locate and fix the source of the problem. 3) Rerun demonstration for those analytes that did not meet criteria.	Analyst	Precision, Accuracy	Analyte-specific limits as per laboratory historical limits.
MDL Study	Once per year, upon any major system change, or quarterly MDL check.	MDLs established as described in 40 Code of Federal Regulations (CFR) Part 136, App. B shall not exceed one-half the RL.	MDLs that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples.	Analyst	Sensitivity	MDLs established as described in 40 CFR Part 136, App. B shall not exceed one-half the RL.
Field duplicate	Sampling: 1 for every 10 field samples Lab: NA	RPD ≤ 20%	1) Review lab QC data to determine if they are in control. 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further CA.	Analyst	Precision-overall	RPD ≤ 20%

QC SAMPLE	Number/ Frequency	Method / SOP Acceptance Limits	Corrective Action	Title/ Position Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MS/MSD	Sampling: 1 pair per every 20 samples Lab: same	In-house control limits (or, if not established, control limits in DoD QSM 5.1 [2017] Table G-4. RPD ≤ 20 %	Qualify data.	Analyst	Precision-overall and accuracy/ bias	In-house control limits (or, if not established, control limits in DoD QSM 5.1 [2017] Table G-4. RPD ≤ 20 % For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.
LCS	One per preparatory batch	In-house control limits (or, if not established, control limits in DoD QSM 5.1 [2017] Table G-4. Up to 3 marginal exceedances if full list of 66 analytes is run.	1) The analytical batch must be reprocessed. 2) Reprep and analyze LCS and affected samples. 3) Qualify the data if CA was unsuccessful or was not performed	Analyst	Precision-lab	In-house control limits (or, if not established, control limits in DoD QSM 5.1 [2017] Table G-4. Up to 3 marginal exceedances if full list of 66 analytes is run. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.
Surrogate spike recoveries	Every sample, spike, standard, and reagent blank	In-house control limits (or, if not established, control limits in DoD QSM 5.1 [2017] Table G-3.	1) Recalculate result, and reanalyze sample if still out. 2) Re-extract and reanalyze sample, if still out. 3) Report both analyses and document in report that steps 1 and 2 were performed. 4) Qualify the data.	Analyst	Accuracy/ bias	In-house control limits (or, if not established, control limits in DoD QSM 5.1 [2017] Table G-3. Apply Q-flag to all associated analytes if acceptance criteria are not met.

QC SAMPLE	Number/ Frequency	Method / SOP Acceptance Limits	Corrective Action	Title/ Position Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method blanks	One per preparatory batch	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL.	1) Take and document appropriate CA 2) Reanalyze all samples processed with a contaminated blank. 3) Qualify the data if the CA was not successful or was not performed.	Analyst	Accuracy/ bias-contamin-ation	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.
Trip blank	Sampling: 1 for each batch of samples shipped to laboratory Lab: NA	No analytes detected at > RL	1) Review lab QC data to determine if there is a laboratory problem. 2) If same compounds are found in field samples at similar concentrations, qualify the data. OR 3) Resample the batch	Analyst	Accuracy/ bias-contamin-ation	No analytes detected at > RL Apply B-flag to all results for the specific analyte(s) in all samples associated with the trip blank.
Ambient blank	Sampling: Collected when samples are collected downwind of possible volatile sources. Lab: NA	No analytes detected at > RL	1) Review lab QC data to determine if there is a laboratory problem. 2) If same compounds are found in field samples at similar concentrations, qualify the data. OR 3) Resample the batch.	Analyst	Accuracy/ bias-contamin-ation	No analytes detected at > RL Apply B-flag to all results for the specific analyte(s) in all samples associated with the ambient blank.

QC SAMPLE	Number/ Frequency	Method / SOP Acceptance Limits	Corrective Action	Title/ Position Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Rinsate blank	Sampling: 1 per day per sampling team per matrix if using non-dedicated equipment Lab: NA	No analytes detected at > RL	Qualify data.	Analyst	Accuracy/ bias-contamin-ation	No analytes detected at > RL Apply B-flag to all results for the specific analyte(s) in all samples associated with the rinsate blank.
Results reported between MDL and RL.	--	--	--	--	--	Apply J-flag to all results between MDL and RL.

Matrix: Groundwater
Analytical Group: Metals by ICP
Analytical Method/SOP: SW-846 6010 / L-5

QC SAMPLE	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Second-source calibration verification	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Analyst	Accuracy/ Bias	No samples shall be analyzed until calibration has been verified with a second source. Flagging is not appropriate.
LLICV	Daily	All reported analytes within ± 20% of true value.	Correct problem and repeat ICAL.	Analyst	Accuracy/ Bias/ Sensitivity	No samples shall be analyzed without a valid LLICV. Low level calibration check standard should be less than or equal to the LOQ. Flagging is not appropriate.
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within ± 10% of true value.	Dilute samples to within the calibration range, or re-establish/ verify the LDR.	Analyst	Accuracy/ Bias/ Sensitivity	Data cannot be reported above the high calibration range without an established/passing high level check standard. Flagging is not appropriate.
Field duplicate	Sampling: 1 for every 10 field samples Lab: NA	RPD ≤ 20%	1) Review lab QC data to determine if they are in control. 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further CA.	Analyst	Precision-overall	RPD ≤ 20%

QC SAMPLE	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
MS	Sampling: One per 20 samples. Lab: One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst	Precision-overall and accuracy/ bias	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative. If MS results are outside the limits, the data shall be evaluated to the source(s) of difference, i.e., matrix effect or analytical error.
MSD or Matrix Duplicate (MD)	Sampling: One per 20 samples (if MSD). Lab: One per preparatory batch (MD).	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst	Precision-overall and accuracy/ bias	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative. The data shall be evaluated to determine the source of difference.
Dilution Test	One per preparatory batch if MS or MSD fails.	Five-fold dilution must agree within ± 10% of the original measurement.	No specific CA, unless required by the project.	Analyst	Precision-overall and accuracy/ bias	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative. Only applicable for samples with concentrations > 50 x LOQ (prior to dilution). Use along with MS/MSD and PDS data to confirm matrix effects.

QC SAMPLE	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
PDS Addition (ICP only)	Perform if MS/MSD fails. One per preparatory batch (using the same sample as used for the MS/MSD if possible).	Recovery within 80-120%.	No specific CA, unless required by the project.	Analyst	Precision-overall and accuracy/ bias	For the specific analyte(s) in the parent sample, apply J flag if acceptance criteria are not met and explain in the case narrative. Criteria applies for samples with concentrations <50 X LOQ prior to dilution.
Method of Standard Additions (MSA)	When dilution test or post digestion spike fails and if required by project.	NA	NA	Analyst	Precision-overall and accuracy/ bias	Document use of MSA in the case narrative.
LCS	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst	Precision-lab	Must contain all reported analytes. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.

QC SAMPLE	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method blanks	One per preparatory batch	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Analyst	Accuracy/ bias-contamin-ation	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
ICB/CCB	Before beginning a sample run, after every 10 field samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Analyst	Accuracy/ bias-contamin-ation	Results may not be reported without a valid calibration blank. For CCB, failures due to carryover may not require an ICAL. Flagging is not appropriate.
Interference Check Solutions (ICS) (also called Spectral Interference Checks)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all nonspiked project analytes <LOD (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	Analyst	Accuracy/ bias-contamin-ation	If CA fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the failed ICS. All analytes must be within the LDR. ICS-AB is not needed if instrument can read negative responses.

QC SAMPLE	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Rinsate blank	Sampling: 1 per day per sampling team per matrix if using non-dedicated equipment Lab: NA	No analytes detected at > RL	Qualify data.	Analyst	Accuracy/ bias-contamin-ation	No analytes detected at > RL Apply B-flag to all results for the specific analyte(s) in all samples associated with the rinsate blank.
Results reported between MDL and RL.	--	--	--	--	--	Apply J-flag to all results between MDL and RL.

Matrix: Air (Soil Vapor)
Analytical Group: VOCs
Analytical Method/SOP: TO-15 / L-6

QC SAMPLE	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Second-source calibration verification	Once per ICAL	All analytes within ± 30% of expected value	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL. Problem must be corrected. Samples may not be analyzed until the calibration has been verified.	Analyst	Accuracy/ Bias	All analytes within ± 30% of expected value Flagging criteria are not appropriate.
ISs	Every field sample, standard, and QC sample.	Area response within ± 40% of the area response for each IS in the most recent valid calibration (CCV or mid-point from the ICAL, whichever is most current). RT within ± 0.33 minutes of the RT for each IS in the most recent valid calibration (CCV or mid-point from the ICAL, whichever is most current).	1) Inspect MS and GC for malfunctions. 2) Take appropriate CAs. 3) Reanalyze samples analyzed while system was malfunctioning. 4) If sample exceeds criteria, reanalyze sample. If still out, report both analyses and document CA.	Analyst	Accuracy	Area response within ± 40% of the area response for each IS in the most recent valid calibration (CCV or mid-point from the ICAL, whichever is most current). RT within ± 0.33 minutes of the RT for each IS in the most recent valid calibration (CCV or mid-point from the ICAL, whichever is most current). Apply Q-flag to analytes associated with the non-compliant IS.

QC SAMPLE	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Method blank (humid zero air)	Immediately after ICV or daily CCV, and whenever a high concentration sample is encountered and carryover is suspected	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected > RL. Blank result must not otherwise affect sample results.	The source of the contamination must be investigated and appropriate corrective measures need to be taken and documented before further sample analysis proceeds. If an analyte in the blank is found to be out of control (i.e., contaminated) and the analyte is also found in associated samples, those sample results should be "flagged" by the lab as possibly contaminated.	Analyst	Accuracy/ bias-contamina-tion	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected > RL. Blank result must not otherwise affect sample results. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.
LCS for all analytes	One LCS per analytical batch	All analytes within ± 30% of expected value	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL. Problem must be corrected. Samples may not be analyzed until the calibration has been verified.	Analyst	Precision-lab	All analytes within ± 30% of expected value Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.

QC SAMPLE	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator	Measurement Performance Criteria
Surrogate spike recoveries	Every sample, spike, standard, and reagent blank	70-130% recovery	1) Recalculate result, and reanalyze sample if still out. 2) Re-extract and reanalyze sample, if still out. 3) Report both analyses and document in report that steps 1 and 2 were performed. 4) Qualify the data.	Analyst	Accuracy/ bias	70-130% recovery Apply Q-flag to all associated analytes if acceptance criteria are not met.
Field duplicate	Sampling: 1 for every 10 field samples Lab: NA	RPD ≤ 30%	1) Review lab QC data to determine if they are in control. 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further CA.	Analyst	Precision-overall	RPD ≤ 30%
Laboratory duplicate	Daily	RPD ≤ 30%	1) Review lab QC data to determine if they are in control. 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further CA.	Analyst	Precision-laboratory	RPD ≤ 30%
MDL study	Once per year, upon any major system change, or quarterly MDL check.	MDLs established as described in 40 CFR Part 136, App. B shall not exceed one-half the RL	MDLs that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples.	Analyst	Sensitivity	MDLs established as described in 40 CFR Part 136, App. B shall not exceed one-half the RL

QAPP Worksheet #29: Project Documents and Records
(UFP-QAPP Manual Section 3.5.1)
(EPA 2106-G-05 Section 2.2.8)

Sample Collection and Field Records			
Record	***Generation***	***Verification***	***Storage location/archival***
Field Notes, and Logbooks	HDR FTL	HDR PM, Project Chemist	HDR Network Drive Project Folder
GIS files	HDR FTL	HDR PM	HDR Network Drive Project Folder
CoC Forms	HDR FTL	HDR PM, Project Chemist	HDR Network Drive Project Folder
Airbills	HDR FTL	HDR PM, Project Chemist	HDR Network Drive Project Folder
DQCRs	HDR FTL	HDR PM	HDR Network Drive Project Folder
Equipment Calibration Forms	HDR FTL	HDR PM	HDR Network Drive Project Folder
Logbooks	HDR FTL	HDR PM	HDR Network Drive Project Folder
Instrument data files	HDR FTL	HDR PM	HDR Network Drive Project Folder
Photo-documentation	HDR FTL	HDR PM	HDR Network Drive Project Folder

Project Assessments			
Record	***Generation***	***Verification***	***Storage location/archival***
Analytical Data Packages	Lab	HDR Project Chemist	HDR Network Drive Project Folder
Validation Checklists	DSA/ HDR Project Chemist	HDR Project Chemist/ HDR PM	HDR Network Drive Project Folder
Data Validation Reports	DSA/ HDR Project Chemist	HDR Project Chemist/ HDR PM	HDR Network Drive Project Folder
CA Reports	DSA/ HDR Project Chemist	HDR Project Chemist/ HDR PM	HDR Network Drive Project Folder

Laboratory Records			
Record	***Generation***	***Verification***	***Storage location/archival***
CoC Forms	Lab	Lab PM, HDR Project Chemist	Lab archives, HDR Network Drive Project Folder (data package)
Equipment Logs	Lab	Lab PM, HDR Project Chemist	Lab archives, HDR Network Drive Project Folder (data package)
Sample Prep Logs	Lab	Lab PM, HDR Project Chemist	Lab archives, HDR Network Drive Project Folder (data package)
Analytical Run Logs	Lab	Lab PM, HDR Project Chemist	Lab archives, HDR Network Drive Project Folder (data package)
CA Forms	Lab	Lab PM, HDR Project Chemist	Lab archives, HDR Network Drive Project Folder (data package)
Extraction Records	Lab	Lab PM, HDR Project Chemist	Lab archives, HDR Network Drive Project Folder (data package)
Reported Sample Results	Lab	Lab PM, HDR Project Chemist	Lab archives, HDR Network Drive Project Folder (data package)
Raw Data Printouts	Lab	Lab PM, HDR Project Chemist	Lab archives, HDR Network Drive Project Folder (data package)
LIMS Reports	Lab	Lab PM, HDR Project Chemist	Lab archives, HDR Network Drive Project Folder (data package)
Data Packages and Checklists	Lab	Lab PM, HDR Project Chemist	Lab archives, HDR Network Drive Project Folder (data package)

Laboratory Data Deliverables					
Record	***VOCs (Groundwater)***	***VOCs (Air-Soil Vapor)***	***VOCs (IDW Water)***	***Metals (IDW Water)***	***Other***
CoC Forms	X	X	X	X	
Equipment Logs	X	X	X	X	
Sample Prep Logs	X	X	X	X	
Analytical Run Logs	X	X	X	X	
CA Forms	X	X	X	X	
Extraction Records	X	X	X	X	
Reported Sample Results	X	X	X	X	
Raw Data Printouts	X	X			
LIMS Reports	X	X	X	X	
Data Packages and Checklists	X	X	X	X	

QAPP Worksheet #31, 32 & 33: Assessments and Corrective Action
(UFP-QAPP Manual Sections 4.1.1 and 4.1.2)
(EPA 2106-G-05 Section 2.4 and 2.5.5)

Assessments:

Assessment Type	Responsible Party & Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable due date
Field Sampling Technical Systems Audit (TSA)	HDR Project Chemist	Biennial	Semiannual LTM in April or October	Audit Report	30 days after TSA
On-Site Analytical TSA	HDR Project Chemist	Biennial (concurrent with Field Sampling TSA)	Semiannual LTM in April or October	Audit Report	30 days after TSA
Off-Site Laboratory TSA	DoD ELAP personnel or contractor	Annual	NA	ELAP annual certification audit report	NA
Laboratory Performance Audit	DSA Data reviewer/ validator, HDR Project Chemist	Ongoing with data package data validation	NA	Email from DSA and HDR Project Chemist to Laboratory PM	14 days after receipt of analytical data package
Data Review TSA	HDR Project Chemist	Ongoing with review of data validation reports and data qualifications	NA	Email from HDR Project Chemist to DSA; Data Review Summary Report	14 days after receipt of data validation reports

Assessment Response and Corrective Action:

Assessment Type	Responsibility for responding to assessment findings	Assessment Response Documentation	Timeframe for Response	Responsibility for Implementing Corrective Action	Responsible for monitoring Corrective Action implementation
Field Sampling TSA	HDR PM and FTL	Update/ addition to SOP; notification to sampling personnel	Prior to next sampling event	HDR PM and FTL	HDR PM and Project Chemist
On-Site Analytical TSA	HDR PM and FTL	Update/addition to SOP; notification to sampling personnel	Prior to next on-site analytical event (sampling event)	HDR PM and FTL	HDR PM and Project Chemist
Off-Site Laboratory TSA	Laboratory Representative	Per ELAP	Per ELAP	Laboratory PM, Analysts, Technicians	Laboratory PM and HDR Project Chemist
Laboratory Performance Audit	Laboratory PM, Analysts, Technicians	Documented in data package if edits to the data package required	Corrections are to be made before final data package is issued, and included in final data package	Laboratory PM, Analysts, Technicians	HDR Project Chemist
Data Review TSA	DSA Data reviewer/ validator	Summary Report prepared for each sampling event to summarize major issues with the data and note changes made to DSA's Data Review Reports	Within one week from receipt of final Data Review Report for a sampling event	DSA Data reviewer/ validator	HDR Project Chemist

QAPP Worksheet #34: Data Verification and Validation Inputs
(UFP-QAPP Manual Section 5.2.1 and Table 9)
(EPA 2106-G-05 Section 2.5.1)

Item	Description	Verification (completeness)	Validation (conformance to specifications)
Planning Documents/Records			
1	Approved QAPP	X	X
2	Field SOPs	X	X
3	Laboratory SOPs	X	X
Field Records			
4	Field Logbooks	X	X
5	Equipment Calibration Records	X	X
6	CoC Forms	X	X
7	Sampling Forms	X	X
8	Drilling Logs	X	X
9	Relevant Correspondence	X	X
10	Field Audit Reports	X	X
11	Field CA Reports	X	X
Analytical Data Package			
12	Cover Sheet (laboratory identifying information)	X	X
13	Case Narrative	X	X
14	Internal Laboratory CoC	X	X
15	Sample Receipt Records	X	X
16	Sample Chronology (i.e. dates and times of receipt, preparation and analysis)	X	X
17	Communication Records	X	X
18	Standards Traceability	X	X
19	Instrument Calibration Records	X	X
20	Definition of Laboratory Qualifiers	X	X
21	Results of Reporting Forms	X	X
22	QC Sample Results	X	X
23	CA Reports	X	X
24	Raw Data	X	X
25	Electronic Data Deliverable	X	X

QAPP Worksheet #35: Data Verification Procedures
(UFP-QAPP Manual Section 5.2.2)
(EPA 2106-G-05 Section 2.5.1)

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
CoC Forms, Shipping Airbills	CoC Forms, Shipping Airbills	CoC Forms and shipping documents will be reviewed and verified for completeness and accuracy against the actual contents of the coolers represented in the shipment. Three sheet carbon CoC forms will be used with the original and second copy sent with the samples, and the third copy kept by the sampling team.	HDR FTL Laboratory Sample Custodian
Field Notes	Field Logbook, Forms and Drilling Logs	Field notes and forms will be reviewed for completeness and accuracy prior to being placed in the site file and scanned into electronic files.	HDR FTL
Laboratory Data	Laboratory Data	All data packages will be verified internally by laboratory personnel for technical accuracy and completeness prior to delivery to HDR Upon receipt, the HDR Project Scientist will verify all data in accordance with standard data validation procedures.	Laboratory PM, HDR Project Chemist
SOPs	SOPs	Verify that all SOPs associated with field activities were met.	HDR PM, HDR FTL
Documentation of QC Sample Results	Documentation of QC Sample Results	Confirm that all method required QC samples were run and met required limits.	HDR Project Chemist
Off-site laboratory raw data	USEPA National Functional Guidelines for Organic and Inorganic Superfund Data Review (2016)	Compare and evaluate all sampling procedures, sampling plans, duplicate criteria, project quantitation limits, method performance criteria, and data qualifiers as specified in the UFP-QAPP and detailed in the HDR SOP 10, Data Verification. Final qualifiers, as described in SOP 10, will be as shown in Worksheet #36 of this QAPP.	HDR Project Chemist

QAPP Worksheet #36: Data Validation Procedures
(UFP-QAPP Manual Section 5.2.2)
EPA 2106-G-05 Section 2.5.1)

Data Validator: Project Chemist, HDR

Analytical Group/Method	Organics (VOCs in Water, VOCs in Air)	Inorganics (Metals in Water)
Data Deliverable Requirements:	Level 4 data package including all instrument raw data	Level 4 data package including all instrument raw data
Analytical Specifications:	Per method, the QSM 5.1, SOP 10 and this QAPP	Per method, the QSM 5.1, SOP 10 and this QAPP
Measurement of Performance Criteria:	DQOs in this QAPP	DQOs in this QAPP
Percent of Data Packages to be Validated:	100%	100%
Percent of Raw Data to be Reviewed:	10%	10%
Percent of Results to be Recalculated:	One result per analytical method per matrix	One result per analytical method per matrix
Validation Procedure:	National Functional Guidelines for Superfund Organic Methods Data Review, OLEM 9355.0-134, EPA-540-R-2016-002, September 2016	National Functional Guidelines for Inorganic Superfund Methods Data Review, OLEM 9355.0-133, EPA-540-R-2016-001, September 2016
Validation Code:	S2bVM (100%), S3VM (10%)	S2bVM (100%), S3VM (10%)
Electronic Validation Program/Version:	NA	NA

Validation Code and Label Identifier Table:

Validation Code*	Validation Label	Description/Reference
S2bVM	Stage 2b Validation Manual	EPA 540-R-08-005
S3VM	Stage 3 Validation Manual	EPA 540-R-08-005

Qualifier	Explanation
The following data qualifiers will be applied during data validation. Potential impacts on project-specific DQOs will be discussed in the data validation report.	
U	Not detected above MDL
J	Detected, concentration is estimated
J+	Detected, concentration is estimated, possibly biased high
J-	Detected, concentration is estimated, possibly biased low
UJ	Not detected, MDL is estimated
R	Rejected, data not usable

QAPP Worksheet #37: Data Usability Assessment
(UFP-QAPP Manual Section 5.2.3 including Table 12)
(EPA 2106-G-05 Section 2.5.2, 2.5.3, and 2.5.4)

Project Manager: Tom Holmes
Project Chemist: Lynn Lutz
Field Team Leader: Eric North

Step 1	**Review the project's objectives and sampling design** Review the key outputs defined during systematic planning (i.e., Project Quality Objectives or DQOs and Measurement Performance Criteria) to make sure they are still applicable. Review the sampling design for consistency with stated objectives. This provides the context for interpreting the data in subsequent steps.
Step 2	**Review the data verification and data validation outputs** Review available QA reports, including the data verification and data validation reports. Perform basic calculations and summarize the data (using graphs, maps, tables, etc.). Look for patterns, trends, and anomalies (i.e., unexpected results). Review deviations from planned activities (e.g., number and locations of samples, holding time exceedances, damaged samples, non-compliant PT sample results, and SOP deviations) and determine their impacts on the data usability. Evaluate implications of unacceptable QC sample results.
Step 3	**Verify the assumptions of the selected statistical method** Verify whether underlying assumptions for selected statistical methods (if documented in the QAPP) are valid. Common assumptions include the distributional form of the data, independence of the data, dispersion characteristics, homogeneity, etc. Depending on the robustness of the statistical method, minor deviations from assumptions usually are not critical to statistical analysis and data interpretation. If serious deviations from assumptions are discovered, then another statistical method may need to be selected.
Step 4	**Implement the statistical method** Implement the specified statistical procedures for analyzing the data and review underlying assumptions. For decision projects that involve hypothesis testing (e.g., "concentrations of lead in groundwater are below the action level") consider the consequences for selecting the incorrect alternative; for estimation projects (e.g., establishing a boundary for surface soil contamination), consider the tolerance for uncertainty in measurements.
Step 5	**Document data usability and draw conclusions:** Determine if the data can be used as intended, considering implications of deviations and CAs. Discuss data quality indicators. Assess the performance of the sampling design and Identify limitations on data use. Update the conceptual site model and document conclusions. Prepare the data usability summary report which can be in the form of text and/or a table.

The following is a summary of the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:
For samples analyzed by off-site laboratories, results will be subjected to data review, verification and validation, in accordance with the USEPA's National Functional Guidelines for Organic Data Review (USEPA, 2016) or Inorganic Data Review (USEPA, 2016).

Equations used to assess acceptance criteria include:

For Accuracy:

Percent Recovery for MS $\%R = \left(\frac{Spike\ conc. - Sample\ conc.}{Amount\ of\ spike\ added}\right) x\ 100$

Percent Recovery for LCS $\%R = \left(\frac{Spike\ conc.}{Amount\ of\ spike\ added}\right) x\ 100$

For Precision:

Relative Percent Different for MSD, and field duplicates % $RPD = \left[\frac{|Amount\ in\ sample\ 1 - Amount\ in\ Sample\ 2|}{\frac{Amount\ in\ Sample\ 1 + Amount\ in\ sample\ 2}{2}}\right] x\ 100$

For Completeness:

$\%Completeness = \left(\frac{Number\ of\ usable\ measurements}{Number\ of\ planned\ measurements}\right) x\ 100$

All data collected from the SI field activities will be evaluated against the following data quality parameters:

Precision – Precision refers to the degree to which repeated measurements are similar to one another, when obtained under prescribed conditions. Laboratory precision will be assessed by evaluating results of field and laboratory duplicates to determine RPD, LCSs, and MS/MSDs. The requirements for RPD are shown in the Worksheets above.

Accuracy – Accuracy is defined as the measure of the closeness of an individual measurement or the average of a number of measurements to the actual or 'true' value. Laboratory accuracy will be assessed by evaluating LCSs and MSs and calculating the %R. The requirements for %R are shown in the Worksheets above.

Representativeness – Representativeness is defined as a measure of the degree to which data accurately and precisely represents the characteristics and conditions of the sample from where the measurement was taken. Laboratory representativeness is assessed by ensuring that all analytical methods and laboratory procedures were followed consistently. In addition, method and instrument blanks are evaluated against the sample data to determine if results could be due to an outside source, such as glassware cross-contamination or instrument carryover. Field representativeness is evaluated in the same manner, through equipment blanks and review of sampling/decontamination techniques. Target analytes should not be present in any blanks. Data may be qualified accordingly if any analytes are detected in blank samples.

Completeness – Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount of data that was expected or planned for. Qualified data will be considered unless it has been rejected (R), in which case it is unusable. The goal for completeness is 100%, however rejected (unusable) data will be evaluated to determine whether data gaps exist, or if the project objectives were met, without it.

Comparability – Comparability is a measure of the confidence with which data sets may be compared to each other. Comparability is evaluated by reviewing adherence to Work Plans, SOPs, method requirements, and consistency in task execution, both in the field, and at off-site laboratories.

Sensitivity – Sensitivity is the ability of the method or instrument to detect the target analytes at the level of interest. In order to meet the project-specific DQOs, definitive data will be compared to the project's action limits or quantitation goals as listed in Worksheet #15.

Identify the personnel responsible for performing the usability assessment:
Lynn K. Lutz, Project Chemist, HDR

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:
A Data Validation Report will be included as an Appendix to the final report and will document the results of the data review, verification and validation. This report will describe the conclusions made during the data assessment regarding the data usability. Any limitations on the usability of the data will be explained, including the reasons for data qualifiers, the definitions of the qualifiers and a summary of the specific acceptance criteria that was assessed and found to be outside of control limits.

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Tables

TABLE 1
MAIN INSTALLATION LTM WELLS
UNIFORM FEDERAL POLICY – QUALITY ASSURANCE PROJECT PLAN
Environmental Restoration Support at Former Defense Depot Memphis, Tennessee

Well	Aquifer	Area	Sample Frequency	Northing (ft)	Easting (ft)	Top of Casing Elevation (ft, NAVD)	Ground Elevation (ft, NAVD)	Riser Length (ft)	Screen Length (ft)	Total Well Depth (ft, btoc)
DR1-1	Fluvial	TTA-1S	Biennial	276300	800856	293.14	293.42	121.7	20	141.7
DR1-1A	Fluvial	TTA-1S	Biennial	276307	800863	293.00	293.37	89.2	20	109.2
DR1-2	Fluvial	TTA-1N	Biennial	276537	801153	290.00	291.39	97.7	20	117.7
DR1-3	Fluvial	TTA-1S	Semiannual	276527	801416	290.93	291.11	109.7	20	129.7
DR1-4	Fluvial	TTA-1S	Semiannual	276231	801400	292.78	293.00	106.3	20	126.3
DR1-5	Fluvial	TTA-1S	Semiannual	276080	800828	294.46	294.86	124.7	20	144.7
DR1-5A	Fluvial	TTA-1S	Semiannual	276087	800835	294.51	294.87	90.0	20	110.0
DR1-6	Fluvial	TTA-1S	Semiannual	276044	801103	293.17	293.50	114.4	20	134.4
DR1-6A	Fluvial	TTA-1S	Semiannual	276035	801104	293.28	293.58	90.9	20	110.9
DR1-7	Fluvial	TTA-1N	Biennial	276791	801441	289.15	289.46	108.3	20	128.3
DR1-8	Fluvial	TTA-1N	Biennial	276752	800875	290.09	290.47	92.7	20	112.7
DR2-1	Fluvial	TTA-2	Semiannual	276772	806498	304.90	305.08	73.9	20	93.9
DR2-2	Fluvial	TTA-2	Annual	276771	806659	304.30	304.67	78.4	15	93.4
DR2-3	Fluvial	TTA-2	Annual	276539	806203	303.44	303.66	93.0	20	113.0
DR2-4	Fluvial	TTA-2	Annual	276456	806633	303.55	303.96	88.1	20	108.1
DR2-5	Fluvial	TTA-2	Annual	276831	806180	305.41	305.72	84.5	15	99.5
DR2-6	Fluvial	TTA-2	Semiannual	276644	805861	304.70	304.92	94.6	20	114.6
MW-16	Fluvial	Background	Biennial	278838	807100	299.86	300.19	57.6	15	72.6
MW-19	Fluvial	Background	Biennial	278946	800782	290.57	290.86	83.1	10	93.1
MW-21	Fluvial	TTA-1N	Semiannual	276473	800602	295.00	295.30	92.1	15	107.1
MW-22	Fluvial	TTA-1S	Biennial	275912	800702	298.04	298.49	95.4	10	105.4
MW-23	Fluvial	Background	Biennial	275791	801817	298.99	299.24	101.2	10	111.2
MW-24	Fluvial	Background	Biennial	275616	803539	299.51	299.81	97.3	15	112.3
MW-25A	Fluvial	TTA-2	Annual	275975	805521	269.88	270.13	73.0	10	83.0
MW-26	Fluvial	TTA-2	Semiannual	276508	805962	303.69	303.89	97.6	10	107.6
MW-34	Intermediate	Window	Annual	279411	801918	299.97	300.80	136.6	20	156.6
MW-38	Intermediate	Window	Biennial	279141	802450	307.45	308.45	139.9	15	154.9
MW-39	Fluvial	W-C	Semiannual	277281	802598	296.28	296.58	95.5	20	115.5
MW-39A	Upper Claiborne	W-C	Semiannual	277278	802608	298.61	298.70	148.1	20	168.1
MW-50	Fluvial	TTA-2	Annual	276456	807065	298.82	299.32	115.0	10	125.0
MW-52	Fluvial	SE	Annual	275372	805897	279.26	279.71	94.0	10	104.0
MW-53	Fluvial	Background	Biennial	279177	805136	306.38	305.58	72.5	10	82.5
MW-55	Fluvial	Background	Biennial	279301	801205	292.08	292.48	64.0	10	74.0
MW-62	Fluvial	B-835	Semiannual	278290	801858	293.71	293.90	86.1	10	96.1
MW-63A	Fluvial	N-C	Annual	278200	803573	305.96	306.33	130.0	10	140.0
MW-63B	Fluvial	N-C	Annual	278201	803558	305.78	306.22	115.0	10	125.0
MW-64	Fluvial	TTA-2	Semiannual	276952	805006	304.21	304.46	102.0	10	112.0
MW-66A	Fluvial	TTA-1N	Biennial	276626	799793	284.22	284.34	74.6	20	94.6
MW-85	Fluvial	TTA-2	Semiannual	276704	806065	304.13	304.50	95.9	15	110.9
MW-88	Fluvial	TTA-2	Semiannual	276879	806513	305.15	305.47	82.0	15	97.0
MW-89	Intermediate	Window	Semiannual	278287	802555	303.98	304.38	147.0	30	177.0
MW-90	Intermediate	Window	Semiannual	278284	802540	304.19	304.64	115.0	30	145.0
MW-92	Fluvial	TTA-2	Semiannual	276614	806490	304.41	304.78	93.0	15	108.0
MW-93	Fluvial	Background	Biennial	275542	804440	294.08	294.31	92.0	15	107.0
MW-94A	Fluvial	W-C	Semiannual	276806	803086	303.00	303.23	109.6	10	119.6
MW-96	Fluvial	TTA-2	Annual	276310	806320	289.02	289.67	75.5	20	95.5
MW-97	Fluvial	S-C	Semiannual	276074	802139	297.44	297.70	97.5	20	117.5
MW-98	Fluvial	W-C	Semiannual	276891	802573	294.43	294.93	137.0	10	147.0
MW-99	Fluvial	Background	Biennial	277443	801115	285.33	285.69	91.5	20	111.5
MW-100B	Fluvial	TTA-1N	Semiannual	276601	800854	290.92	291.47	107.4	20	127.4
MW-101[1]	Fluvial	TTA-1S	Semiannual	276204	801110	291.74	291.98	89.0	15	104.0
MW-102B	Fluvial	Background	Biennial	275761	800708	311.40	312.07	120.5	20	140.5
MW-103	Fluvial	N-C	Annual	278691	805160	301.37	301.89	70.0	20	90.0
MW-104	Fluvial	N-C	Semiannual	278676	805417	291.98	292.18	70.5	20	90.5

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MW-107[1]	Upper Claiborne	Window	Semiannual	278419	803010	304.92	305.18	128.0	15	143.0
MW-108	Upper Claiborne	W-C	Semiannual	277658	802986	303.07	303.25	160.0	10	170.0
MW-113	Fluvial	TTA-2	Semiannual	276685	806279	304.81	304.92	96.0	10	106.0
MW-140	Memphis	Window	Semiannual	279061	801716	298.12	298.16	224.6	20	244.6
MW-141	Intermediate	Window	Semiannual	278019	802571	303.71	303.70	148.7	20	168.7
MW-142	Fluvial	B-835	Annual	278056	801629	291.18	291.49	85.0	20	105.0
MW-143	Fluvial	B-835	Annual	278301	801201	290.66	290.90	78.6	20	98.6
MW-197A	Upper Claiborne	W-C	Semiannual	276975	802042	291.64	291.54	161.7	15	176.7
MW-197B	Fluvial	W-C	Semiannual	276973	802037	291.43	291.43	93.8	15	108.8
MW-198	Fluvial	B-835	Annual	277776	802142	291.78	292.20	90.3	15	105.3
MW-199A	Intermediate	B835	Semiannual	277756	802574	301.90	301.84	146.1	15	161.1
MW-199B	Fluvial	B-835	Semiannual	277752	802576	302.06	302.07	104.6	15	119.6
MW-200	Fluvial	W-C	Annual	277006	802859	300.18	300.51	103.2	15	118.4
MW-202A	Intermediate	Window	Semiannual	278686	802111	299.67	299.69	176.2	15	191.2
MW-202B	Intermediate	Window	Semiannual	278693	802112	299.92	299.74	118.8	15	133.8
MW-203A	Upper Claiborne	W-C	Annual	276842	801740	290.70	290.80	142.9	20	162.9
MW-203B	Fluvial	W-C	Semiannual	276821	801742	290.87	291.10	93.0	20	113.0
MW-204A	Fluvial	W-C	Semiannual	276725	802168	292.59	292.49	133.3	15	148.3
MW-204B	Fluvial	W-C	Semiannual	276708	802167	292.71	293.00	94.9	15	109.9
MW-205A	Upper Claiborne	W-C	Annual	277157	802277	292.30	292.40	141.3	15	156.3
MW-205B	Fluvial	W-C	Semiannual	277173	802278	292.16	292.30	97.3	15	112.3
MW-206A	Fluvial	W-C	Semiannual	277219	802792	300.32	300.35	127.3	15	142.4
MW-206B	Fluvial	W-C	Semiannual	277201	802795	300.30	300.12	96.7	15	111.7
MW-207A	Upper Claiborne	W-C	Semiannual	277653	803192	304.05	304.45	149.9	15	164.9
MW-207B	Fluvial	W-C	Semiannual	277665	803193	304.06	304.42	108.5	15	123.5
MW-208A	Upper Claiborne	W-C	Semiannual	277382	802799	302.21	302.40	183.4	15	198.5
MW-208B	Fluvial	W-C	Semiannual	277397	802815	302.13	302.08	106.7	15	121.7
MW-209A	Intermediate	B-835	Semiannual	277574	802507	298.45	298.36	189.0	15	204.0
MW-209B	Fluvial	B-835	Semiannual	277582	802520	298.89	298.72	102.3	15	117.3
MW-210A	Intermediate	W-C	Annual	277239	801958	289.61	289.70	177.0	15	192.0
MW-210B	Fluvial	W-C	Semiannual	277228	801952	289.54	289.83	97.0	15	112.0
MW-211	Intermediate	Window	Biennial	278001	802974	304.14	304.09	166.3	15	181.3
MW-212	Fluvial	B-835	Semiannual	278028	802225	295.74	295.68	85.3	15	100.3
MW-213	Fluvial	B-835	Semiannual	278427	801669	294.22	294.20	77.3	15	92.3
MW-214A	Upper Claiborne	N-C	Annual	277878	803907	304.01	303.96	119.1	15	134.1
MW-214B	Upper Claiborne	N-C	Annual	277876	803922	304.10	303.96	101.6	15	116.6
MW-215A	Upper Claiborne	N-C	Annual	277298	804164	304.97	304.86	128.8	15	143.8
MW-215B	Fluvial	N-C	Annual	277298	804177	305.03	304.98	105.4	15	120.4
MW-216	Fluvial	S-C	Biennial	276025	801996	297.72	297.63	99.9	15	115.0
MW-217	Fluvial	TTA-2	Semiannual	276671	805214	304.65	304.51	101.8	15	116.8
MW-218	Fluvial	TTA-2	Semiannual	276937	805628	306.07	306.00	98.9	15	114.0
MW-219	Fluvial	TTA-1N	Semiannual	276429	800461	295.13	295.00	98.0	15	113.0
MW-229	Intermediate	Window	Biennial	279294	802837	311.78	312.09	188.4	20	208.4
MW-252	Intermediate	Window	Biennial	278789	801365	294.16	294.40	126.1	20	146.1
MW-253	Intermediate	Window	Biennial	278287	801191	290.47	290.80	118.3	20	138.3
MW-254	Memphis	Window	Annual	279334	800858	292.84	293.28	285.8	20	305.8
MW-255	Memphis	Window	Annual	279305	801227	291.84	292.38	284.7	20	304.7
MW-256	Intermediate	Window	Semiannual	279302	801244	292.68	293.40	127.1	20	147.1
MW-258	Fluvial	N-C	Semiannual	278126	804427	304.37	304.83	79.3	20	99.3
MW-259	Fluvial	TTA-2	Semiannual	276279	804451	290.77	291.44	98.6	20	118.6
MW-260	Fluvial	N-C	Annual	278398	804376	304.16	304.45	68.0	20	88.3
MW-261	Fluvial	S-C	Annual	276391	802592	293.52	293.79	90.0	20	110.3
MW-262	Intermediate	Window	Annual	279334	800833	293.22	293.50	154.4	10	164.6
MW-263	Fluvial	N-C	Semiannual	278945	805817	291.40	291.78	69.1	10	79.3

TABLE 1
MAIN INSTALLATION LTM WELLS
UNIFORM FEDERAL POLICY – QUALITY ASSURANCE PROJECT PLAN
Environmental Restoration Support at Former Defense Depot Memphis, Tennessee

Well	Aquifer	Area	Sample Frequency	Northing (ft)	Easting (ft)	Top of Casing Elevation (ft, NAVD)	Ground Elevation (ft, NAVD)	Riser Length (ft)	Screen Length (ft)	Total Well Depth (ft, btoc)
MW-264	Upper Claiborne	N-C	Annual	278411	804590	303.72	304.00	104.8	10	115.0
MW-265	Fluvial	N-C	Semiannual	278112	804710	305.15	305.61	85.8	10	96.0
MW-266	Fluvial	TTA-2	Annual	277092	806686	304.68	305.10	77.1	10	87.3
MW-267	Fluvial	TTA-2	Semiannual	277161	806001	303.84	304.30	71.9	10	82.1
MW-268	Upper Claiborne	TTA-2	Annual	277204	805284	304.59	304.92	109.5	10	119.7
MW-269	Fluvial	TTA-1N	Semiannual	276369	800127	290.05	290.50	92.2	10	102.4
MW-270	Fluvial	SE	Semiannual	275483	805042	281.74	282.20	78.4	10	88.6
MW-271	Fluvial	S-C	Annual	276315	803774	294.91	295.50	134.7	10	144.9
MW-272	Fluvial	Background	Annual	275880	804037	293.27	293.70	112.8	10	123.0
MW-273	Intermediate	Window	Annual	279713	800122	284.73	285.00	128.1	10	138.3
MW-274	Fluvial	Background	Semiannual	275726	806543	294.30	294.60	89.3	10	99.5
MW-275	Fluvial	Background	Semiannual	275232	805306	272.31	272.59	80.5	10	90.7
MW-276	Fluvial	Background	Semiannual	275564	804697	288.68	288.91	87.5	10	97.7
MW-277	Fluvial	Background	Semiannual	275532	803998	301.67	301.96	102.3	10	112.5
MW-278	Fluvial	TTA-1N	Semiannual	276294	799814	292.18	292.46	91.0	10	101.2
MW-279	Fluvial	TTA-1S	Semiannual	275982	800579	299.89	300.17	112.0	10	122.2
MW-280	Fluvial	TTA-2	Semiannual	277390	806313	306.36	306.57	76.0	10	86.2
MW-281	Fluvial	N-C	Semiannual	278155	804123	304.56	305.03	81.7	10	91.9
MW-282	Fluvial	Background	Semiannual	278710	804033	307.81	308.14	76.0	10	86.2
MW-283	Fluvial	Background	Semiannual	278176	806074	304.34	304.87	77.0	10	87.2
PMW21-01	Fluvial	TTA-1N	Semiannual	276533	800600	294.76	295.00	88.4	20	108.4
PMW21-02	Fluvial	TTA-1N	Semiannual	276575	800701	292.98	293.19	91.3	20	111.3
PMW21-03	Fluvial	TTA-1N	Semiannual	276573	800743	292.11	292.72	90.3	20	110.3
PMW21-04	Fluvial	TTA-1N	Semiannual	276602	800772	291.87	292.20	89.0	20	109.0
PMW21-05	Fluvial	TTA-1N	Semiannual	276628	801130	288.53	288.92	94.3	20	114.3
PMW85-01	Fluvial	TTA-2	Semiannual	276802	806146	305.08	305.39	93.2	10	103.2
PMW85-05	Fluvial	TTA-2	Semiannual	276752	806222	305.12	305.32	93.2	10	103.2
PMW92-02	Fluvial	TTA-2	Semiannual	276667	806476	304.17	304.35	94.8	10	104.8
PMW92-03	Fluvial	TTA-2	Semiannual	276679	806439	303.91	304.17	92.5	10	102.5
PMW101-02A	Fluvial	TTA-1S	Semiannual	276282	801145	292.00	292.29	117.7	20	137.7
PMW101-02B	Fluvial	TTA-1S	Semiannual	276286	801145	291.98	292.24	97.8	20	117.8
PMW101-03A	Fluvial	TTA-1S	Semiannual	276348	801198	291.61	291.99	119.2	20	139.2
PMW101-03B	Fluvial	TTA-1S	Semiannual	276353	801194	291.55	291.82	99.3	20	119.3
PMW101-04A	Fluvial	TTA-1S	Semiannual	276299	801182	291.07	291.43	117.9	20	137.9
PMW101-04B	Fluvial	TTA-1S	Semiannual	276296	801187	291.47	291.75	98.6	20	118.6
PMW101-06A	Fluvial	TTA-1S	Semiannual	276192	801187	292.13	292.72	120.0	20	140.0
PMW101-06B	Fluvial	TTA-1S	Semiannual	276195	801184	292.17	292.40	99.3	20	119.3
PMW101-07A	Fluvial	TTA-1S	Annual	276143	801172	292.20	292.52	117.9	20	137.9
PMW101-07B	Fluvial	TTA-1S	Semiannual	276142	801177	292.36	292.70	98.0	20	118.0

Notes:
1: MW-101 has three screened sections at the following depths (ft, btoc): 89-104, 109-119 and 124-134.
2: MW-107 has two screened sections at the following depths (ft, btoc): 128-143 and 148-158.
ft: feet
btoc: below top of casing
NAVD: North American Vertical Datum of 1988

TABLE 2
DUNN FIELD LTM WELLS
UNIFORM FEDERAL POLICY – QUALITY ASSURANCE PROJECT PLAN
Environmental Restoration Support at Former Defense Depot Memphis, Tennessee

Well	Aquifer	Area	Sample Frequency	Northing (ft)	Easting (ft)	Top of Casing Elevation (ft, NAVD)	Ground Elevation (ft, NAVD)	Riser Length (ft)	Screen Length (ft)	Total Well Depth (ft, btoc)
MW-03	Fluvial	DF North	Annual	281596	802101	292.35	290.40	65.5	10	75.5
MW-04	Fluvial	Background	Biennial	281279	802369	301.61	300.00	60.0	20	80.0
MW-06	Fluvial	DF West	Semiannual	280604	802069	289.11	288.10	51.0	20	71.0
MW-07	Fluvial	DF North	Annual	281840	802482	295.10	293.10	67.0	10	77.0
MW-08	Fluvial	DF North	Annual	282001	802728	292.59	292.74	56.5	10	66.5
MW-13	Fluvial	Background	Biennial	281034	802369	300.01	300.10	66.0	15	81.0
MW-15	Fluvial	DF West	Biennial	280349	801985	295.12	295.23	63.4	15	78.4
MW-28	Fluvial	Background	Biennial	281569	803154	294.79	294.89	54.3	15	69.3
MW-31	Fluvial	DF North	Annual	281652	801784	290.37	287.50	64.1	15	79.1
MW-44	Fluvial	Off Depot	Annual	281074	800601	269.07	269.40	64.0	10	74.0
MW-54	Fluvial	Off Depot	Annual	281159	801184	295.39	295.57	84.5	10	94.5
MW-57	Fluvial	DF West	Semiannual	280184	802006	290.77	291.10	60.0	10	70.0
MW-58	Fluvial	Background	Biennial	279845	802066	290.51	290.70	57.0	10	67.0
MW-67	Memphis	Background	Biennial	280473	800934	278.21	275.53	260.0	15	275.0
MW-68	Fluvial	DF North	Biennial	281501	802040	291.69	291.60	72.5	10	82.5
MW-69	Fluvial	DF West	Biennial	281203	802011	307.02	304.90	82.1	10	92.1
MW-70	Fluvial	DF West	Semiannual	281030	801988	304.99	302.80	80.8	10	90.8
MW-71	Fluvial	DF West	Annual	280585	801805	294.40	291.90	65.5	10	75.5
MW-76	Fluvial	Off Depot	Biennial	281312	801643	302.71	303.30	73.0	20	93.0
MW-77	Fluvial	Off Depot	Semiannual	281143	801815	304.42	304.70	68.0	20	88.0
MW-78	Fluvial	DF North	Biennial	282052	802065	275.00	275.40	44.5	20	64.5
MW-79	Fluvial	DF North	Annual	281794	800899	285.03	285.40	82.5	20	102.5
MW-80	Fluvial	Background	Biennial	281418	800199	273.81	274.00	53.0	20	73.0
MW-87	Fluvial	DF West	Semiannual	280696	802039	294.93	292.80	63.0	15	78.0
MW-91	Fluvial	DF West	Biennial	280475	802014	291.99	289.30	55.0	15	70.0
MW-126	Fluvial	Background	Biennial	282390	800492	252.22	252.49	16.0	10	26.0
MW-129	Fluvial	DF North	Annual	282271	803129	293.01	293.33	65.0	15	80.0
MW-130	Fluvial	DF North	Annual	282117	803241	293.17	293.77	59.5	20	79.5
MW-134	Fluvial	DF West	Biennial	281013	802103	300.81	301.05	75.0	15	90.0
MW-144	Fluvial	Off Depot	Semiannual	281139	801529	291.60	291.89	55.9	20	76.3
MW-145	Fluvial	Off Depot	Annual	280968	800823	284.72	284.86	76.6	20	97.0
MW-147	Fluvial	DF North	Biennial	281502	801674	289.76	289.93	57.8	20	78.1
MW-148	Fluvial	Off Depot	Biennial	281378	801462	294.71	294.87	67.8	20	88.1
MW-149	Fluvial	Off Depot	Semiannual	281130	800983	287.18	287.44	81.6	20	101.9
MW-150	Fluvial	Off Depot	Semiannual	281240	801284	296.86	297.00	71.2	20	91.4
MW-151	Fluvial	Off Depot	Annual	281290	800875	284.27	284.42	76.5	20	96.8
MW-152	Fluvial	Off Depot	Annual	281516	800893	289.59	289.82	90.9	20	111.1
MW-153	Fluvial	DF North	Biennial	282119	800952	279.17	279.26	76.1	20	96.3
MW-154	Fluvial	Background	Biennial	280502	800919	273.81	274.07	52.5	15	67.7
MW-155	Fluvial	Off Depot	Annual	281325	801169	291.54	291.83	76.6	20	96.8
MW-157	Fluvial	Off Depot	Annual	281051	801348	286.47	286.55	57.0	20	77.3
MW-158	Fluvial	Off Depot	Annual	281434	801005	294.07	294.38	91.1	15	106.6
MW-158A	Fluvial	Off Depot	Annual	281444	801006	293.95	294.22	78.4	15	93.6
MW-159	Fluvial	Off Depot	Semiannual	281304	801007	286.36	286.68	79.3	20	99.5
MW-160	Fluvial	Off Depot	Annual	281367	801304	293.84	294.13	65.8	20	86.0
MW-163	Fluvial	Off Depot	Annual	281153	801487	290.63	290.81	56.5	20	76.7
MW-164	Fluvial	Off Depot	Annual	280998	801497	287.48	287.71	55.3	20	75.5
MW-165	Fluvial	Off Depot	Semiannual	281385	800855	287.06	287.35	88.1	15	103.3
MW-165A	Fluvial	Off Depot	Annual	281384	800866	287.26	287.53	71.3	15	86.5
MW-166	Fluvial	Off Depot	Semiannual	281225	800928	282.72	283.29	84.2	15	99.4
MW-166A	Fluvial	Off Depot	Semiannual	281213	800927	282.90	283.36	67.6	15	82.8
MW-167	Fluvial	Background	Biennial	281394	800619	284.82	285.21	67.7	15	82.9
MW-169	Upper Claiborne	Background	Biennial	282491	800957	261.90	262.17	68.0	20	88.2
MW-170	Fluvial	Background	Biennial	282443	801260	273.75	273.98	59.7	20	79.9
MW-171	Fluvial	DF North	Annual	282315	801058	270.69	271.02	53.2	15	68.4
MW-174	Fluvial	DF West	Biennial	280352	802092	296.56	296.83	67.0	10	77.0
MW-176	Fluvial	DF West	Biennial	280824	802032	299.68	299.92	76.0	10	86.0
MW-180	Fluvial	DF North	Biennial	281476	802132	296.14	296.39	72.0	10	82.0

TABLE 2
DUNN FIELD LTM WELLS
UNIFORM FEDERAL POLICY – QUALITY ASSURANCE PROJECT PLAN
Environmental Restoration Support at Former Defense Depot Memphis, Tennessee

Well	Aquifer	Area	Sample Frequency	Northing (ft)	Easting (ft)	Top of Casing Elevation (ft, NAVD)	Ground Elevation (ft, NAVD)	Riser Length (ft)	Screen Length (ft)	Total Well Depth (ft, btoc)
MW-182	Fluvial	Background	Biennial	280524	800623	272.63	272.90	59.2	10	69.2
MW-184	Fluvial	Off Depot	Annual	280903	801442	283.12	283.34	58.0	10	68.0
MW-187	Fluvial	Background	Biennial	280563	802348	302.74	303.21	76.0	10	86.0
MW-190	Fluvial	Off Depot	Semiannual	281139	801596	297.32	297.58	78.0	10	88.0
MW-220	Fluvial	DF North	Annual	281617	802167	293.29	290.31	64.9	15	79.9
MW-221	Fluvial	DF West	Biennial	281400	802100	301.52	298.37	73.1	15	88.1
MW-222	Fluvial	DF West	Biennial	280986	802146	303.82	301.06	74.2	15	89.2
MW-223	Fluvial	DF West	Biennial	280914	802104	303.00	300.41	73.9	15	88.9
MW-224	Fluvial	DF West	Annual	281018	802182	304.13	301.18	73.7	15	88.7
MW-225	Fluvial	DF West	Biennial	280947	802071	304.52	301.30	75.0	15	90.0
MW-226	Fluvial	DF West	Biennial	280932	802147	303.19	300.56	74.2	15	89.2
MW-227	Fluvial	DF West	Biennial	280258	802081	299.70	296.64	63.6	15	78.6
MW-228	Fluvial	DF West	Biennial	280252	802157	301.65	298.59	64.1	15	79.1
MW-230	Fluvial	DF North	Annual	281843	802800	286.57	286.66	59.2	15	74.2
MW-235	Fluvial	Background	Biennial	280728	800448	264.00	264.21	50.6	10	60.8
MW-237	Intermediate	Off Depot	Annual	281356	800964	289.18	289.53	166.5	10	176.7
MW-241	Fluvial	Off Depot	Annual	281390	801397	292.97	293.16	73.4	15	88.4
MW-242	Fluvial	Off Depot	Annual	281297	801229	295.40	295.94	73.2	16	88.7
MW-243	Fluvial	Off Depot	Annual	281371	801116	292.26	292.53	80.7	20	100.7
MW-244	Fluvial	Off Depot	Annual	281333	801101	288.72	289.45	76.3	20	96.3
MW-245	Fluvial	Off Depot	Annual	281379	801035	290.48	290.62	85.1	20	105.1
MW-246	Fluvial	Off Depot	Semiannual	281387	800952	288.17	288.49	85.2	20	105.2
MW-247	Fluvial	Off Depot	Annual	281319	800900	286.17	286.63	80.5	20	100.5
MW-248	Fluvial	Off Depot	Biennial	281254	800720	275.45	275.93	67.5	20	87.5
MW-249	Fluvial	Off Depot	Semiannual	281030	800790	285.53	285.89	78.0	20	98.0
MW-250	Intermediate	Off Depot	Biennial	281046	800900	289.66	290.19	168.7	15	183.7
MW-251	Intermediate	Off Depot	Annual	281212	801022	285.83	286.16	160.2	15	175.2

Notes:
ft: feet
btoc: below top of casing
NAVD: North American Vertical Datum of 1988

Figures

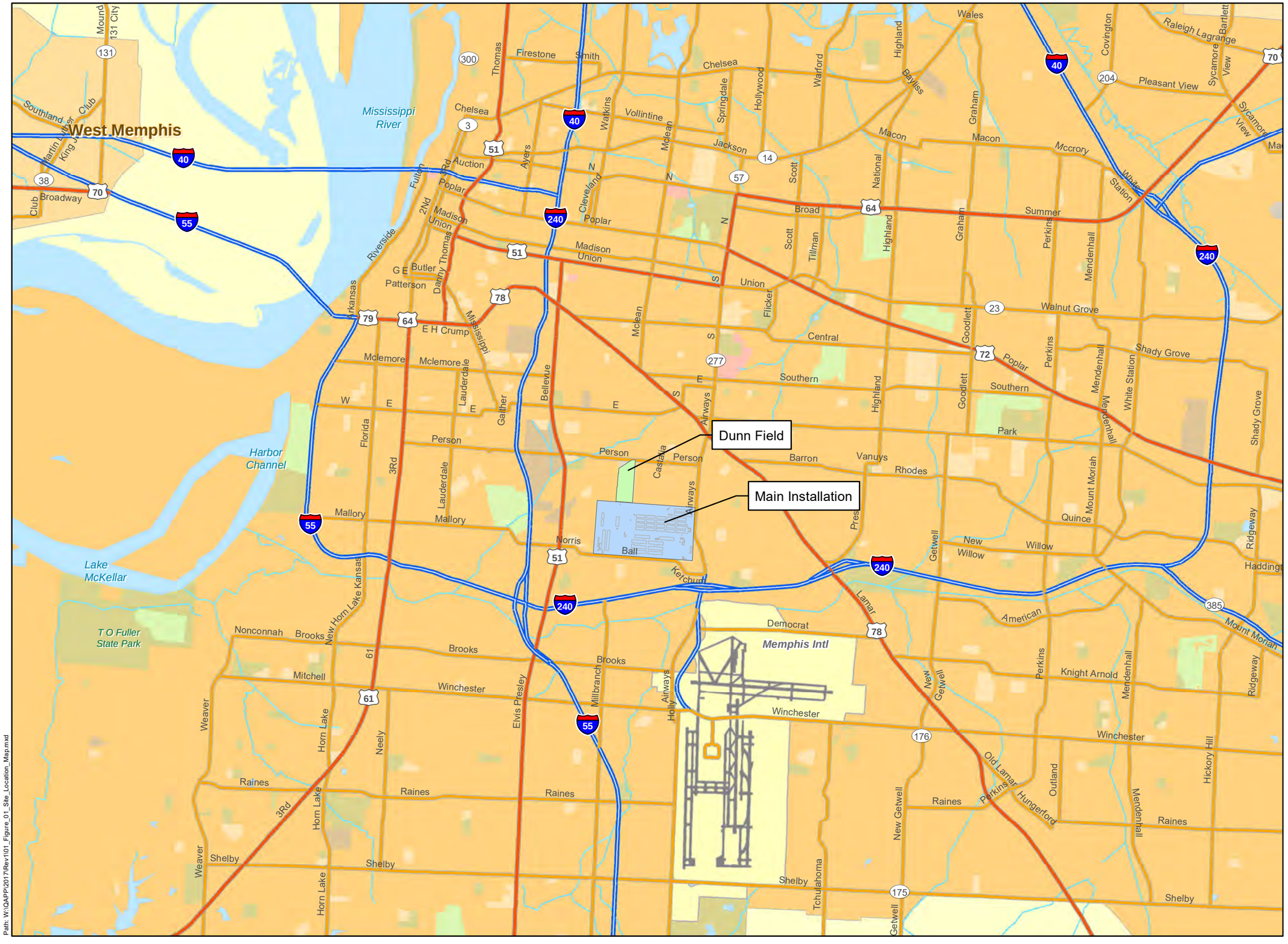

Figure 1

Site Location Map

Environmental Restoration Support
Uniform Federal Policy –
Quality Assurance Project Plan

Former Defense Depot
Memphis, Tennessee

Projection: NAD 1927 StatePlane Tennessee
Units: Feet, Elevation Units: Feet, NAVD88

Date: 3/8/2018
Edition: Rev 1

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Figure 2

Main Installation Response Actions

Environmental Restoration Support
Uniform Federal Policy –
Quality Assurance Project Plan

Former Defense Depot
Memphis, Tennessee

OU-1
OU-2
OU-3
OU-4
FU-1
FU-2
FU-3
FU-4
FU-5
FU-6
Bldg. 949 Surface Soil Removal Action (2001)
Former PCP Dip Vat Soil Removal Action (1985)
Cafeteria (Bldg 274) Surface Soil Removal Action (1998)
BRAC Parcels 35 & 28 Surface Soil Removal Action (2000)
Building 873 Soil Removal Action (1985)
Housing Area Surface Soil Removal Action (1998)
925
949
835
865
720
737
629
630
649
650
529
530
549
550
429
430
449
450
329
330
349
350
319
210
229
230
249
250
144
756
770
560
468
469
360
263
260
265
270
271
274
970
972
1089
1086
1088
1087
1091
995
670
689
685
690
470
489
490
Hays
Castalia
Airways
Perry
Amido
Sitler
Ball
Memphis Depot Pkwy
Rozelle
Vista

Legend

- Operable Units (OU-#)
- Past Response Actions
- Functional Unit Boundary (FU-#)
- Area Available for Unrestricted Use
- Buildings *490 - DDMT Assigned Number*
- Roads

N
W E
S

0 500 1,000
Feet

Projection: NAD 1927 StatePlane Tennessee
Units: Feet, Elevation Units: Feet, NAVD88

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Figure 3

Main Installation
EBT Wells

Environmental Restoration Support
Uniform Federal Policy –
Quality Assurance Project Plan

Former Defense Depot
Memphis, Tennessee

Legend

- EBT Injection Well
- EBT Performance Monitoring Well
- Fluvial Aquifer Well
- Intermediate Aquifer Well
- Memphis Aquifer Well
- Holding Pond
- Surface Water
- Buildings *490 - DDMT Assigned Number*
- Roads

N
W E
S

0 500 1,000
Feet

Projection: NAD 1927 StatePlane Tennessee
Units: Feet, Elevation Units: Feet, NAVD88

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TTA-1 North

PMW21-04
PMW21-02
PMW21-01
IW21-03A/B
IW21-04A/B
IW21-01A/B
MW-21
IW21-02A/B
1089
1086

TTA-1 South

PMW101-02A/B
PMW101-04A/B
IW101-05A/B/C
IW101-04A/B/C
IW101-02A/B/C
IW101-03A/B/C
IW101-07A/B/C
PMW101-07A/B
DR1-6
DR1-6A
970
972
Twenty First
Amido

TTA-2

IW92-08
DR2-5
IW85-05
PMW85-01
IW85-06
PMW85-05
MW-85
MW-113
IW92-07
PMW92-03
IW92-01
DR2-2
IW92-02
IW92-03
360
263
260
261
265
274
270
271
Danielson
Truitt

Legend

Aquifer

- Fluvial
- Intermediate/Upper Claiborne
- Memphis/ Upper Claiborne (Deep)

Area

- B-835
- Background
- North Central
- South Central
- South East
- TTA-1 North
- TTA-1 South
- TTA-2
- West Cental
- Window

- Property Boundary
- Buildings *490 - DDMT Assigned Number*
- Roads
- Clay Elevation Exceeds Groundwater Elevation

Figure 4

Main Installation LTM Wells

Environmental Restoration Support
Uniform Federal Policy –
Quality Assurance Project Plan

Former Defense Depot
Memphis, Tennessee

N W E S

0 250 500
Feet

Projection: NAD 1927 StatePlane Tennessee
Units: Feet, Elevation Units: Feet, NAVD88

Date: 3/8/2018
Edition: Rev 1

HDR

Path: W:\QAPP\2017\Rev1\04_MI_LTM_Wells.mxd

Note
1. Groundwater elevations for IAQ wells MW-107 and MW-169 are shown and included to aid interpretation of elevation contours at the entrance to the window and northwest of Dunn Field. Both wells are screened in Upper Claiborne sand where a clay layer is not present below the Fluvial sand.

Legend

- Fluvial Aquifer Well
- Intermediate/Upper Claiborne Well
- Potentiometric surface of the Fluvial Aquifer 1-ft. contour
- Potentiometric surface of the Fluvial Aquifer 5-ft. contour
- Potentiometric surface extrapolation
- Groundwater Flow Direction
- Property Boundary
- Roads
- Buildings
- Clay Elevation Exceeds Groundwater Elevation

MW-03 229.40 — Blue: Fluvial Aquifer, used for groundwater contours (10/4-5/17)

MW-169 190.40 — Orange: Intermediate/ Upper Claiborne Aquifer, used for groundwater contours (10/4-5/17)

Groundwater elevations are in ft, NAVD

Figure 5

Fluvial Aquifer Groundwater Elevations, October 2017

Environmental Restoration Support
Uniform Federal Policy –
Quality Assurance Project Plan

Former Defense Depot
Memphis, Tennessee

N W E S

0 250 500
Feet

Projection: NAD 1927 StatePlane Tennessee
Units: Feet, Elevation Units: Feet , NAVD88

Date: 3/8/2018
Edition: Rev 1

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Path: W:\QAPP2017\Rev1\05_FAQ_GWeleOct17.mxd

Legend

- Intermediate/Upper Claiborne Well
- Intermediate Aquifer Contours
- Groundwater Flow Direction (Intermediate)
- MW-237 176.56 Intermediate Aquifer, used for groundwater contours (10/4-5/17)
  Groundwater elevations are in ft, NAVD.
- Property Boundary
- Roads
- Buildings
- Clay Elevation Exceeds Groundwater Elevation

Figure 6

Intermediate Aquifer Groundwater Elevations, October 2017

Environmental Restoration Support
Uniform Federal Policy – Quality Assurance Project Plan

Former Defense Depot
Memphis, Tennessee

N W E S

0 250 500

Feet

Projection: NAD 1927 StatePlane Tennessee Units: Feet, Elevation Units: Feet, NAVD88

Date: 3/9/2018
Edition: Rev 1

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Legend

- Memphis Aquifer Well
- Potentiometric surface of the Memphis Aquifer 1-ft. contour
- MW-254 173.56 Memphis Aquifer Groundwater Elevation (10/4-5/17) Groundwater elevations are in ft, NAVD.
- Property Boundary
- Roads
- Buildings
- Clay Elevation Exceeds Groundwater Elevation

Figure 7
Memphis Aquifer Groundwater Elevations, October 2017

Environmental Restoration Support
Uniform Federal Policy – Quality Assurance Project Plan

Former Defense Depot
Memphis, Tennessee

N W E S

0 250 500
Feet

Projection: NAD 1927 StatePlane Tennessee
Units: Feet, Elevation Units: Feet, NAVD88

Date: 3/9/2018
Edition: Rev 1

Figure 8
Main Installation Fluvial Aquifer PCE Concentrations, October 2017

Environmental Restoration Support
Uniform Federal Policy –
Quality Assurance Project Plan

Former Defense Depot
Memphis, Tennessee


Note:
1. Color-coded wells symbols are based on the most recent analytical result at each well. Results are from the October 2016 and October 2017 LTM events. Only concentrations from the October 2017 LTM event are shown below the well ID.
2. Groundwater contours are from the October 2017 LTM event.
Legend
TCE Range (µg/L)
Well Symbols
0-5
5-10
10-50
50-100
>100
Contour Shading
5-10
10-50
50-100
>100
Potentiometric surface of the Fluvial Aquifer 5-ft. contour
Potentiometric surface of the Fluvial Aquifer 1-ft. contour
Potentiometric surface of the Intermediate Aquifer 5-ft. contour
Property Boundary
Groundwater Flow Direction (Fluvial)
Groundwater Flow Direction (Intermediate)
Clay Elevation Exceeds Groundwater Elevation
Figure 9
Main Installation Fluvial Aquifer TCE Concentrations, October 2017
Environmental Restoration Support
Uniform Federal Policy –
Quality Assurance Project Plan
Former Defense Depot
Memphis, Tennessee
0 250 500
Feet
Projection: NAD 1927 StatePlane Tennessee
Units: Feet, Elevation Units: Feet, NAVD88
Date: 3/9/2018
Edition: Rev 1
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MW-273
<1
MW-262
<1
MW-256
24.5
MW-34
2.16
MW-229
MW-38
MW-252
MW-202A
4
MW-202B
36.8
MW-253
<1
MW-107B
5.43
MW-107T
8.8
MW-90
13.5
MW-89
2.11
MW-141
27.1
MW-211
MW-199A
<1
MW-108
3.3
MW-209A
0.507
MW-207A
10.1
MW-208A
10.1
MW-39A
3.93
MW-210A
2.93
MW-205A
9.32
MW-197A
17.9
MW-203A
0.884
MW-264
<1
MW-214A
5.25
MW-214B
5.1
MW-215A
5.23
MW-268
<1
175
180
185
190
195
200
205
210
215
Ledger
Cincinnati
Dunn
Silver
Raven
Corry
Warren
Sparks
Merlin
Cane
Starr
Elliston
Rayner
Eloise
Mallory
Dempster
Norris
Dottie
Gausco
Whitmore
Antona
Perry
Twenty Seventh
Twenty Third
Twenty Fifth
Twenty First
Amido
Ball
Rozelle
Manchester
Kingsview
Vista
Fairmeade
Sherrie
Verdun
Johanna
Lapaloma
Thames
Gladney
Carbondale
W E Freeman
Sitler
Behnke
Fifth
L
Danielson
Memphis Depot Pkwy
Alley
Troyer
L Gillette
Kotick
Third
Lloyd
Truitt
Airways
Dwight
Heyde
Hays
State
Barksdale
Freemont
Ferber
Leltner
Murley
Castalia
Custer
Filmore
Frisco
925
949
835
865
972
970
1089
1086
1088
1087
1090
995
737
720
629
630
649
650
756
770
529
530
549
550
429
430
449
450
319
329
330
349
350
210
229
230
249
250
144
560
670
689
685
690
470
489
490
468
469
360
263
260
261
265
270
274
271
Legend
PCE Range (µg/L)
Well Symbols
0 - 5
5 - 10
10 - 50
>50
Contour Shading
5-10
10-50
>50
Potentiometric surface of the Intermediate Aquifer 5-ft. contour
Groundwater Flow Direction (Intermediate)
Property Boundary
Clay Elevation Exceeds Groundwater Elevation
Note:
1. Color-coded wells symbols are based on the most recent analytical result at each well. Results are from the October 2016 and October 2017 LTM events. Only concentrations from the October 2017 LTM event are shown below the well ID.
2. Groundwater contours are from the October 2017 LTM event.
Figure 10
Main Installation Intermediate Aquifer
PCE Concentrations,
October 2017
Environmental Restoration Support
Uniform Federal Policy –
Quality Assurance Project Plan
Former Defense Depot
Memphis, Tennessee
N
W
E
S
0
250
500
Feet
Projection: NAD 1927 StatePlane Tennessee
Units: Feet, Elevation Units: Feet, NAVD88
Date: 3/9/2018
Edition: Rev 1
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Figure 11

Main Installation Intermediate Aquifer TCE Concentrations, October 2017

Environmental Restoration Support
Uniform Federal Policy –
Quality Assurance Project Plan

Former Defense Depot
Memphis, Tennessee

CHEMICAL WARFARE MATERIAL REMOVAL ACTION (2001)

PISTOL RANGE REMOVAL ACTION (2003)

GROUNDWATER INTERIM REMEDIAL ACTION (2009)

CHEMICAL WARFARE MATERIAL REMOVAL ACTION (2001)

NORTHEAST OPEN AREA

DISPOSAL AREA

STOCKPILE AREA

McLean

Person

Carver

Hearst

Hays

State

Ferber

Perry

Dunn

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Figure 12

Dunn Field Removal Actions and Interim Remedial Action

Environmental Restoration Support
Uniform Federal Policy –
Quality Assurance Project Plan

Former Defense Depot
Memphis, Tennessee

Legend

- IRA Recovery Well
- Railroad
- IRA Discharge Conveyance Line
- Area Designations
- Original Dunn Field Boundary
- Removal
- Unrestricted Use
- Paved
- Roads

N
W E
S

0 250 500
Feet

Projection: NAD 1927 StatePlane Tennessee
Units: Feet, Elevation Units: Feet, NAVD88

Date: 3/9/2018
Edition: Rev 1

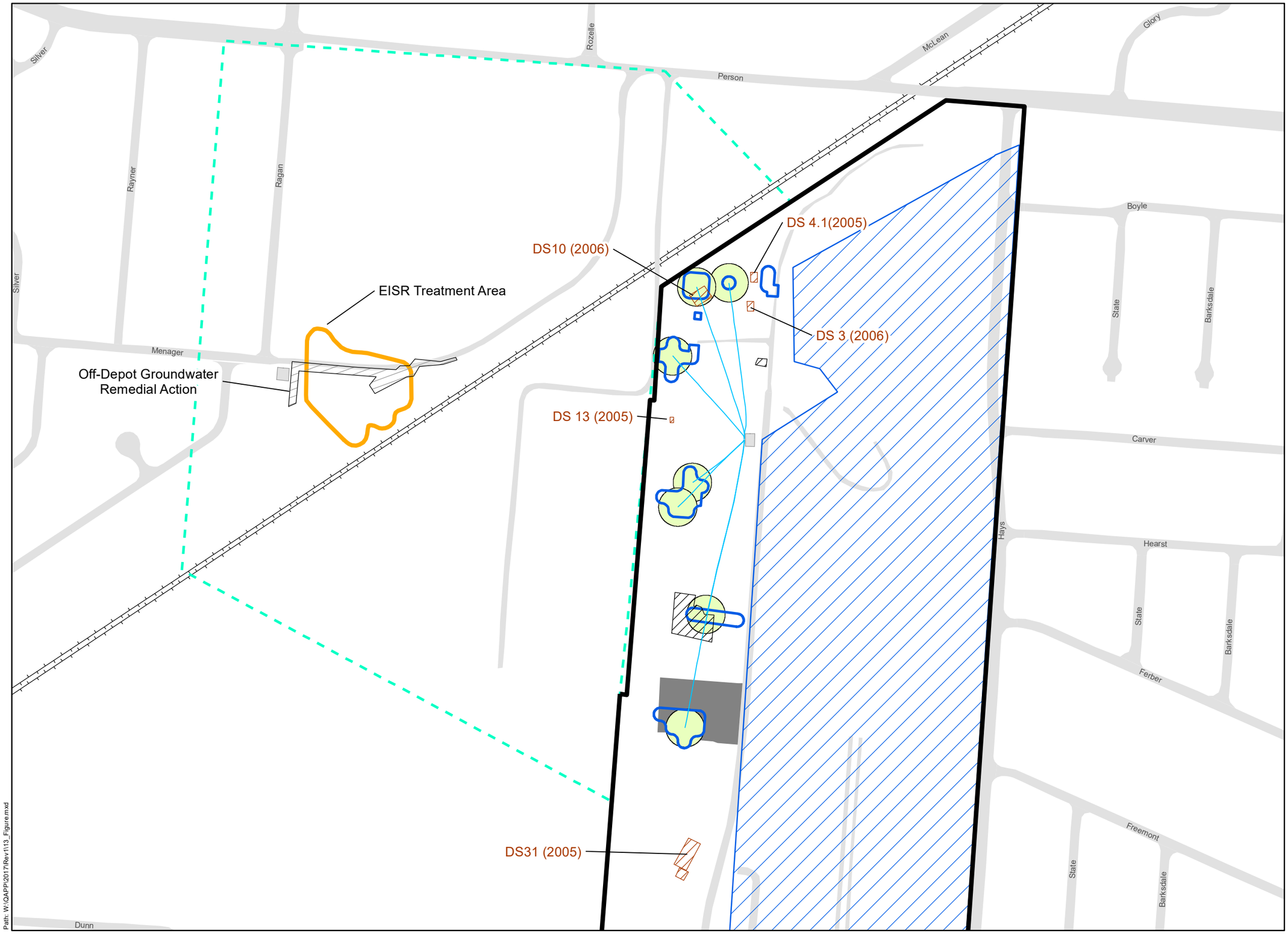

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Figure 13

Dunn Field
Remedial Actions

Environmental Restoration Support
Uniform Federal Policy –
Quality Assurance Project Plan

Former Defense Depot
Memphis, Tennessee

Legend

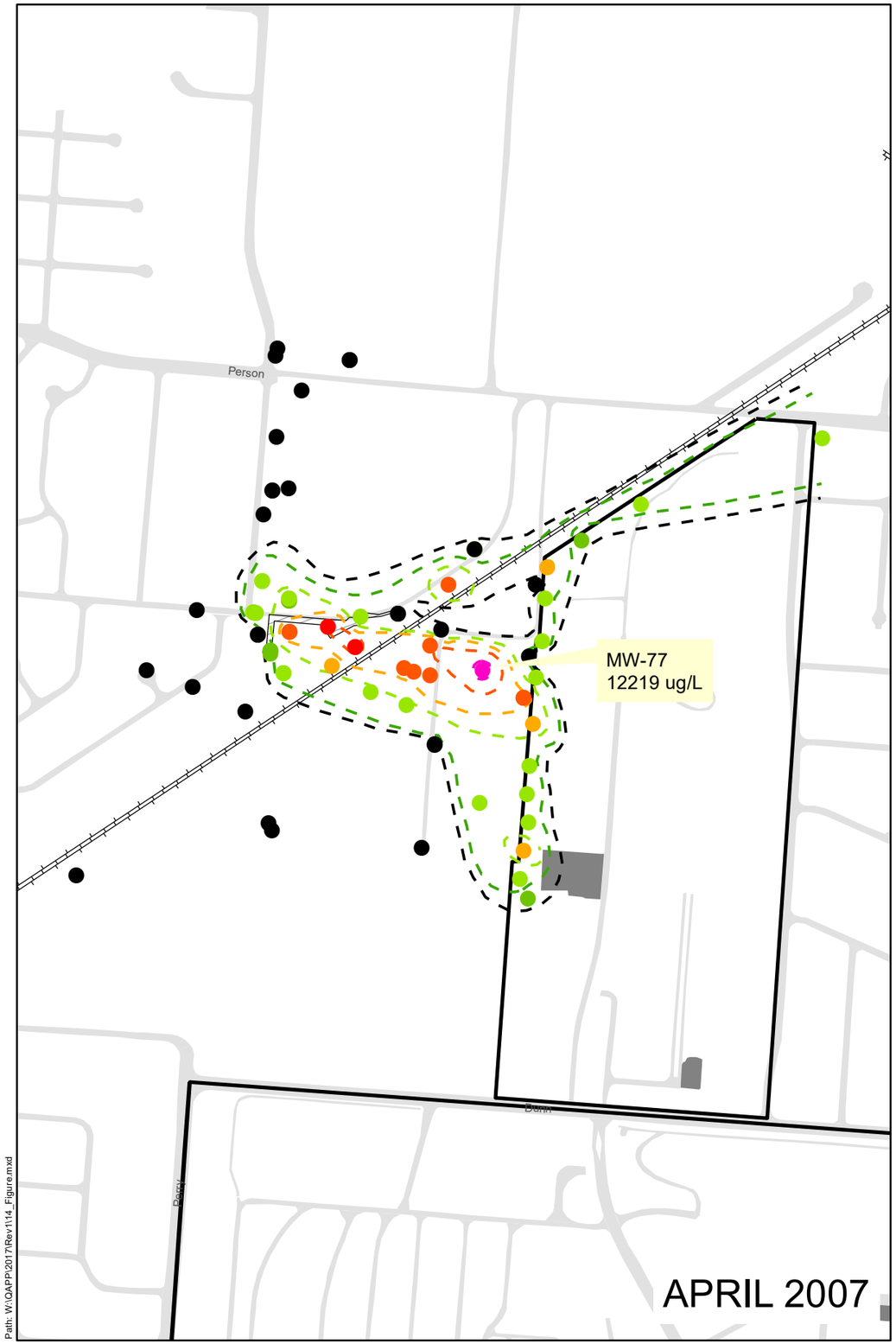
- Original Dunn Field Boundary
- Fluvial SVE Conveyance Line
- Disposal Sites Excavation Area
- Off Site Treatment Areas
- Off Depot Air Sparge Area
- EISR Treatment Area
- *Loess Excavation Areas*
- *Loess Thermal-Enhanced SVE Treatment Areas*
- Fluvial SVE Well 60-foot radius of influence
- Unrestricted Use Area from ROD
- Buildings
- Paved Area
- Roads
- Railroad

N
W E
S

0 250 500
Feet

Projection: NAD 1927 StatePlane Tennessee
Units: Feet, Elevation Units: Feet, NAVD88

Date: 3/9/2018
Edition: Rev 1



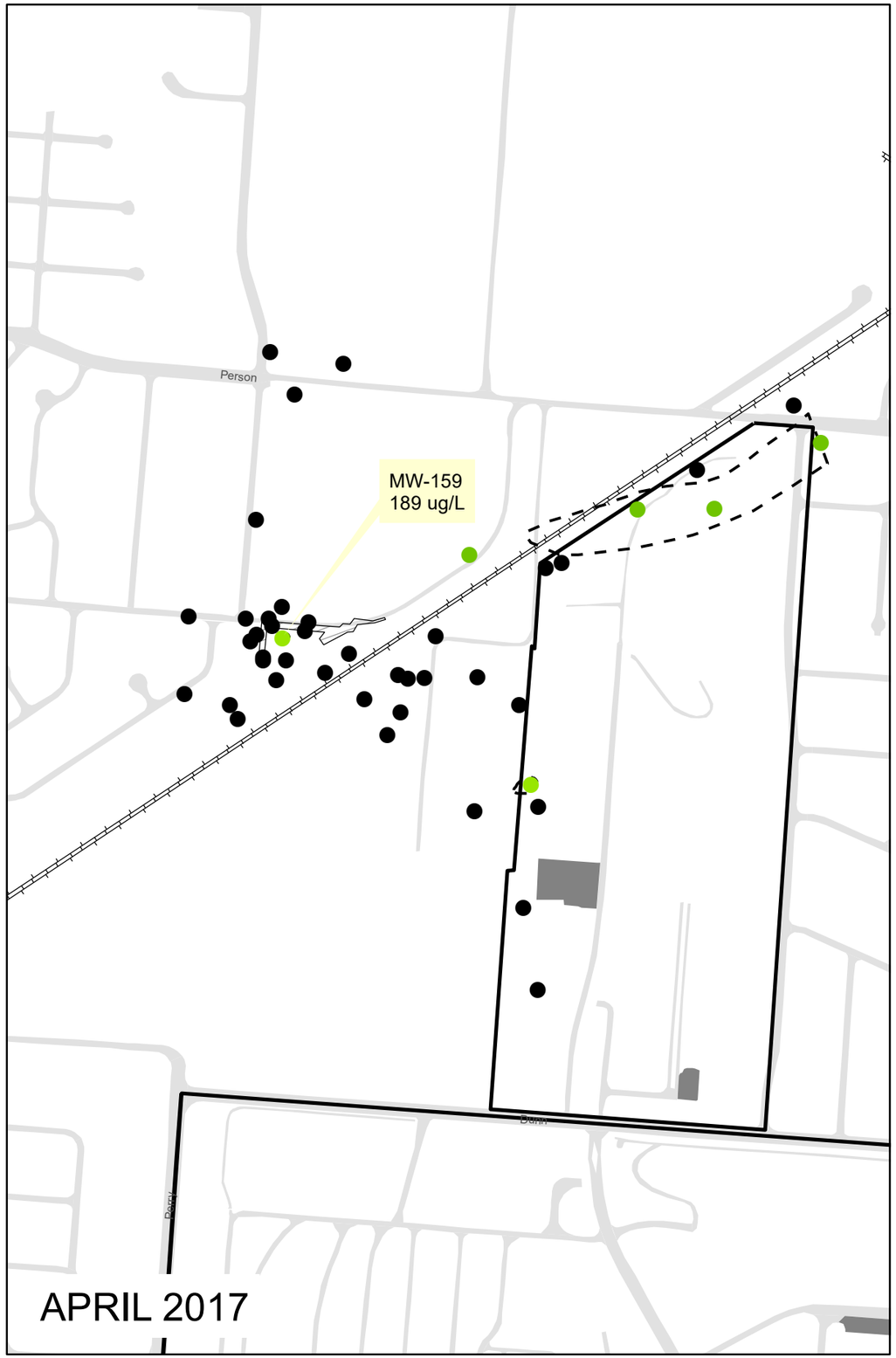


Figure 14

Dunn Field Total CVOC Concentrations, 2007 - 2017

Environmental Restoration Support
Uniform Federal Policy –
Quality Assurance Project Plan

Former Defense Depot
Memphis, Tennessee

Legend

Total CVOC Isopleth (ug/L)

- 0-50
- 50-100
- 100-500
- 500-1000
- 1000-5000
- 5000-10000
- 10000-50000

Total CVOC Ranges (ug/L)

- 50
- 100
- 500
- 1000
- 5000
- 10000
- Original Property Boundary
- Off Depot Air Sparge Area
- Paved Area
- Roads
- Railroad

N
W E
S

0 500 1,000
Feet

Projection: NAD 1927 StatePlane Tennessee
Units: Feet, Elevation Units: Feet, NAVD88

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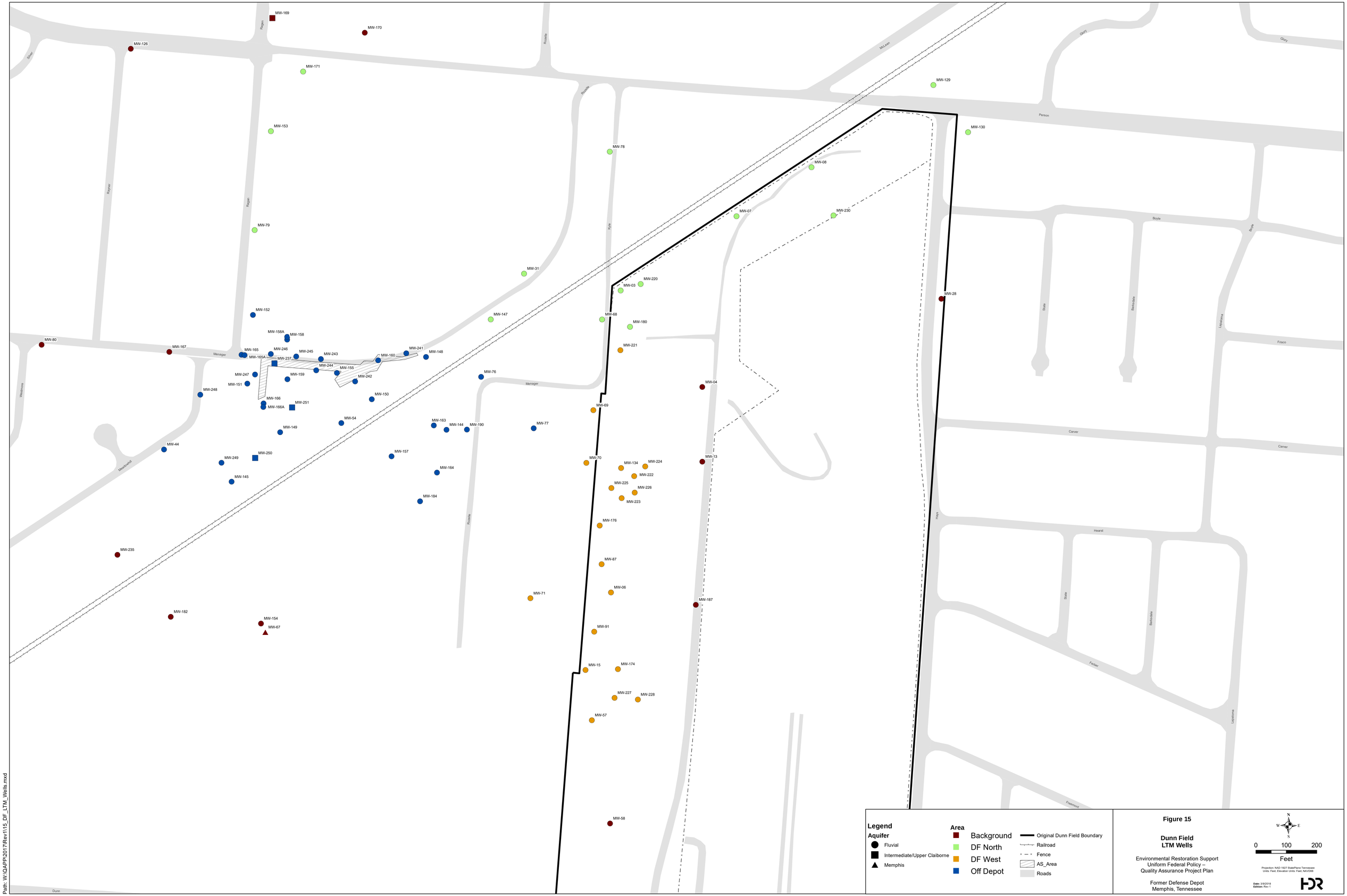

Legend

Aquifer
- Fluvial
- Intermediate/Upper Claiborne
- Memphis

Area
- Background
- DF North
- DF West
- Off Depot

- Original Dunn Field Boundary
- Railroad
- Fence
- AS_Area
- Roads

Figure 15

Dunn Field
LTM Wells

Environmental Restoration Support
Uniform Federal Policy –
Quality Assurance Project Plan

Former Defense Depot
Memphis, Tennessee

0 100 200
Feet

Projection: NAD 1927 StatePlane Tennessee
Units: Feet, Elevation Units: Feet, NAVD88

Date: 3/9/2018
Edition: Rev 1

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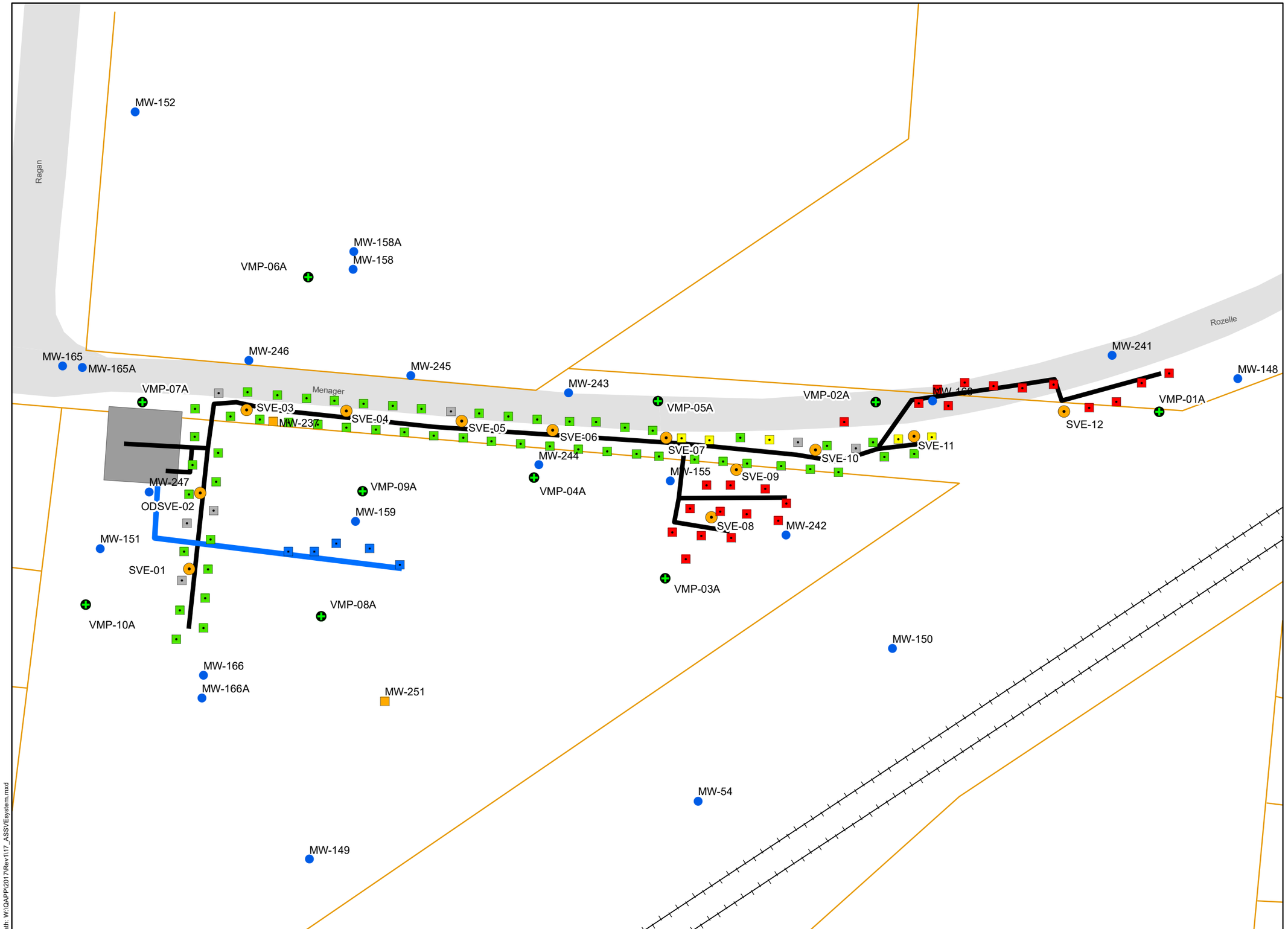

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Figure 17

AS/SVE System

Environmental Restoration Support
Uniform Federal Policy –
Quality Assurance Project Plan

Former Defense Depot
Memphis, Tennessee

Legend

- Monitoring Well Screened in the Fluvial Aquifer
- Monitoring Well Screened in the Intermediate Aquifer
- AS Location (1/2 time)
- AS Location (full time)
- AS Location (closed)
- Closed by leak/damage
- New AS Location
- VMP Location
- SVE Location
- Conveyance Lines
- New Conveyance Line
- SVE Compound
- Roads
- Railroad
- Parcel Boundary

N
W E
S

0 25 50
Feet

Projection: NAD 1927 StatePlane Tennessee
Units: Feet, Elevation Units: Feet, NAVD88

Date: 3/12/2018
Edition: Rev 1

Appendix A
Field Standard Operating Procedures

STANDARD OPERATING PROCEDURE 1 - GENERAL PROCEDURES FOR FIELD PERSONNEL

Lead Organization: Department of the Army (DA)
Preparing Organization: HDR
SOP Approved by: Field Team Leader: Eric North
Project QA Officer: Lynn Lutz
Project Manager: Tom Holmes

1 Purpose

This Standard Operating Procedure (SOP) provides guidance for the general field practices to be followed during field activities at Defense Depot Memphis, Tennessee (DDMT); review is mandatory prior to the start of each field event. This SOP provides general guidance; the project-specific work plan must be reviewed for specific project requirements.

2 Health and Safety

Each individual assigned to field work must participate in the HDR Medical Monitoring Program, must have taken the Occupational Safety and Health Administration (OSHA) 40-Hour course (updated with the 8-Hour OSHA Refresher, when necessary), and must be certified as able to wear respiratory protection.

Each individual is required to have read and understood the project Site Safety and Health Plan (SSHP) for the specific project activity. Upon arrival at the site, each person shall sign the acknowledgement sheet confirming their review of the SSHP. Personal protective equipment (PPE) and other provisions for site safety requirements are discussed in the project specific Health and Safety plan.

All equipment will only be used by properly trained personnel. Only personnel that have received forklift operator safety training are permitted to use the forklift. Proper tools will be made available to each employee as necessary. Any questions should be addressed to the Field Team Leader (FTL).

3 Personnel Qualifications and Responsibilities

Field activities will be directed by the FTL, an environmental professional (engineer, geologist or scientist) with experience in performing and directing the planned activities. Field staff will be junior to mid-level environmental professionals or environmental technicians. Field work will be conducted by persons with experience in performing the planned activities. At least one person on each team will have a current certification in first aid and CPR.

The FTL will provide direction to field staff to ensure work is performed in accordance with the project documents (Quality Assurance Project Plan [QAPP], project work plan and SOPs). The field staff will carefully review the project documents, conduct the work as planned, seek direction from the FTL when questions or problems arise, and carefully complete field documentation.

4 Equipment and Supplies

The required equipment and supplies will be identified in the SOPs for the specific field activities to be performed and in the project work plan. Field activities should not proceed until the proper tools and equipment are available and in good working order.

Each team will have use of a truck/van during field activities. An initial safety check should be performed at the start of each shift to confirm the vehicle is in good working condition. The vehicle should then be checked daily for damage or required maintenance. For each HDR owned vehicle, mileage will be recorded on the vehicle mileage log at the start and end of each field event.

5 Procedure

5.1 Start-Up Activities

5.1.1 Office

Prior to leaving the office for field work, personnel will perform the following actions:

1. The Project Manager (PM) will assign an FTL to direct field activities and coordinate with project personnel. Task specific responsibilities of the FTL will be addressed in the appropriate SOP; general responsibilities include:
    a. Review project work plan, SSHP, and QAPP.
    b. Work with PM to properly staff the field activity.
    c. Coordinate sampling activities with the project chemist and analytical laboratory.
    d. Confirm availability and condition of DDMT-owned equipment and order additional equipment/supplies for delivery prior to the start of each event.
    e. Prepare field forms and other documentation for the planned event.
    f. If work is to be subcontracted, review the subcontract agreement, work plan, and SSHP.
    g. Confirm that field staff have Driver's License (or other picture identification) and current OSHA Certification in their possession prior to leaving the office.

5.1.2 Field

After arrival on site, but prior to commencement of operations, the following activities will be performed:

- Complete equipment and supply checklists and verify that required documentation and equipment for field activities are on site.
- Review condition of DDMT-owned and rental equipment; inventory field supplies and laboratory-provided sampling supplies.

- Review locations for planned field activities for hazards, determine requirements for site preparation and clearance, and select location for the storage of purge and decontamination waters.
- Conduct team safety meetings as required by the SSHP.
- Conduct team review of the project documents including SOPs to be utilized.
- Complete the Field Event Startup Report and submit to PM (Attachment 1-1).

5.2 Field Operations

Field staff responsibilities are project-specific. At a minimum, field personnel will perform the following activities:

1. Document field activities in a log book for each team and/or field records as required by the work plan or SOPs.
2. Record the following additional information for field measurements:
    a. The identification number and calibration results for each field instrument
    b. The numerical value and units of each measurement
    c. A description of any unexpected delays or problems observed during purging or sampling activities
3. Complete required data collection/sample control forms (e.g., Chain-of-Custody, Field Sampling Report, etc.).
4. Communicate with the PM regarding site conditions and out of scope work to be performed.
5. Perform following activities daily before leaving the site:
    a. Decontaminate and check condition of field equipment.
    b. Provide log books and other field documentation to FTL for review and scanning.
    c. Properly dispose of trash, debris and used PPE.
    d. Safely store purge and decontamination water, or transfer to large storage tanks at Dunn Field.
    e. Make arrangements for shipment of samples (if applicable) and follow-up with the analytical laboratory to confirm samples arrived in good condition.
    f. Complete activity-specific field reports as required by applicable SOPs.
    g. Complete the Daily Field Report and submit to PM (Attachment 1-2).

5.3 Field Log Books and Documentation

Dedicated log books will be used by each field team in addition to documentation required by activity-specific SOPs.

- The first page of each log book will list the following information:
    - Site Name: Former Defense Depot Memphis Tennessee

  - EPA ID (TN4210020570)
  - Project Location: 2241 Truitt Street, Memphis, TN 38114
- The first entry for each field event will list the following information: log books:
  - Project Name and Number
  - FTL (full name) and initials
  - Sample team leader and members (full names) and initials
- At minimum, the log book will describe general activities performed, date and time, personnel and weather conditions. All field equipment calibration and maintenance records will be documented in the logbook. Communications with the FTL, PM or project chemist regarding field activities will be documented. Additional field data will be recorded in the log book if other field records are not used.
- Any deviations from the QAPP or work plan will be noted in the log books.
- Errors will be crossed out with a single line, the correction added and the entry initialed.
- Each page will be numbered and dated. A diagonal line will be drawn through any unused portion of a page containing an entry. To indicate the end of an entry, personnel are required to initial and date the page at the conclusion of each day.

5.4 Closeout

Upon the completion of field activities, the FTL will view each site to verify the area has been cleared and restored as closely as possible to its prior condition. Trash will be removed from the site, and surface damage, including ruts caused by vehicles, will be repaired.

Confirm all equipment is accounted for and properly decontaminated and in good working condition. Notify PM if repairs are needed. Properly package and ship all rental equipment to the vendor. When shipping equipment, use the proper HDR FedEx number and insure the package for the cost of the equipment. Follow manufacturer's instructions on long and short term storage when storing government and/or HDR equipment.

Rental trucks should be fueled and returned to the rental company as soon as possible. HDR leased trucks should also be fueled and cleaned prior to storing at the shop.

Work areas should be cleaned with tools and equipment properly stored.

The FTL will make a final check of all logbooks and other field records to ensure there are no blanks or missing data and the entries are legible. FTL will organize scanned forms in proper order and transmit to PM.

The FTL will complete Field Event Closeout Report and submit to PM (Attachment 1-3).

6 Data and Records Management

All field forms and log book entries will be scanned and copied to the project folder on the HDR network file share drive within one week of the field event completion. All photographs taken during the field event will be uploaded along with a typed photograph log (date, project and subject) to the HDR network file share drive. The photographs will then be erased from the camera. All original forms will be stored on site in Memphis in the filing cabinet in the proper folder labeled for the project. The PM, project chemist and project administrator will be sent a link for the data.

7 Quality Control and Quality Assurance

All work will be performed in accordance with the QAPP, the specific work plan, and applicable SOPs. All field activities will be recorded in the log books in sufficient detail to reconstruct the events. No erasures or mark outs will be made on field forms or log books. A single line will be used to strike out errors and will be annotated with the initials and date of the editor.

8 References

HDR, 2017a. *2017 Uniform Federal Policy-Quality Assurance Project Plan, Environmental Restoration Support at Former Defense Depot Memphis, Tennessee, Revision 0*. Prepared for the U.S. Army Corps of Engineers, Mobile District. May 2017.

USEPA Region 4 SESD Guidance, *Soil Sampling* (SESDPROC-010-4). October, 2010.

Field Event Startup Report

Prepared by: Date:

Event Name:

Project-Activity Number:

Summary of Planned Event:

Planned Performance Period: _____to_____

Project Documents - Title, Date

Work Plan:

Health and Safety Plan:

Other SOPs – List number/revision and title:

Field Event Staffing

Position	Name	OSHA Cert. (Y/N)	First Aid/ CPR (Y/N)	Driver's License (Y/N)	Proj. Plans reviewed (Y/N)	Experience (Hi-Med-Low-None)
Field Team Leader						

DDMT Field Equipment

Name/Use	Mfr./Model No.	Condition	Calibration Req'd.(Y/N)	Calibration supplies	Other supplies (batteries, etc.)

Rental Equipment

Name/Use	Mfr./Model No.	Condition	Calibration Req'd.(Y/N)	Calibration supplies	Other supplies (batteries, etc.)

Lab-provided Sampling Supplies

Sample Type	Number	Supplies

Additional Tools/Supplies

Camera
Field forms (list):
Sample supplies (list):
Water/Ice cooler
Sample cooler

Final Check

1. All required equipment/tools received and condition checked

Yes ___ No ___ Comment:

2. Initial equipment calibration completed

Yes ___ No ___ Comment:

3. Vehicles inspected

Yes ___ No ___ Comment:

4. Field locations reviewed

Yes ___ No ___ Comment:

5. Weather forecast checked

Yes ___ No ___ Comment:

6. Staff documents (OSHA, DL) checked

Yes ___ No ___ Comment:

7. Review of project plans confirmed and activities discussed

Yes ___ No ___ Comment:

8. Initial Safety Meeting held and SSHP signed

Yes ___ No ___ Comment:

Daily Field Report

Project Number/Activity:	**Date:**
Project Name:	**Field Team Leader:**
Brief Work Description:	

Weather:	**Temp:**

Previous Day's Samples received at laboratory – Y / N Comment:

Time	Description

Name/Organization of Field Staff, Subcontractors and Site Visitors	

Samples Collected

Problems or Deviations from Work Plan

Tasks to be completed next workday

_____ _____ _____

Name **Signature** **Date**

Field Event Close-Out Report

Prepared by: Date:

Event Name:

Project-Activity Number:

Performance Period: _____to_____

Field Team Leader:

Field Staff:

Summary of Completed Event:

Field problems and/or changes from planned activities:

Change in number/type of samples collected:

Health and Safety problems/Injuries:

Close-out Checklist

1. Log book and field forms scanned and originals placed in project file

Yes ___ No ___ Comment:

2. Equipment/tools decontaminated

Yes ___ No ___ Comment:

3. Rental equipment shipped to supplier

Yes ___ No ___ Comment:

4. Rental vehicles returned

Yes ___ No ___ Comment:

5. DDMT equipment and tools properly stored

Yes ___ No ___ Comment:

6. List damaged equipment

Yes ___ No ___ Comment:

7. Replacement supplies ordered

Yes ___ No ___ Comment:

8. Field locations inspected and trash/debris removed

Yes ___ No ___ Comment:

9. Field shop/office cleaned

Yes ___ No ___ Comment:

STANDARD OPERATING PROCEDURE 2 – DRILLING AND SOIL SAMPLING

Lead Organization: Department of the Army (DA)
Preparing Organization: HDR
SOP Approved by: Field Team Leader: Eric North
Project QA Officer: Lynn Lutz
Project Manager: Tom Holmes

1 Purpose and Summary

This Standard Operating Procedure (SOP) provides guidance for drilling and soil sampling operations in support of investigative activities at Defense Depot Memphis, Tennessee (DDMT). Drilling activities will enable collection of subsurface soil samples and allow the installation of monitoring wells. This SOP provides general guidance; the project-specific work plan must be reviewed for specific project requirements.

2 Overview

There are several methods by which drilling operations may be conducted including, manual (hand) augering, power augering with hollow-stem augers, sonic drilling, and cable tool or mud rotary drilling with installation of surface casing. Generally, hand augering is useful only for surficial soil sampling while the other methods are used for deeper, subsurface investigations, sampling and installation of monitoring wells. Sonic drilling is the recommended drilling method for well installation at DDMT; it has proven to be the most effective method for boring advancement and well installation based on the depth to water (i.e. 75-105 feet below ground surface [ft bgs]) and geologic characteristics of the fluvial aquifer (i.e. tight sands mixed with gravel up to cobble size).

Drilling activities that require the use of a truck-mounted drill rig will be conducted by a Tennessee-licensed subcontractor with experience on similar projects. The drilling subcontractor will advance boring to the target depth using the selected drilling technology and provide equipment sufficient to carry out the work as specified. Drilling and soil sampling will be overseen by the field team leader (FTL), an environmental professional (engineer, geologist or scientist), with support staff if required. HDR personnel will prepare soil boring logs with lithologic descriptions and observations relevant to investigative activities, collect soil samples for field or laboratory analysis and monitor compliance with the project Site Safety and Health Plan (SSHP).

3 Health and Safety

Proper safety precautions must be observed during drilling activities and when collecting soil samples in accordance with the SSHP. Each individual assigned to field work must: (1) participate in the HDR Medical Monitoring Program, or subcontractor medical surveillance program, as applicable, (2) must have taken the Occupational Safety and Health Administration (OSHA) 40-Hour course (updated with the 8-Hour OSHA Refresher, when necessary), and (3) must be certified as able to wear respiratory protection.

Each individual is required to have read and understood the SSHP for the specific project activity. Upon arrival at the site, each person shall sign the acknowledgement sheet confirming their review of the SSHP. Personal protective equipment (PPE) and other provisions for site safety requirements are discussed in the SSHP. At a minimum for drilling all personnel will wear a hard hat, steel toe shoes, safety glasses, hearing protection, and a high visibility outer garment.

All equipment will only be used by properly trained personnel. In particular, the use of a photoionization detector (PID) will only be performed by personnel familiar with the equipment. Proper tools will be made available to each employee as necessary. Any questions should be addressed to the FTL.

All drilling locations will be cleared for underground and above ground utilities prior to beginning drilling activities. Prior to setting up on the drilling location, the FTL will confirm the location has been cleared with the appropriate utility companies and the property owner/tenant. Drilling will only proceed where no aboveground or subsurface obstructions exist. Locations will be offset if these obstructions are identified prior to drilling, or encountered after drilling has begun. The new locations will be as close as possible to the originally proposed locations; utility clearance will be performed again as necessary.

If drilling is to occur in the vicinity of overhead utilities, HDR personnel will measure utility line height from the ground surface using a clinometer (or similar device) to ensure a minimum safe clearance distance is maintained between on-site equipment and overhead utility lines. As needed, the appropriate utility company will be contacted in order to determine a recommended safe clearance distance from all aboveground or underground on-site utilities.

Prior to the start of drilling activities, the drilling subcontractor will hand auger at each drilling location to a depth of 4 ft bgs, in order to verify that no underground utilities or objects are present.

4 Personnel Qualifications and Responsibilities

Field activities will be directed by the FTL, an engineer/geologist with experience in the planned drilling activities; junior to mid-level geologists will assist, if necessary. Field activities will be overseen by a Tennessee-licensed geologist or engineer. Drilling will be conducted by a licensed driller and crew familiar with planned activities, the project-specific work plan and SSHP. At least one person on each team will have a current certification in first aid and CPR. Operation of fork lifts on site will be limited to personnel that have documentation for forklift operator safety training.

The FTL will provide direction to field staff to ensure work is performed in accordance with the project documents (Quality Assurance Project Plan [QAPP], project-specific work plan, SSHP, and SOPs). The field staff will carefully review the project documents, conduct the work as planned, seek direction from the FTL when questions or problems arise, and carefully complete field documentation.

5 Equipment and Supplies

The required equipment and supplies will be identified in the project-specific work plan. Field activities should not proceed until the proper tools and equipment are available and in good working order. Usual equipment/supplies for a drilling project will include: a PID, tape measure, Munsell color chart, knife, nitrile gloves, field drill log forms, camera, and work table.

Each team will have use of a truck/van during field activities. An initial safety check should be performed at the start of each shift to confirm the vehicle is in good working condition. The vehicle should then be checked daily for damage or required maintenance.

6 Procedures

6.1 Start-Up Activities

6.1.1 Office

Prior to leaving the office for field work, personnel will perform the following actions:

1. The Project Manager (PM) will assign a FTL to direct field activities and coordinate with project personnel. Task specific responsibilities of the FTL will be addressed in the appropriate SOP; general responsibilities include:
    a. Review project-specific work plan, SSHP, QAPP, and for subcontracted work, review of the subcontract agreement.
    b. Work with PM to properly staff the field activity.
    c. Arrange site access with the property manager (Colliers International-Memphis Depot Industrial Park), tenants and/or property owners.
    d. Have a surveyor locate the proposed drilling locations, and mark each location with a wooden stake and white flagging or white paint.
    e. Notify the Tennessee One Call underground utility location and, if necessary, a private utility location service.
    f. Provide drilling subcontractor with proposed boring location and depth for well permits from Shelby County Health Department (SCHD); confirm receipt of permits.
    g. Coordinate sampling activities and supplies with the project chemist and analytical laboratory.
    h. Confirm availability and condition of DDMT-owned equipment and order additional equipment/supplies for delivery prior to the start of each event.
    i. Prepare field forms and other documentation for the planned event.
    j. Provide all HDR and subcontracted field personnel with time and location for personnel to meet prior to beginning field activities.
    k. Confirm that field staff have a valid Driver's License (or other picture identification) and current OSHA Certification in their possession prior to leaving the office.

6.1.2 Field

After arrival on site, but prior to commencement of operations, the following activities will be performed:

- Complete equipment and supply checklists and verify that required documentation and equipment for drilling and soil sampling activities are on site.
- Notify SCHD prior to start of drilling activities in accordance with permit requirements.
- Review condition of DDMT-owned and rental equipment; inventory field supplies and laboratory-provided sampling supplies.
- Confirm drilling and soil sampling locations are clearly marked and review locations for hazards; determine if the utility locators have adequately marked utilities on the site. Check for overhead dangers such as power lines, and make necessary height measurements to ensure safe clearance distances are maintained.
- Determine requirements for site preparation and clearance, and select location for the placement of the decontamination area, storage of decontamination waters, and soil cuttings.
- Confirm locations and requirements for each sample to be collected.
- Conduct site set up activities to include posting of signage (if applicable) and delineation of work zones as required in the SSHP.
- Calibrate field equipment.
- Conduct team safety meetings as required by the SSHP.
- Conduct team review of the project documents including SOPs to be utilized.
- Complete the Field Event Startup Report and submit to PM.

6.2 Field Operations

Field staff responsibilities are project-specific. At a minimum, field personnel are required to ensure the following items are completed as part of field operations during drilling and soil sampling activities.

6.2.1 Field Documentation

Field activities will be documented in a bound logbook for each team and in field records as required by the project-specific work plan or SOPs. At minimum, the logbook will describe general activities performed, date and time, personnel performing the activity, and weather conditions.

For field measurements, the following additional information will be required:

- The numerical value and units of each measurement
- The identity of and calibration results for each field instrument

For sampling activities, the following additional information will be required:

- Sampling type and method

- The identity of each sample and the depth(s) from which it was obtained
- The amount of each sample
- Sample description (e.g., color, odor, clarity)
- Identification of sampling devices
- Identification of conditions that might reflect representativeness of a sample (e.g., refueling operations, damaged well casings)

Field personnel will complete required data collection/sample control forms (e.g., Chain-of-Custody [COC], Drill logs, Field Sampling Report, etc.).

6.2.2 Drilling Logs

The geologist/engineer will log the subsurface conditions encountered in the boring, and record the information on the drilling log and the logbook. Additional pertinent information will be recorded on the drilling log, including, but not limited to, the following:

- Drilling date
- Drilling method
- Geologist name
- Location of boring/Boring identification
- Driller's name/Drilling subcontractor name/Type of drill rig
- Diameter of inner and outer sonic drill casings
- Diameter of surface casing, casing type and method of installation
- Types of drilling fluids and depths at which they were used
- Weather conditions
- Start and completion time for each boring
- Standard Penetration Test blow counts per six inch advance, if applicable
- Recovery length of each sample
- Visual description of soil using the Unified Soil Classification system (ASTM-D-2488-00)
- Depths at which each soil sample was collected for chemical or physical analysis
- Total number of samples taken
- Total depth of boring
- Boring refusal
- Water losses (if applicable)
- Water bearing strata (depth and thickness)
- Depth at which saturated conditions were first encountered

- Lithologic descriptions and depths of lithologic boundaries
- Zones of caving or heaving
- Depths at which drilling fluid was lost and amount lost
- Drilling rate
- Drill rig reactions such as chatter, rod drops, or bouncing
- Location of the boring relative to an easily identifiable landmark.

6.2.3 Drilling Procedures

Generally, drilling activities will be completed in accordance with the planned activities presented in project work plan. Additionally, the following requirements will apply to drilling activities at DDMT:

- Drilling will conform to Shelby County rules and regulations, and Rules of Tennessee Department of Environment and Conservation (TDEC), Division of Water Supply, Chapter 12-4-10.
- All necessary precautions will be taken to prevent leakage of hydraulic oil or other contaminants from the drilling rig into the borehole or onto equipment that is placed in the hole.
- The only acceptable drilling fluid to be used while advancing the borehole is water. However, water will be used only when necessary as approved by the FTL, and will be from an approved potable water source. If the onsite subcontractor and HDR personnel determine drilling fluid additives (e.g. sodium bentonite) are necessary for drilling operations, PM authorization must be obtained prior to their use.
- During drilling of boreholes with a sonic rig, soil will be collected continuously as 10-foot sections of soil core. These cores will be deposited from the drill casing into 10-foot polyethylene liners; the liners will be laid out for visual logging and sampling for headspace readings and laboratory analysis.
- The drilling subcontractor will place all soil cores on the ground near the drill rig in order for the HDR geologist/engineer to safely examine, log, and collect samples from the recovered soil core.
- The HDR geologist/engineer will maintain visual and verbal communication with the onsite subcontracted driller in order to maintain awareness of any changes in subsurface conditions, amount of water used (if any) during drilling, quantities of materials used during drilling and well installation, or any mechanical problems with the drill rig or support equipment.
- The HDR geologist/engineer will carefully and thoroughly complete all required field documentation in order to provide a complete record of drilling activities, including drill rig maintenance and repairs, subcontractor down time, subsurface conditions and geologic materials encountered.
- The HDR geologist/engineer will determine and record the depth to groundwater observed during drilling.

- When the HDR geologist/engineer is finished with visual logging and sampling of a given 10 foot section of soil core, the drilling subcontractor will place the core in an approved soil cutting disposal container.
- During drilling activities, the drilling subcontractor will notify the onsite HDR geologist/engineer of any significant changes in lithology encountered, significant changes in amount of water being used, and any mechanical problems with the drill rig.
- The HDR geologist/engineer will monitor the breathing zone for organic vapors in accordance with the procedures contained in the SSHP. The tops of the boreholes will be monitored for organic vapors using a PID.
- The HDR geologist/engineer collect soil samples at specified intervals in borings for soil classification and/or chemical analysis or field screening as specified in the project-specific work plan.
- All drilling equipment will be decontaminated prior to drilling activities in accordance with SOP 9 *Equipment Decontamination*.
- Any investigative-derived waste (i.e., drill cuttings, drilling fluid) that is contaminated will be disposed as specified in the project work plan.
- Soil cuttings will be examined for contamination. If contamination is suspected, they will be noted on the boring log form and the suspect soil cuttings will be segregated.
- The HDR geologist/engineer will communicate with the PM regarding site conditions and out of scope work to be performed.

6.2.4 Boring Diameter

The boring diameter is based on a minimum of 2 inches of annular space between the outside diameter of the well casing and the borehole wall. The majority of borings and wells at DDMT are completed in the fluvial aquifer, which is underlain by the uppermost clay of the Jackson Formation/Upper Claiborne Group. For these borings, a 6-inch diameter borehole is advanced 5-10 feet into the clay; after the depth to the clay is confirmed, the boring is back-filled to just below the top of clay or to the target well depth. A borehole diameter of 6 inches allows proper installation of a nominal 2-inch outside diameter well casing.

For wells to be installed in the deeper intermediate or Memphis aquifer, a surface casing is typically installed into the uppermost clay of the Jackson Formation/Upper Claiborne Group in order to prevent cross contamination between formations. For the deeper borings, a 12-inch borehole will be advanced 10 feet into the uppermost clay and an 8-inch diameter surface casing will be installed, either welded sections of carbon steel or threaded Schedule 80 polyvinyl chloride (PVC). After placing the surface casing, the driller will lower a galvanized or PVC tremie pipe connected to a grout pumping unit through the inner annulus of the casing. The driller will pump grout through the injection pipe until the grout returns to the ground surface. The grout will cure for 24 hours before continuing to advance the borehole. Water present in the inner annulus of the casing will be pumped to a holding tank before the borehole is advanced to the target depth. A 6-inch diameter borehole will then be advanced to the target depth for installation of a 2-inch diameter well.

6.2.5 Soil Sampling Procedures

During drilling of boreholes with a sonic rig, soil samples will be collected continuously as 10-foot sections of soil cores. These cores are deposited from the drill casing into 10-foot polyethylene liners, and the liners laid out for visual logging, and to obtain samples for headspace readings and laboratory analysis, if required by the project work plan.

During advancement of the soil borings, the following sampling devices may also be used:

- Chemical Sample Collection: 2 or 3-inch diameter carbon steel split-barrel sampler lined with California brass rings (CBRs)
- Geotechnical Sample (disturbed) Collection: 2-inch diameter carbon steel split-barrel sampler
- Geotechnical Sample (undisturbed) Collection: 3-inch diameter “Shelby Tube” or thin-walled tube sampler

6.2.5.1 Soil Description

Soils will generally be described in accordance with the 1990 ASTM D-2488-90, *Standard Practice for Description and Identification of Soils* (Visual-Manual Procedure). Descriptive information to be recorded in the field will include:

- Identification of the predominant particle size and range of particle sizes
- Percent of gravel, sand, fines, or all three
- Description of grading and sorting of coarse particles
- Particle angularity and shape
- Maximum particle size or dimension

The plasticity of fines description will include:

- Color using Munsell Color System
- Moisture (dry, wet, or moist)
- Consistency of fine grained soils
- Structure of consolidated materials
- Cementation (weak, moderate, or strong)

The Unified Soil Classification System (USCS) group symbols will be used for identification. Additional information to be recorded includes: depth to the water table, caving or sloughing of the borehole, changes in drilling rate, depths of laboratory sample collection, presence of organic materials, presence of fractures or voids in consolidated materials, and other noteworthy observations or conditions, such as the locations of geologic boundaries.

6.2.5.2 Headspace Sampling

At five-foot intervals within the soil cores, the headspace will be screened with a PID. The headspace samples will be collected and analyzed using the following procedure:

- From the sampling location within the soil core, remove the top 1 to 2 inches of soil using a decontaminated stainless steel spoon.
- Partially fill two decontaminated 16-ounce containers with soil using the stainless steel spoon.
- Cover the jars immediately with aluminum foil and fasten the jar lids.
- Allow the sample vapors to equilibrate in the jars (approximately 5 minutes). If necessary, the headspace samples will be brought to a temperature of 20 degrees Celsius (°C) (68 degrees Fahrenheit [°F]) to 32°C (90°F)
- Collect a reading from the first sample jar by puncturing the aluminum foil with the tip of a calibrated PID and recording the highest reading.
- If the reading is > 10 parts per million, collect a reading with the activated charcoal filter on the calibrated FID for the second jar. Determine corrected hydrocarbon measurement of the sample by subtracting the filtered reading from the unfiltered reading.

6.2.5.3 Soil Sample Collection for Laboratory Analysis

Selected soil samples may be collected for laboratory analysis based upon the results of the headspace screening. At these selected locations, samples for volatile organic compound (VOC) analysis will be collected using an Encore or Terracore sampler, or acceptable equivalent. (Note: There is no difference in field criteria for the two samplers. Different laboratories supply different devices and there is a difference in cost.) Samples collected for VOC analysis should be collected from the soil cores in a manner that minimizes disturbance of the sample.

The following items should be considered when collecting soil samples:

- A clean pair of new, non-powdered, disposable gloves will be worn each time a sample is collected.
- Samplers must use new, verified/certified-clean disposable or non-disposable equipment cleaned in accordance with SOP 9 *Equipment Decontamination*.
- Document field sampling, including field conditions, any problems encountered during sampling and sample appearance, in the field logbook. Samples collected will also be noted on the drilling log sheet at the corresponding depth.
- Place any unused sample material into the approved transport/disposal containers along with other drill cuttings generated during sonic drilling activities.
- When soil sampling is completed or when time permits, transfer samples to site office for final packaging. Complete COC documentation and shipping procedures in accordance with relevant SOPs. The completed COC will remain with the samples until custody is relinquished.
- Note any problems encountered during sampling in the Field Sampling Report Form and Daily Quality Control Report Form.

- For borings where a monitoring well will be installed, a sample for total organic carbon (TOC) analysis may be collected from the interval to be screened. The TOC samples will be collected from the soil core using a pre-cleaned stainless steel spoon and placed in the appropriate laboratory supplied container.

6.2.5.3.1 Encore ™ Sampler Procedure

The procedure for collection of VOC samples using an Encore ™ Sampler are as follows:

- Remove sampler and cap from package and attach T-handle to the 5-gram sampler body.
- Quickly push the sampler into a freshly exposed surface of soil until the sampler is full.
- Carefully wipe the exterior of the sampler head with a clean disposable paper towel so that the cap can be tightly attached.
- Push cap on with a twisting motion to attach and seal the sampler.
- Attach the label onto the sampler body, place the sampler into a plastic Ziploc™ bag and place into a cooler with ice.
- Repeat steps a) through e) for the remaining 5-gram and 25-gram sampler.
- Collect a bulk soil sample for screening and moisture determination in a 2 or 4-ounce wide mouth glass jar. Fill the jar completely allowing no headspace. Place the sample in a cooler containing ice.
- Thoroughly mix remaining soil and place into specified labeled containers for remaining parameters.
- Place sample bottles into Ziploc™ or bubble bag and in an iced cooler.
- Complete COC documentation and shipping procedures in accordance with relevant SOPs.

6.2.5.3.2 Terracore Sampler Procedures

The procedure for collection of VOC samples using a Terracore Sampler are as follows:

- Label appropriate laboratory containers
- Quickly push the sampler (Terracore or equivalent) into a freshly exposed surface of soil to collect 5 grams (+ 0.5g) of sample. Also collect a bulk aliquot container for moisture content analysis in the laboratory supplied 4 ounce container.
- Carefully wipe the exterior of the sampler head with a clean disposable paper towel.
- Empty sampler into appropriate laboratory container. The cored samples must be extruded from the selected coring tool to a volatile organic analysis (VOA) vial in accordance with collection and preservation methods described in EPA method 5035A. The extruded core is transferred into a laboratory pre-weighed (tared) VOA vial with septum cap. Unpreserved VOA vials must be analyzed within 48 hours of collection, VOA vials preserved with sodium bisulfate or methanol must be analyzed within 14 days of collection.
- Place the sample into a plastic Ziploc™ bag and place into a cooler with ice.
- Complete COC documentation and shipping procedures in accordance with relevant SOPs.

6.2.6 Post Run Tubing Boring Construction

The post run tubing (PRT) drill rod will be advanced into the subsurface to 5.5 feet bgs by a direct-push drill rig. After reaching 5.5 feet bgs, the PRT rod will be retracted approximately 6 inches exposing the soil interval (5 feet bgs to 5.5 feet bgs) to be sampled. Teflon® tubing will be threaded into the PRT adaptor through the center of the PRT rod and capped to prevent soil gas venting. The annulus around the PRT rod where it penetrates ground surface will be packed with bentonite crumbles and hydrated. The boring will not be disturbed or sampled for a minimum of 2 hours to allow the bentonite crumbles to seal the annulus and allow soil gas to equilibrate. After the soil gas sample has been collected, the PRT drill rod and tubing will be removed from the boring and the boring will be filled to ground surface with neat cement.

6.3 Closeout

6.3.1 Daily Closeout

Perform following activities daily before leaving the site:

- Decontaminate and check condition of field equipment.
- Provide logbooks and other field documentation to FTL for review.
- Properly dispose of trash, debris and used PPE.
- Make arrangements for shipment of samples (if applicable) and follow-up with the analytical laboratory to confirm samples arrived in good condition.
- Secure the site for the night and/or weekend.
- Prepare the daily field report as required by the project-specific work plan or SOPs and submit report to the PM. Note any problems or deficiencies in field activities.

6.3.2 Field Event Closeout

Upon completion of field activities, the FTL will view each site to verify the area has been cleared and restored as closely as possible to its prior condition. The following activities will be performed prior to the completion of each field event:

- All trash will be removed from site and disposed of appropriately
- Any damage to the ground surface, including ruts, will be repaired
- All equipment is accounted for, properly decontaminated, and in good working condition. The FTL will be notified if repairs are needed
- Rental equipment has been properly cleaned, packaged, and shipped to the appropriate vendor
- Shipments are made using the correct HDR FedEx number and packages insured for the cost of the rental item
- Manufacturer’s instructions are followed regarding long and short term storage for all equipment

- Rental vehicles are refueled and returned to the rental company
- HDR leased vehicles are cleaned and refueled
- All work areas have been cleaned, and tools and equipment have been stored properly

The FTL will make a final check of all drilling logs, logbooks and other field records to ensure there are no blanks or missing data and the entries are legible. The FTL will complete Field Event Closeout Report and submit to PM.

7 Data and Records Management

All field forms and logbook entries will be scanned and copied to the project folder on the network file share drive within one week of the field event completion. All photographs taken during the field event will also be uploaded along with a typed photograph log (date, project and subject) to the network file share. All uploaded photographs will then be erased from the camera. All original forms will be stored on site at the field office in Memphis in the appropriate project-specific filing cabinet and task-specific labeled folder.

8 Quality Control and Quality Assurance

All work will be performed in accordance with the QAPP, the project-specific work plan, and applicable SOPs. All field activities will be recorded in the logbooks in sufficient detail to reconstruct the events. No erasures or mark outs will be made on field forms or logbooks. A single line will be used to strike out errors and will be annotated with the initials and date of the editor. Boring logs will be typed into a spreadsheet provided by the CAD operator for the inclusion into computerized drill logs.

9 References

HDR, 2017a. *2017 Uniform Federal Policy-Quality Assurance Project Plan, Environmental Restoration Support at Former Defense Depot Memphis, Tennessee, Revision 0*. Prepared for the U.S. Army Corps of Engineers, Mobile District. May 2017.

Shelby County Health Department, Pollution Control Section, Water Quality Branch, <http://www.shelbycountytn.gov/DocumentCenter/Home/View/767>.

USEPA Region 4 SESD Guidance, *Design and Installation of Monitoring Wells* (SESDGUID-101-R1), January, 2013.

USEPA Region 4 SESD Guidance, *Field Equipment Cleaning and Decontamination* (SESDPROC-205-R2), December, 2011.

USEPA Region 4 SESD Guidance, *Soil Sampling* (SESDPROC-300-R3), August, 2014.

STANDARD OPERATING PROCEDURE 3 – WELL INSTALLATION, DEVELOPMENT AND ABANDONMENT

Lead Organization: Department of the Army (DA)
Preparing Organization: HDR
SOP Approved by: Field Team Leader: Eric North
Project QA Officer: Lynn Lutz
Project Manager: Tom Holmes

1 Purpose and Summary

This Standard Operating Procedure (SOP) provides guidance for installation, development and abandonment of monitoring wells at Defense Depot Memphis, Tennessee (DDMT). This SOP provides general guidance; the project-specific work plan must be reviewed for specific project requirements.

2 Overview

Monitoring wells will be installed, developed and abandoned by a Tennessee-licensed subcontractor and supervised by an HDR geologist/engineer. Well installation and development will occur immediately after drilling and preparations should be made prior to beginning drilling operations, which are described in SOP 2 *Drilling and Soil Sampling*. This SOP incorporates past practice at DDMT as described in work and test procedures (WTPs) from the RA SAP (MACTEC, 1995) and SOPs prepared by United States Environmental Protection Agency (USEPA) Region 4.

3 Health and Safety

Proper safety precautions must be observed during drilling activities and when collecting soil samples in accordance with the site-specific Health and Safety Plans (HASPs). Each individual assigned to field work must: (1) participate in the HDR Medical Monitoring Program, or subcontractor medical surveillance program, as applicable, (2) must have taken the OSHA 40-Hour course (updated with the 8-Hour OSHA Refresher, when necessary), and (3) must be certified as able to wear respiratory protection.

Each individual is required to have read and understood the HASP for the specific project activity. Upon arrival at the site, each person shall sign the acknowledgement sheet confirming their review of the HASP. Personal protective equipment (PPE) and other provisions for site safety requirements are discussed in the HASP. At a minimum for drilling all personnel will wear a hard hat, steel toe shoes, safety glasses, hearing protection, and a high visibility outer garment.

All equipment will only be used by properly trained personnel. In particular, the use of a photoionization detector (PID) will only be performed by personnel familiar with the equipment. Proper tools will be made available to each employee as necessary. Any questions should be addressed to the Field Team Leader (FTL).

4 Personnel Qualifications and Responsibilities

Field activities will be directed by the FTL, a mid- or senior level engineer/geologist with experience in monitoring well installation, development and abandonment; junior to mid-level geologists will assist, if necessary. Field activities will be overseen by a Tennessee-licensed geologist or engineer. Field activities will be overseen by a Tennessee-licensed geologist or engineer. The well installation, development and/or abandonment will be conducted by a TN-licensed driller and crew familiar with planned activities, the project-specific work plan and HASP. At least one person on each team will have a current certification in first aid and CPR. If a fork lift is used on site the person driving the fork lift will have the proper Occupational Safety and Health Administration (OSHA) training.

The FTL will provide direction to field staff to ensure work is performed in accordance with the project documents (Quality Assurance Project Plan [QAPP], project-specific work plan HASP, and SOPs). The field staff will carefully review the project documents, conduct the work as planned, seek direction from the FTL when questions or problems arise, and carefully complete field documentation.

5 Equipment and Supplies

The required equipment and supplies will be identified in the project-specific work plan. Field activities should not proceed until the proper tools and equipment are available and in good working order. Usual equipment/supplies for a monitoring well installation, well development, and well abandonment will include: a PID, tape measure, knife, nitrile gloves, well pump, compressor, grout mixer, grout pump, bleach, sand, bentonite, Portland cement, well construction forms, well abandonment forms, camera, and development water containers.

Each team will have use of a truck/van during field activities. An initial safety check should be performed at the start of each shift to confirm the vehicle is in good working condition. The vehicle should then be checked daily for damage or required maintenance.

6 Procedures

6.1 Start-Up Activities

6.1.1 Office

Prior to leaving the office for field work, personnel will perform the following actions:

1. The Project Manager (PM) will assign a FTL to direct field activities and coordinate with project personnel. Task specific responsibilities of the FTL will be addressed in the appropriate SOP; general responsibilities include;
    a. Review project project-specific work plan, HASP, and QAPP and for subcontracted work, review of the subcontract agreement.
    b. Work with PM to properly staff the field activity.

c. Arrange site access with the Memphis Depot Associates, tenants and/or property owners.

d. Confirm availability and condition of DDMT-owned equipment and order additional equipment/supplies for delivery prior to the start of each event.

e. Prepare field forms and other documentation for the planned event.

f. Prepare the required Shelby County Health Department (SCHD) well installation and abandonment forms.

g. Provide all HDR and subcontracted field personnel with time and location for personnel to meet prior to beginning field activities.

h. Confirm that field staff have a valid Driver's License (or other picture identification) and current OSHA Certification in their possession prior to leaving the office.

6.1.2 Field

After arrival on site, but prior to commencement of operations, the following activities will be performed:

- Complete equipment and supply checklists and verify that required documentation and equipment for field activities are on site.
- Review condition of DDMT-owned and rental equipment, and inventory field supplies.
- Review locations for planned field activities for hazards, including overhead dangers such as power lines, and select location for the placement of the decontamination area, storage of decontamination and development waters.
- Confirm the exact locations of the wells to be abandoned and that the correct well is being abandoned.
- Confirm the location and length of the screened interval and the total depth of the well to be installed and developed.
- Conduct site set up activities to include posting of signage (if applicable) and delineation of work zones as required in the HASP.
- Calibrate field equipment.
- Conduct team safety meetings as required by the HASP.
- Conduct team review of the project documents including SOPs to be utilized.
- Complete the Field Event Startup Report and submit to PM.

6.2 Field Operations

Field activities will be documented in a logbook for each team and in field records as required by the project-specific work plan or SOPs. At minimum, the logbook will describe general activities performed, date and time, personnel and weather conditions. Additional information will be recorded in the log book if other field records are not used.

6.2.1 Monitoring Well Installation

Monitoring well installation will be completed in a manner consistent with relevant sections of USEPA Region 4 SESD Guidance, *Design and Installation of Monitoring Wells* (SESDGUID-101-R1), and applicable state/local requirements.

Monitoring well installation will be conducted by a licensed driller and well installation subcontractor. A qualified geologist/engineer will oversee well installation activities.

The following information will be required as part of the field documentation.

- The length of risers, screens, and end caps for each monitoring well including adjustment for riser sections cut off during installation.
- Record the type, manufacturer, and gradation of the filter sand, and the volume used for each well.
- The type and manufacturer of the Portland cement and bentonite and the volume used for the bentonite seal and grout at each well.
- Record surface completion details including: completion type, number of bollards installed, and a description of surface completion materials.

Borings for monitoring wells will be advanced using sonic drilling. The following procedure will be used to install the well casing and screen:

- If the boring was drilled deeper than the total depth of the well, backfill the boring to approximately 1 foot below the planned well depth in accordance with the work plan, either with bentonite or by allowing the formation material to collapse as the casing is raised.
- Remove the new polyvinyl chloride (PVC) or stainless steel screen and riser from manufacturer packaging and decontaminate as described in SOP9 *Equipment Decontamination*.
- Install a 10 to 20-foot section of minimum 2-inch (I.D.), threaded, flush jointed, pre-manufactured PVC or stainless steel screen inside the steel drill casing.
- Install solid riser to ground surface, plus stick-up (if required).
- Install the filter pack using the gravity method through the annular opening between drill casing and well screen as the drill casing is removed. Continue removing drill casing and installing filter pack until at least 4 feet above the top of the well screen. Use the sonic drilling head to vibrate the steel casing as it is slowly withdrawn to distribute and compact the filter pack around the screen and to prevent bridging. Measure the thickness of the filter pack as it is placed.
- Install a minimum 5-foot bentonite seal. If bentonite is gravity fed in dry form, the seal will be hydrated with potable water. Allow the bentonite seal a minimum of 4 hours of hydration time before grouting the annulus. If the seal is in the saturated section of if potential for bridging is an issue, a bentonite slurry can be installed using a side-discharge tremie pipe.
- Remove remaining drill casing and grout boring annulus to ground surface with grout/bentonite mixture.

- Develop the well at least 24 hours after grout installation.

6.2.1.1 Well Construction Materials

Well risers will consist of material durable enough to retain their long-term stability and structural integrity and be relatively inert to minimize alteration of groundwater samples. Selection of PVC or stainless steel for the monitoring wells is based on the primary purpose of the well, which is the detection of potential contaminants, and site-specific conditions, such as planned remedial actions.

Well materials will consist of new, threaded, flush joint PVC or stainless steel pipe, with a minimum inside diameter of 2 inches. If PVC is used, the riser pipe will conform to ASTM D 1785, Standards for Schedule 40 Pipe; deeper wells installed in the intermediate or Memphis aquifers require Schedule 80 Pipe. Materials will be new and unused and will be decontaminated prior to installation. Casing will only be joined with compatible welds or couplings that do not interfere with the primary purpose of the well. Use of solvent or glue will not be permitted.

Well screens will consist of new, commercially fabricated, threaded, flush joint, minimum 2-inch inside diameter (ID), factory slotted or continuous wrap PVC or stainless steel screen. Screen slot size will be based on previously available soil information, but will be generally sized to prevent 90 percent of the filter pack from entering the well. The screen slot size will be adjusted if site geologic conditions significantly differ from the expected conditions. Previous well installation at DDMT have generally used factory-slotted or wire-wrapped screens with 0.010-inch openings, no less than 10-feet in length, and no greater than 20-feet in length.

Silt traps will not be used in monitoring wells. A notch will be cut in the top of the casing to be used as a measuring point for water levels.

6.2.1.2 Well Design

Monitoring wells will be designed and installed in a manner to accomplish the following objectives: to collect representative groundwater samples; to prevent contamination of the aquifer by the drilling equipment; to prevent vertical seepage of surface water or inter-aquifer contamination.

Well design includes placement of the screen, installation of filter pack, bentonite seal, and grout seal. The FTL and PM will collectively make decisions on well depths, locations, screened intervals, etc. Borings at DDMT are generally drilled 10-feet into the clay unit at the base of aquifers to confirm the local presence of the lower confining unity. Well screens are generally set above the clay at the base of the aquifer; the deeper portion of the boring is filled with bentonite or formation material.

The well pipe assembly will be hung in the borehole, prior to placement of the filter pack, and not allowed to rest on the bottom of the hole to keep the well assembly straight and plumb. Centralizers will be installed at roughly 30-foot intervals beginning above the bentonite seal.

6.2.1.2.1 Screen Location

The screened intervals will be selected for each proposed well, based on visual observations of aquifer materials encountered, as recorded on the drilling log, and objectives in the project work plan. There are several water bearing units of interest at DDMT (fluvial, intermediate, and Memphis aquifer). Both the fluvial and intermediate aquifers can be found in unconfined conditions, with

significant saturated thickness (>50 feet). In many areas, the saturated thickness of the fluvial aquifer is 20 feet or less. For most wells at DDMT the screen will start from the top of clay upward, for a maximum of 20 feet of screen per well. If the saturated thickness is substantially greater than 20 feet, cluster (or nested) wells may be installed to screen the entire saturated interval.

6.2.1.2.2 Filter Pack

A filter pack will be installed in the annular space between the boring and the well screen. The filter pack will consist of clean, inert, well rounded silica sand and contain less than 2 percent flat particles. The filter pack will be certified as free of contaminants by the supplier and have a grain size distribution compatible with the formation materials and the screen.

A filter pack size of (20-40) is generally used based on site conditions at DDMT. This sand size was determined from grain-size analysis of the screened intervals by previous consultants at the site. If the site conditions show significant change (i.e. more gravelly, or much more clayey) from those previously encountered a grain-size analysis will be completed and filter pack design based on those results.

The filter pack will be placed from the bottom of the hole to a minimum of 4 feet above the top of the well screen. The filter pack will not extend across more than one water-bearing unit. When sonic drilling methods are used, the filter pack will be emplaced through the nominal 6-inch diameter steel casing using the gravity method.

Prior to installation of the well casing, the total depth of the borehole depth will be measured from the top of the 6-inch steel drill casing by the drilling contractor to verify that the target depth has been reached. The sand filter pack will be gravity-placed through the 6-inch steel casing in lifts of approximately 1 foot. Care will be taken to prevent bridging by frequently measuring the thickness of the filter pack as it is placed. As the steel casing is slowly withdrawn between lifts, it will be vibrated with the sonic drilling head to compact the sand filter pack.

6.2.1.2.3 Bentonite Seal

A minimum 5-foot thick bentonite seal will be installed above the filter pack in the annular space of the well. Only 100 percent sodium bentonite (pellets or chips) will be used and care will be taken to prevent bridging by frequently measuring the thickness of the bentonite as it is gravity placed. When the seal is installed above the water table, the bentonite will be hydrated with water from an approved water source. At least 5 gallons of water will be added after each 24 to 30 inches of bentonite is placed. The bentonite seal will be allowed to hydrate for a minimum of 4 hours prior to placement of the grout collar around the wells. When the seal is placed below the water table, a bentonite slurry may be installed using a side-discharge tremie pipe.

6.2.1.2.4 Grout Seal

A non-shrinking cement-bentonite grout mixture will be placed in the annular space from the top of the bentonite seal to approximately 6-inches below the ground surface. Concrete will be added in the remaining annular space during installation of the protective casing and concrete pad.

The cement-bentonite mixture will consist of 94 pounds of neat Type I Portland or American Petroleum Institute (API) Class A Cement, not more than four pounds of 100 percent sodium bentonite powder, and not more than 8 gallons potable water. A side discharge tremie pipe will be used to place the grout mixture into the annular space. The tremie pipe will be located a maximum of 10 feet from the top of the bentonite seal in deep wells to ensure even placement of grout in the annular space. Pumping will continue until undiluted grout is visible at the surface.

6.2.1.2.5 Surface Completion

Surface completion (flush-mount or stick-up) will be selected by the PM based on well location and planned land use. For flush-mount completions, the casing will be cut approximately 3 inches below ground surface and secured with a water-tight locking cap to prevent surface water from entering the well. The casing will be covered by a bolted manhole cover set in a 3-foot by 3-foot by 4-inch thick concrete pad that slopes away from the manhole.

If an aboveground surface completion is used, the well casing will be extended 2 or 3 feet above ground surface and secured with a water-tight cap. The protective casing will be a steel sleeve placed over the casing and cap; the steel sleeve diameter will be at least 4 inches greater than the casing diameter. The protective casing will be set in a 3-foot by 3-foot by 4-inch concrete surface pad. A vent hole will be drilled in the steel sleeve about 1 inch above the top of the well pad. The pad will be sloped away from the well sleeve and a lockable cap or lid will also be installed. Three 3-inch diameter concrete-filled steel guard posts will be installed around each well unless the well is located in an area receiving vehicular traffic. These guard posts will be 5 feet in total length and installed radially from the well head. The guard posts will be installed approximately 2 feet into the ground and set in concrete just outside the concrete pad. The protective sleeve and guard posts will be brush-painted yellow or orange.

Wells will be secured immediately after well completion. Corrosion-resistant locks will be provided for both flush and aboveground surface completions. A brass survey marker will be installed in each concrete pad and the well ID will be stamped in the marker.

6.2.1.2.6 Location Survey

Following installation of the surface completion for each well, the wells will be surveyed for horizontal locations and elevations at top of casing, ground surface and well pad by a Tennessee-licensed surveyor. Vertical coordinates will be based on the North American Datum, 1927 used for all survey data at DDMT. Horizontal coordinates will be provided in the Tennessee State Plane coordinate system. Accuracy for well locations will be within 0.01 foot for elevations and within 0.1 feet for horizontal coordinates.

6.2.1.3 Well Installation Diagrams

The HDR geologist/engineer will maintain suitable logs detailing drilling and well construction practices. Well dimensions, amount, type and manufacture of materials used to construct each well will be recorded in the logbook. Additional information to be recorded in the field for the well installation diagram will include:

- Well identification.

- Drilling method.
- Installation date(s).
- Total boring depth.
- Lengths and descriptions of the screen and riser.
- Thickness and descriptions of filter pack, bentonite seal, annular grout, and any backfilled material.
- Quantities of all well construction materials used.

6.2.2 Well Development

The purpose of well development is to create good hydraulic contact between the well and the aquifer and to remove accumulated sediments from the well. Each newly installed monitoring well will be developed no sooner than 24 hours after installation to allow for adequate grout curing time. The water volume purged during development will exceed the volume of potable water or other drilling fluids used during drilling and well installation.

The wells will be developed with a surge block in conjunction with a pump sized to effectively develop the well. No detergents, soaps, acids, bleaches, or additives will be used during well development. Development will continue until clear, sand-free formation water is produced from the well and until pH, conductivity, turbidity, and temperature measurements have stabilized. Stabilization is defined when the pH is within + or - 0.1, the conductivity is + or - 5%, and the turbidity is less than 10 nephelometric turbidity units (NTUs).

The monitoring well development protocol is as follows:

- Measure the static water level (SWL) and the depth to the top of sediment in the well.
- Record the total depth of the well (from the Well Installation Diagram).
- Calculate the volume of water in the well and saturated annulus.
- Begin developing the well using a combination of surging and pumping. Continue pumping and periodically surging until each the following criteria have been met:
    - Fluids lost to the formation during drilling and well installation have been removed (this is a minimum requirement where conditions permit).
    - pH, temperature, turbidity, and specific conductance have stabilized. In general, field parameters are stable when NTUs are less than 10, pH is within 0.1 on consecutive readings, and temperature and specific conductance are within 10 percent of previous readings. Natural turbidity levels in ground water may exceed 10 NTU.
    - If feasible, monitor the SWL during purging. Adjust the purge rate to keep the SWL from dropping more than 0.3 meter from the initial SWL.
    - No sediment remains in the bottom of the well. However, it can be accepted if the sediment thickness remaining within the well is less than 1 percent of the screen length.
- In the event that the above criteria have not been met after six hours of pumping, surging, and bailing (including recharge time for poorly recharging wells), development activities will

be temporarily discontinued at that well. The FTL and PM will decide whether or not to continue development.

- In the event of slowly recharging wells that will not sustain pumping or bailing, the field staff will advise the FTL as soon as a determination of estimated recharge time has been made.
- Physical characteristics of the water (suspended sediment, turbidity, temperature, pH, EC, purge rate, odor, etc.) will be recorded throughout the development operation. At a minimum, they will be recorded initially and after each well volume has been removed, or every 30 minutes, whichever comes first.
- The total quantity of water removed and final depth to the top of sediment (total depth of well) will be recorded.
- Well development equipment will be decontaminated prior to use in each newly-installed monitoring well.

6.2.2.1 Well Development Records

Well development data will be recorded on Well Development Data Sheets, which should include the following information:

- Method of development.
- The model number and type of water quality instruments.
- The model and type of water pump used for development.
- The flow rate of the pump.
- The type and technique used for surging of the well.
- Final water quality description (e.g., color, odor, clarity).
- Identification of conditions that might reflect the results of the development if it was successful or why it was not.
- Volume of water removed from the well.

6.2.2.2 Well Development Water

Development water will be drummed or stored in bulk containers. The containers will be clearly labeled with site name, well name, date, and contents. The development water will be properly disposed in accordance with investigation derived waste (IDW) procedures set forth in the project work plan.

6.2.3 Well Abandonment

Monitoring wells at DDMT are reviewed annually with regard to classification, sample frequency and utility. Wells are recommended for abandonment based on the following criteria:

1. The well is redundant: duplicates information; not in the flow pathway of on-coming plumes and not required to establish background; or analytical data will have no clear, reasonable use in future decision making.

2. The monitoring well (MW) has sustained damage and cannot be repaired, or an object that cannot be removed has become lodged in the MW.
3. The MW was installed for a specific reason that no longer applies.

Wells are scheduled for abandonment after recommendations are approved by USEPA and Tennessee Department of Environment and Conservation (TDEC).

Well abandonment will be completed in accordance with SCHD requirements following issuance of a fill and abandonment construction permit from SCHD. Well abandonment will be conducted by a TN-licensed well contractor. An HDR geologist/engineer will oversee well abandonment activities. The following procedure will be used for well abandonment:

- Total well depths will be measured and compared to depths recorded during well installation to determine if obstructions are present in the well.
- One-half gallon of bleach will be poured into the well as a disinfectant.
- The well screen and casing will be filled with grout (Portland type II cement with 5 percent bentonite) from the bottom up using a tremie pipe. After allowing the grout time to settle, additional grout will be added to fill the well casing to approximately 6 inches below ground surface (bgs).
- Surface completions including well pads and manholes will be removed at wells located in grassed or graveled areas. If necessary, the well casing will be cut off a few inches below the ground surface. The pad areas will be recovered with either topsoil/grass seed or gravel. At wells located in concrete or asphalt-paved areas, the manhole covers will be removed and the manholes filled with concrete. Bollards will be removed at all abandoned wells.
- Surface completion materials including manholes, bollards, well lids and wells casings will be placed in a roll-off and properly disposed.

The following information will be recorded to document the well abandonment:

- The total depth of the abandoned wells and whether obstructions had to be removed.
- The amount and type of Portland and bentonite used for grouting.
- The volume of grout used to fill the well casing and the volume of water recovered during grouting.
- Disposal of surface completion materials removed during well abandonment.

6.3 Closeout

6.3.1 Daily Closeout

Perform following activities daily before leaving the site:

- Decontaminate and check condition of field equipment.
- Provide log books and other field documentation to FTL for review.
- Properly dispose of trash, debris and used PPE.

- Secure the site for the night and/or weekend.
- Prepare daily report as required by the project-specific work plan or SOPs and submit report to the PM. Note any problems or deficiencies in field activities.

6.3.2 Field Event Closeout

Upon completion of field activities, the FTL will view each site to verify the area has been cleared and restored as closely as possible to its prior condition. Trash will be removed from the site, and surface damage including ruts caused by vehicles, will be repaired.

Confirm all equipment is accounted for and properly decontaminated and in good working condition. Notify FTL if repairs are needed. Properly package and ship all rental equipment to the vendor. When shipping equipment, use the proper HDR FedEx number and insure the package for the cost of the equipment. Follow manufacturer's instructions on long and short term storage when storing government and/or HDR equipment.

Rental trucks should be fueled and returned to the rental company as soon as possible. HDR leased trucks should also be fueled and cleaned prior to storing at the shop.

Work areas should be cleaned with tools and equipment properly stored.

The FTL will make a final check of all logbooks and other field records to ensure there are no blanks or missing data and the entries are legible.

The FTL will complete Field Event Closeout Report and submit to PM.

7 Data and Records Management

All field forms and logbook entries will be scanned and copied to the project folder on the network file share drive within one week of the field event completion. All photographs taken during the field event will also be uploaded along with a typed photograph log (date, project and subject) to the network file share. All uploaded photographs will then be erased from the camera. All original forms will be stored on site at the field office in Memphis in the appropriate project-specific filing cabinet and task-specific labeled folder.

Well logs and sample results for new wells will be submitted to the SCHD in accordance with permit requirements.

8 Quality Control and Quality Assurance

All work will be performed in accordance with the QAPP, the project-specific work plan, and applicable SOPs. All field activities will be recorded in the logbooks in sufficient detail to reconstruct the events. No erasures or mark outs will be made on field forms or logbooks. A single line will be used to strike out errors and will be annotated with the initials and date of the editor. Well completion diagrams will be typed into a spreadsheet provided by the CAD operator for the inclusion into computerized well diagrams.

9 References

MACTEC, *RA SAP Volume I: Field Sampling Plan, Defense Depot Memphis, Tennessee, Revision 1*, *WTP-3 Well Installation, Development and Sampling*. November, 2005.

Shelby County Health Department, Pollution Control Section, Water Quality Branch, <http://www.shelbycountytn.gov/DocumentCenter/Home/View/767>.

USEPA Region 4 SESD Guidance, *Design and Installation of Monitoring Wells* (SESDGUID-101-R1), January, 2013.

STANDARD OPERATING PROCEDURE 4 – GROUNDWATER SAMPLE COLLECTION

Lead Organization: Department of the Army (DA)
Preparing Organization: HDR
SOP Approved by: Field Team Leader: Eric North
Project QA Officer: Lynn Lutz
Project Manager: Tom Holmes

1 Purpose and Summary

This Standard Operating Procedure (SOP) provides guidance for groundwater sample collection at Defense Depot Memphis, Tennessee (DDMT). The project work plan must be reviewed for specific requirements.

2 Health and Safety

General Information on Health and Safety requirements are provided in SOP 1. Each individual is required to have read and understood the Health and Safety Plan (HASP) for the specific project activity and signed the acknowledgement sheet confirming their review.

Health and safety concerns for groundwater sampling include the use of lead-acid batteries with bladder pumps, contact with contaminated groundwater, and contact with sample container preservatives. Material safety data sheets (MSDS) will be available on site for each chemical to be utilized during sampling activities. Staff will wear appropriate personal protective equipment (PPE), as outlined in the site safety health plan. Many of the wells are located in or near streets and parking lots with traffic; field staff should wear vests with reflective stripes or other high visibility clothing while sampling. Some wells may be located in areas with biological threats such as spiders, fire ants, snakes, and wasp nests; the wells should be checked for hazards before starting sampling activities.

3 Personnel Qualifications and Responsibilities

Groundwater sampling will be directed by a Field Team Leader (FTL), a mid- or senior level environmental professional (engineer, geologist or scientist) with appropriate experience. Field staff will be junior to mid-level environmental professionals or environmental technicians overseen by the FTL. Sampling will be performed by two-person teams and at least one person on each team will have a current certification in first aid and CPR.

4 Equipment and Supplies

The required equipment and supplies will be identified in the work plan for the specific field activities to be performed. Field activities should not proceed until the proper tools and equipment are available and in good working order. Usual equipment/supplies for groundwater sampling will

include: a photoionization detector (PID), nitrile gloves, pump controller, portable bladder pump, compressor, water quality meter, water level indicator tape, camera, and purge water containers.

Each team will have use of a truck/van during field activities. An initial safety check should be performed at the start of each shift to confirm the vehicle is in good working condition. The vehicle should then be checked daily for damage or required maintenance.

5 Procedure

5.1 Start-Up Activities

5.1.1 Office

The Project Manager (PM) will assign a FTL to direct field activities and coordinate with project personnel. General responsibilities are described in SOP 1. Task specific responsibilities include:

- Coordinate sampling activities with the PM, project chemist (PC) and analytical laboratory.
- PC will prepare the sampling plan detail (SPD) listing the wells and sample bottles for planned analyses. FTL will review the SPD, discuss any questions with PC and confirm shipment of laboratory-supplied sample containers and equipment for arrival prior to the start of sampling.
- The FTL will update the list of wells to be included in the water level sweep. An example list is provided in Attachment 4-1.
- Confirm availability and condition of DDMT-owned equipment and order additional equipment/supplies for delivery prior to the start of sampling event.
- Obtain well location maps and prepare tables showing screened interval and previous water level measurements to confirm planned sample depths.
- Prepare field forms and other documentation for the planned event.
- Schedule time and location for the initial meeting with field staff to review project information and begin work.

5.1.2 Field

After arrival on site, but prior to commencement of operations, the following activities will be performed:

- Complete equipment and supply checklists and verify that required documentation and equipment for field activities are on site.
- Review condition of DDMT-owned and rental equipment; inventory field supplies.
- View well locations and confirm the wells are accessible and well IDs are clearly marked.
- Review locations for planned field activities for hazards. Determine requirements for site preparation and clearance, and select location for storage of decontamination and purge waters. Confirm sufficient storage capacity for wastewater.

- Confirm the location and length of the screened interval and the total depth of the well to be sampled if not equipped with a diffusion bag.
- Conduct site set up activities to include posting of signage (if applicable) and delineation of work zones as required in the HASP.
- Review sampling activities and assignments with field staff.

5.2 Field Operations

Field records will be prepared in accordance with SOP 7 – Sample Control and Documentation. Each sampling site will be characterized by the following factors:

- Location of work
- Weather conditions: rainfall, temperature, and wind direction
- Ongoing activities that may influence or disrupt sampling efforts
- Accessibility to the sampling locations (e.g., rough terrain, fallen trees, flooding, etc.)

5.2.1 Water Level Sweep

Prior to sampling, a water level sweep will be made at listed monitoring wells to produce an accurate potentiometric map.

1. Determine if the water level probes are working properly by using two or more in one well to confirm the same depth is measured. If the depths differ by more than 0.1 feet, determine which one is malfunctioning and replace it for the project.
2. Using the water level sweep list proceed to the wells requiring water level readings. Confirm the well location by checking the well ID on the pad.
3. Inspect the area around the well for hazards, then remove the well box lid, lock and well cap. Assess the well condition and note cracks in the pad, missing bolts, missing caps, etc. in the field log.
4. Allow the water level at each well to equilibrate for at least 3 minutes after removing the well cap and prior to measurement. Multiple wells within an area can be inspected and opened to make efficient use of field time, but all open wells must be within clear sight of field staff.
5. Note appearance of positive or negative pressure in casing when cap is removed (air pressure lifting cap or suction on cap). If pressure is noted, re-measure water level at least 3 minutes after the initial measurement; if the two measurements vary by more than 0.05 feet, make a third measurement.
6. Turn the water level indicator on and slowly lower it into the well until it alerts to the water level.
7. Bring up the probe slowly until the beeping stops and slowly lower it again until it beeps do this three times and record the average level recorded. All readings should be taken at the location marked on top of the casing; if no mark is present, use the north side of the casing.
8. Depth measurement should be recorded to the nearest 0.01 feet.

9. Put the cap and lock back on the well casing and then close the well box.
10. Decontaminate the water level probe before proceeding to the next well. The decontamination procedure for the water level indicator is: Hand wash the calibrated tape and probe with Alconox solution (or equivalent) and rinse with deionized (Reagent Grade II) water.

5.2.2 Water Quality Measurements

Field measurements of groundwater physical parameters are used for groundwater sampling and for independent measurements during remedial actions. The field equipment will be properly calibrated per manufacturer's instructions; calibrations will be made at the start of the day after lunch and at the end of the day. The calibrations will be checked during the day if abnormal measurements are observed. All calibration activities will be recorded in the field log books.

Field measurements will be made with multi-probe device with flow-through cell. Flow cells add efficiency to low flow purging and field sampling applications. Follow calibration procedures provided in the operations manual.

Groundwater samples will be collected when the water level and water quality indicators of dissolved oxygen (DO), pH, specific conductivity, and turbidity stabilize. Readings will be taken every 5 to 10 minutes and recorded on the Sample Collection Data sheet (Attachment 4-2).

There should be only a slight and stable drawdown of the water column after pumping begins. The pumping rate should be adjusted to reduce drawdown, if possible. If the water level does not stabilize, the sample will be collected after water quality indicators stabilize and at least three well casing volumes are removed, or the well is purged dry.

Stabilization of water quality indicators is achieved after three successive readings are within ± 0.1 for pH, ± 5% for specific conductance, ± 10% for DO, and <10 nephelometric turbidity units (NTU) for turbidity. Temperature and redox potential (ORP) will be measured and recorded, but will not be used as a stabilization parameter. Sampling may begin once the well has stabilized. If stabilization does not occur or turbidity cannot be reduced below 10 NTU, the FTL should be contacted for direction.

5.2.3 Sample Collection Procedures

Groundwater samples are collected from monitoring wells using passive diffusion bags (PDBs) where the saturated screened interval is 5 ft or greater and field water quality measurements are not required. Samples are collected with portable bladder pumps where PDBs are not installed, or with a disposable Teflon® bailer if there is insufficient water depth for the pump. Decontamination of portable pumps is required prior to each use in accordance with SOP 9.

For new wells, sampling will be performed no less then 24 hours after well development is completed. Observations made during sample collection will be recorded in the logbook and on a monitoring well purge and sampling form. The following initial steps will be followed before collecting groundwater samples in the field.

1. Locate the well to be sampled, confirm well ID and record the condition of the well.

2. Caution shall be used when opening each well to avoid fumes which may have accumulated and to prevent foreign materials from entering the well. If a persistent odor is observed, air monitoring with a PID will conducted in accordance with the HASP.
3. Measure the water level from the measuring point to the nearest 0.01-foot and record the measurement in the field logbook and on the Sample Collection Data sheet (Attachment 4-2)
4. Water levels will be measured before and during sampling. The water level probe should be carefully lowered down the well to minimize disturbance.
5. Decontaminate the water-level indicator and tape prior to use in a well. The decontamination procedure for the water level indicator is: Hand wash the calibrated tape and probe with Alconox solution (or equivalent) and rinse with deionized (Reagent Grade II) water.
6. Well depth should be obtained from well logs. Measuring total depth of wells prior to sampling should be avoided to limit suspension of settled solids.

5.2.3.1 Sampling Using a Passive Diffusion Bag Sampler

Groundwater samples will be collected for VOC analyses using PDB sampling from most monitoring wells. A typical PDB sampler consists of a low-density polyethylene tube closed at both ends and filled with deionized water. It is positioned in the well at the desired target depth by attaching it to weighted tether. The water within the bag is allowed to equilibrate with the ambient groundwater before retrieval. The sampler water is then decanted into 40 mL volatile organic analysis (VOA) vials and sent to the lab for analysis. Detailed procedures for using PDB samplers in wells can be found in "User's Guide for Polyethylene-Based Passive Diffusion Bag Samplers to Obtain Volatile Organic Compound Concentrations in Wells" (USGS, 2001). The following is a generalized summary of PDB sampling:

1. The top and bottom of the PDB sampler will be attached to 3/16" polyester or similar non-buoyant rope strong enough to support the weight of the sampler and subject to minimal stretch. The PDB will be suspended within the well screen at selected depths based on the depth to water, location of the screen and total depth of the well. Weights will be attached to the bottom of the sampler to keep it in place in the well. The sampler will be allowed to equilibrate for at least two weeks before being carefully retrieved with the attached line and the sample collected.
2. The PDBs will carefully be withdrawn from the well and inspected. Any evidence of algae or other coatings on the bag or tears in the membrane will be noted in the field book. If there are tears, the sample will be rejected.
3. The contents of the intact bag will then be transferred to pre-preserved VOA vials causing as little agitation of the sample as possible.
4. A new PDB will be filled with deionized water, and attached to the tether with zip ties, then carefully lowered into the well. The well cap, lock, and cover will be securely fastened once the PDB is in place.

5.2.3.2 Sampling Using a Bladder Pump

The sampling protocol will be as follows for the collection of groundwater samples using low-flow sampling with a portable bladder pump:

1. Wells should be sampled in order of increasing contamination (i.e. - samples that are expected to be least contaminated will be collected before those that are more highly contaminated).
2. The bladder pump will be decontaminated prior to use in each well.
3. Slowly and carefully lower the pump inlet to the mid-point of the screened interval. In cases where the entire screen is not saturated, place the pump inlet near the middle of the saturated screen, keeping in mind the limitations stated below.
4. Do not place pump inlet less than 2 feet above the bottom of the well, as this may cause the mobilization of bottom sediments. If saturated screen length is 2 feet or less, collect sample using disposable bailer.
5. Allow at least 1-foot of water above the inlet so there is little risk of entrainment or air in the sample.
6. Begin purging the well at a rate of 200 to 500 milliliters per minute (mL/min). All purge water will be containerized as investigation derived waste (IDW). The appropriate purge rate will be determined by monitoring groundwater drawdown.
7. The discharge during purging and sampling should flow with minimal turbulence or agitation.
8. The water level should stabilize and the pump rate should allow water to recharge the well so that little or no water level drawdown is observed. Adjust discharge rate to limit drawdown.
9. Record groundwater level frequently until water level stabilization occurs. After stabilization, measure water levels at regular intervals.
10. If drawdown is greater than 0.1 meter (4 inches), decrease the discharge rate of the pump and repeat discharge and water level measurements. Repeat until the water level stabilizes to closely match the recharge rate. Record pumping rates and depths to water on the Sample Collection Data sheet (Attachment 4-2).
11. If the water level does not stabilize, at least three well volumes will have to be removed and the water quality indicators will have to stabilize prior to sampling.
12. An in-line multi-probe flow-through cell will be used to monitor the indicator parameters so as not to expose the water to the atmosphere prior to measurement. During purging, water quality indicator parameters (pH, turbidity, specific conductivity, and DO) will be measured every 5-10 minutes until the parameters have stabilized. Measurement should be recorded on Attachment 4-2. A minimum of 5 sets of water quality indicator parameters should be recorded.
13. Stabilization is achieved after three successive readings are within ± 0.1 for pH, ± 5% for specific conductance, ± 10% for DO, and <10 NTU for turbidity. Temperature and ORP will also be measured and recorded, but will not be used as stabilization parameters. Sampling may begin once the well has stabilized.
14. Specific conductance and DO usually take the longest to stabilize. Up to 2 hours of purging may be required to reach stabilization. Stabilized purge indicator trends are generally obvious and follow either an exponential or asymptotic change to stable parameter values during purging.

15. The pump will not be turned off between the purging and sampling processes.
16. If stabilization does not occur or turbidity is >10 NTU after two hours of purging, the FTL should be contacted for direction.
17. Groundwater samples will be collected by gently filling the sample bottles with minimum turbulence once equilibrium is established. Lower the flow rate to 100 mL/min and fill sample containers as described in Section 5.2.3.4.

5.2.3.3 Sampling using a Disposable Bailer

Wells will be sampled with bailers where necessary due to small diameter casing in piezometers and slow recharge or thin saturated layer in wells. A new disposable bailer will be used for sampling at each well. Purging and sampling will be conducted in a manner that minimizes the agitation of sediments in the well and formation; the bailer will not be allowed to free fall into a well.

The sampling protocol will be as follows for the collection of groundwater samples using a disposable Teflon bailer:

1. Measure the static water level prior to purging using a decontaminated electronic water level indicator. The probe of the water level indicator will be lowered into the well bore and the water level will be recorded.
2. Attach the Teflon coated stainless steel leader rope to the bailer and polypropylene (or nylon) rope to the Teflon coated rope. Lower the bailer into the well, until it contacts the water surface. Allow the bailer to sink and fill with a minimum of water surface disturbance. Slowly withdraw the bailer from the well, preventing the bailer and bailing line from touching the ground.
3. The well should be purged until a minimum of three well volumes is removed from the well, and the water quality indicators of DO, pH, specific conductivity, and turbidity stabilize. Readings will be taken after each well volume is removed and recorded on the Sample Collection Data sheet (Attachment 4-2). Stabilization is achieved after three successive readings are within ± 0.1 for pH, ± 5% for specific conductance, ± 10% for DO, and <10 NTU for turbidity. Temperature and ORP will also be measured and recorded, but will not be used as stabilization parameters. Sampling may begin once the minimum well volume has been removed and water quality indicators have stabilized. If stabilization does not occur or turbidity cannot be reduced below 10 NTU after three well volumes have been removed, additional purging (up to five well volumes), should be performed. If the parameters have not stabilized within five volumes, the field team leader should be contacted for direction.
4. If the well is purged dry, a sample will be collected as soon as sufficient recharge has occurred and within 24 hours. Temperature, specific conductance, turbidity, pH, and DO will also be measured and recorded; however, stabilization of these parameters is not required.
5. After water quality indicators stabilize or the well recharges, collect samples by pouring the water from the bailer into the appropriate sample containers. This process will be repeated as necessary to fill each container.
6. After samples have been collected, replace the well cap and lock the security casing.

7. Place samples into the cooler with ice, record samples in the logbook, and enter sample times into the computer on the digital chain-of-custody (COC).
8. Record field conditions, problems encountered during sampling, and sample appearance in the field logbook and include the information in the Daily Field Report (SOP 1, Attachment 1-2).

5.2.3.4 Sample Collection

Groundwater samples will be collected by gently filling the sample bottles with minimum turbulence. Fill the sample bottles in the following order, as needed for the required analyses:

- Volatile organic compounds (VOCs) (no headspace)
- Carbon Dioxide, Methane, Ethane, Ethene (no headspace)
- Metabolic fatty acids (MFAs) (no headspace)
- Total organic carbon (TOC) (no headspace)

Collect the samples to be analyzed for volatile organics first, leaving zero headspace. Once the VOC sample is filled, carefully secure the cap to the vial. Turn the container upside down and look for any bubbles inside the vial. If bubbles are observed, gently remove the cap and carefully add a small amount of sample water to the container until a small meniscus forms at the rim of the vial. Gently place the cap over the meniscus and secure to the vial. Re-inspect the container for any air bubbles. If air bubbles are observed again, repeat the sample process using a new clean VOC container. Proceed with the collection of samples for the remaining analyses, collecting the more volatile parameters first.

5.3 Closeout

Perform following activities daily before leaving the site:

- Decontaminate and check condition of field equipment.
- Provide log books and other field documentation to FTL for review.
- Properly dispose of trash, debris and used PPE.
- Store purge water in the designated area.
- Make arrangements for shipment of samples (if applicable) and follow-up with the analytical laboratory to confirm samples arrived in good condition in accordance with SOPs 7 and 8.
- Complete the Daily Field Report (SOP 1, Attachment 1-2) and submit to PM.

Upon the completion of groundwater sampling activities, the FTL will perform closeout activities per SOP 1 and complete Closeout Report (SOP 1, Attachment 1-3) and submit to PM.

6 Data and Records Management

All field forms and log book entries will be scanned and copied project folder on the “Z” drive within one week of the field event completion. All photographs taken during the field event will be uploaded along with a typed photograph log (date, project and subject) to the “Z” drive.

7 Quality Control and Quality Assurance

All work will be performed in accordance with the QAPP, the specific work plan, and applicable SOPs.

8 References

MACTEC *RA SAP Volume I: Field Sampling Plan, Defense Depot Memphis, Tennessee, Revision 1*, *WTP-4 Groundwater Sampling*. November, 2005.

User’s Guide for Polyethylene-Based Passive Diffusion Bag Samplers to Obtain Volatile Organic Compound Concentrations in Wells (USGS, 2001).

SESDPROC-301-R3, *Operating Procedure: Groundwater Sampling,* 2013.

SESDPROC-105-R2, *Operating Procedure: Groundwater Level and Well Depth Measurement,* 2013.

Attachment 4-1

Water Level Measurement and Well Assessment Record

Sample Event: _____

Well I.D	Previous Measurement 3/30/2011 Depth to Water (ft, btoc)	Depth to Water (ft, btoc)	Date	Well Assessment
MW-03	63.56			
MW-04	71.00			
MW-05	75.49			
MW-06	58.96			
MW-07	63.60			
MW-08	59.68			
MW-10	-			
MW-13	69.39			
MW-14	72.17			
MW-15	64.90			
MW-28	54.45			
MW-31	65.50			
MW-32	-			
MW-33	52.04			
MW-37	-			
MW-42	52.89			
MW-43	116.05			
MW-44	50.81			
MW-45	54.51			
MW-51	39.22			
MW-54	75.83			

Attachment 4-2

Water Sample Collection Sheet

Site Name:_____ Project No.:_____
Sample No.:_____ Well ID.:_____
Date/Time Collected:_____ Personnel:_____
Sample Method:_____
Sample QC: Duplicate Yes No Duplicate Sample ID:_____
MS/MSD: Yes No
Well Purging Data (Fill In All Blanks)
Depth Of Sample Collection (ft, btoc) _____
Date:_____ Depth To Water (ft, btoc)_____
Time Completed:_____ Total Purge_____ Units_____

Field Measurements: Fill In All Blanks

Time (24 hour)	Amount purged (ml)	pH	COND (mS/m)	TURB (NTU)	DO (mg/L)	TEMP (C°)	ORP (mV)	Water Depth (ft, btoc)

Flow Rate_____

General Comments: _____

STANDARD OPERATING PROCEDURE 5 – VAPOR SAMPLE COLLECTION

Lead Organization: Department of the Army (DA)
Preparing Organization: HDR
SOP Approved by: Field Team Leader: Eric North
Project QA Officer: Lynn Lutz
Project Manager: Tom Holmes

1 Purpose and Summary

This Standard Operating Procedure (SOP) provides guidance for vapor sample collection at Defense Depot Memphis, Tennessee (DDMT). The project work plan must be reviewed for specific requirements.

2 Health and Safety

General Information on health and safety requirements are provided in SOP 1. Each individual is required to have read and understood the Site Safety and Health Plan for the specific project activity and signed the acknowledgement sheet confirming their review.

Health and safety concerns for vapor sampling include the use of lead-acid batteries, pressurized tubing, hot surfaces, and biological hazards. Batteries should be handled and transported properly to avoid acid spills. Some vapor samples locations are under positive pressure, and safety glasses should be worn at all times. Equipment in the machine rooms, including metal piping, can be very hot and care should be taken to not come in contact with the hot surfaces. Biological hazards include spiders, wasps, bees, and possibly snakes; care should be taken when reaching into areas that cannot be visually inspected.

3 Personnel Qualifications and Responsibilities

Vapor sampling will be directed by a Field Team Leader (FTL), an environmental professional (engineer, geologist or scientist) with appropriate experience. Field staff will be junior to mid-level environmental professionals or environmental technicians.

4 Equipment and Supplies

The required equipment and supplies will be identified in the project work plan for the specific field activities to be performed. Field activities should not proceed until the proper tools and equipment are available and in good working order. Usual equipment/supplies for a vapor sampling will include: a photoionization detector (PID), a vacuum pump, a set of tedlar bags, 6-liter Summa canisters and Summa canisters wrenches.

Each sampler will have use of a truck/van during field activities. An initial safety check should be performed at the start of each shift to confirm the vehicle is in good working condition. The vehicle should then be checked daily for damage or required maintenance.

5 Procedure

5.1 Start-Up Activities

5.1.1 Office

The Project Manager (PM) will assign a FTL to direct field activities and coordinate with project personnel. General responsibilities are described in SOP 1. Task specific responsibilities include:

- Coordinate sampling activities with the project chemist (PC) and analytical laboratory; prepare a sampling plan detail listing the sample locations and schedule shipment of laboratory-supplied Summa canisters and equipment for arrival prior to the start of sampling.
- Confirm availability and condition of DDMT-owned equipment and order additional equipment/supplies (tubing and Tedlar bags) for delivery prior to the start of sampling event.
- Prepare field forms and other documentation for the planned event.

5.1.2 Field

After arrival on site, but prior to commencement of operations, the following activities will be performed:

- Complete equipment and supply checklists and verify that required documentation and equipment for field activities are on site.
- Review condition of DDMT-owned and rental equipment; inventory field supplies and laboratory-provided sampling supplies. Sample tubing and Tedlar bags should be replaced every three months.
- Confirm the exact locations of the samples to be collected.
- Check that monitoring equipment is functioning properly, and calibrated as needed.
- Due to the limited field activities for vapor sampling, completion of the Field Event Startup Report (SOP 1, Attachment 1-1) is not required.

5.2 Field Operations

Prior to sampling, a field station will be established. The station will contain equipment, supplies, safety gear, and instrumentation necessary for the collection of samples. Environmental conditions will also be noted. Each sampling site will be characterized by the following factors:

- Location of work
- Weather conditions including precipitation, temperature and wind direction
- Ongoing activities that may influence or disrupt sampling efforts

- Accessibility to the sampling locations

All laboratory sampling will be documented in the field logbook. The logbook will summarize sampling events include sampling locations and times, field conditions and other significant information.

5.2.1 Sample Locations

Field screening and laboratory vapor samples are collected from individual soil vapor extraction (SVE) wells, the SVE system effluent (vapor stream from all SVE wells), and vapor monitoring points (VMPs), sub-slab vapor ports, and soil borings. Samples for field measurements will be collected using an oil-less vacuum pump and captured in Tedlar bags for photoionization detector (PID) and/or helium detector readings. Laboratory vapor samples will be collected via Summa canisters.

5.2.2 Sample Containers

Laboratory samples will be collected by field personnel in accordance with the project work plan and at the direction of the PM. Sample collection will follow United States Environmental Protection Agency (USEPA) TO-15 volatile organic compounds (VOCs) procedures. Laboratory samples from the SVE system effluent will be collected in 6-liter Summa canisters; a 200 milliliter/minute (mL/min) regulator for laboratory analysis will be used when collecting samples from VMPs. Laboratory samples from sub-slab sample ports and soil borings will be collected in 1-liter Summa canisters restricted with a 200 mL/min regulator. Standard turnaround time (TAT) for laboratory results is 15 workdays.

Summa canisters will be delivered from the analytical laboratory; a pressure gauge and a flow regulator (if needed) for each canister should be included. Arrangement for delivery will be coordinated by the PC.

5.2.3 SVE Wells and System Effluent

Field measurements and samples for laboratory analysis will be collected to monitor system performance and VOC concentrations in emissions.

5.2.3.1 Field Measurements

Field measurements will be collected from individual SVE wells and the system effluent. While online, the SVE system is continuously pulling vapor from the subsurface; thus, no purging of wells or the system is required prior to field (PID) sample collection. Ensure all wells to be sampled are online for a minimum of two hours prior to sample collection. Field measurement procedures are as follows:

- Connect sampling pump inlet hose to SVE well sample port located on SVE manifold.
- Open appropriate well sample port ball valve.
- Turn on sampling pump and allow it to run for five seconds to purge the pump and tubing.
- Connect tedlar bag to discharge of sampling pump by inserting nipple of bag into pump discharge tube.

- Allow tedlar bag to fill (approximately 20 seconds).
- Once filled, disconnect tedlar bag from sampling pump.
- Close SVE well sample port ball valve.
- Connect calibrated PID Meter to tedlar bag.
- Allow PID Meter to measure VOC concentration. Ensure reading on PID meter stabilizes before recording VOC concentration. This usually takes 10 to 15 seconds.
- Record peak VOC concentration and time.

5.2.3.2 Laboratory Samples

Procedures for sample collection from the SVE wells and system effluent are as follows:

- Fill out Summa canister tag with sampling information using a pen with blue or black waterproof ink.
- Remove the Summa canister valve cap.
- Run dedicated tubing from SVE manifold to canister by connecting swagelock.
- Open appropriate SVE well/effluent sample port ball valve.
- Record starting Summa canister pressure on chain-of-custody (COC). The starting Summa canister pressure should be at least -25 inches of mercury (in. Hg) or greater. If not, the canister has leaked and should not be used for sampling.
- Open Summa canister valve located at top of sampling canister.
- Record sampling start time on COC.
- Allow Summa canister to fill until pressure gauge reads -5 in. Hg. (approximately 2 minutes with a 6-liter canister).
- Close sampling port ball valve at SVE well.
- Disconnect Summa canister from regulator.
- Record time of sample collection, date, and Summa canister serial and regulator numbers on COC form.

5.2.4 Vapor Monitoring Points

Field measurements and samples for laboratory analysis will be collected from VMPs to evaluate system performance and determine SVE well vacuum influence. It is necessary to purge VMPs prior to sample collection. Procedure will be repeated for the ‘A’ and ‘B’ screens at each VMP.

<u>Purging:</u>

- Unlock VMP well casing (secured by padlock).
- Attach regulator to “quick connect” on well cap, run line to a “T” connection.
- Run one line out from the “T” to the pump.

- Attach second line to the Summa canister via swagelock.
- Turn on sampling pump and allow lines to purge for approximately five minutes. Purge time is based on tubing diameter and length and is intended to remove three tubing volumes.

Field (PID) Measurements:

- Attach tedlar bag to discharge of sampling pump by inserting nipple of bag into pump discharge tube.
- Allow tedlar bag to fill (approximately 20 seconds).
- Once filled, disconnect tedlar bag from sampling pump.
- Connect calibrated PID Meter to tedlar bag.
- Allow PID Meter to measure VOC concentration. Ensure reading on PID meter stabilizes before recording VOC concentration. This usually takes 10 to 15 seconds.
- Record peak VOC concentration and time.
- Open valve on tedlar bag to completely deflate bag.
- Collect additional PID readings following the previous steps until three consecutive readings are within 10% of each other.

5.2.4.1 Laboratory Samples

Procedures for sample collection from the Dunn Field VMPs are as follows:

- Attach the vacuum pump and its dedicated tubing to the VMP well quick connect fitting.
- Close the valve to the “T” fitting and open the valve to the pump and start the pump.
- Each VMP has a purge time determined and it is on the VMP sample collection sheet for each VMP; run the vacuum pump for the allotted time.
- Attach a tedlar bag to the output from the vacuum pump and allow it to fill.
- Read the tedlar bag with a PID and record the result; repeat this procedure until three readings are within 10%.
- Fill out the Summa canister tag with sampling information using a pen with blue or black waterproof ink.
- Remove the Summa canister valve cap.
- Attach regulator to 6-liter Summa canister. An individual regulator should be provided by the laboratory for each Summa canister to be used for VMPs.
- Connect the Summa canister to the “T” tubing that was used to purge the VMP tubing. The Summa canister is connected before the vacuum pump, because the Summa canister is under its own vacuum.
- Open appropriate VMP sample port ball valve.

- Record starting Summa canister pressure on COC. The starting Summa canister pressure should be at least -25 in. Hg or greater. If not, the canister has leaked and should not be used for sampling.
- Open Summa canister valve located at top of sampling canister.
- Record sampling start time on COC.
- Allow Summa canister to fill until pressure gauge on regulator reads -5 in. Hg (approximately 30 minutes with a 6-liter canister and a 200 mL/min flow regulator).
- Close sampling port ball valve at VMP cap.
- Disconnect Summa canister from regulator.
- Record time of sample collection, date, and Summa canister serial and regulator numbers on COC form.

5.3 Sub-Slab and Soil Boring Vapor Sampling

Sub-slab sample ports and temporary soil borings will be installed to evaluate VOC concentrations beneath or adjacent to impervious surfaces. VaporPin, or equivalent device, will be constructed in accordance with manufacture specifications to allow for collection of sub-slab samples. Shallow soil gas samples will be collected from soil borings via the post run tubing (PRT) method. Procedures for these sampling methods are presented below.

Leak Check

Leak check should be performed prior to sample train purging and sampling to verify the integrity of the sub-slab sample port (Water Dam Procedures) and PRT annular seal (Tracer Test) and to identify the presence of leaks in the sample train (Shut-In Test).

5.3.1 Water Dam Procedure

Leak testing of the Vapor Pin annular seal will be performed with a water dam in accordance with the method prescribed in the Vapor Intrusion Technical Guidance (NJDEP, 2016). The water dam will be constructed from PVC and surround the sub-slab sample port. The water dam will be sealed to the concrete floor with modeling clay or other VOC free inert material. The water dam will be filled with distilled/deionized water so that it is covering the sub-slab sample port annular seal. The water level will be briefly observed to verify that the water level is not receding. If the water level remains stable the sample train will be purged and a sample will be collected while continuing to observe the water level. Should the water level decrease, the sampling will be stopped, a new sub-slab sample port will be installed and the process repeated.

5.3.2 Tracer Test

A tracer test is used to determine whether ambient air is introduced into the soil gas sample during the collection process. The tracer test and well purging will be performed simultaneously by the steps described below:

1. Connect aboveground sample train to PRT tubing and place a shroud over the drive rod and sample train;

2. Inject He gas under the shroud to achieve a target shroud concentration of two orders of magnitude greater than the He meter minimum detection limit;
3. Purge three casing volumes from the sample train with a syringe or pump, at a flow rate of 100 to 200 ml/min and contain the purged gas in a Tedlar bag;
4. Use the He detector to measure the He concentration in the Tedlar bag and beneath the shroud;
5. If the He concentration in the Tedlar bag is less than 5 percent (%) of the concentration beneath the shroud then a sample can be collected; and
6. If the He concentration in the Tedlar bag is greater than 5% of the concentration beneath the shroud than the annular seal may be compromised. A sample can still be collected, but the technician will note that the He test failed and the result will be flagged.

5.3.3 Shut-In Test

A shut-in test will be performed prior to purging and sampling soil gas and sub-slab aboveground sampling trains to locate leaks. This test is performed by assembling the sample train, as if a sample was being collected, and a vacuum of at least 100 inches of water (7.4 inches of mercury) is applied. At this point, the sample train should be isolated from the sub-slab sample port either by a valve or disconnected and plugged so that the vacuum can be applied without evacuating soil gas. After the vacuum is applied, the field technician will observe the vacuum gauge on the sample train for any change in vacuum. If the vacuum in the sample train dissipates then the leak will be located, corrected, and the test will be repeated until the sample train can hold a vacuum for at least thirty seconds.

Sampling

After the field technician has completed the shut-in test, tracer test, and purged three casing volumes, the soil gas sample will be collected. This will be performed by opening the valve on the top of the 1-liter Summa canister and allowing the canister vacuum to remove one liter of soil gas at a 200 ml/min flow rate. The Summa canister will be labeled with the sample identification, starting vacuum, ending vacuum, sampler's initials, sample date, and sample time.

Field records will be maintained that detail site activities and observations so that an accurate, factual account of field procedures may be reconstructed. At a minimum, the field records will contain sample identification, collection time, location description, methods used, daily weather conditions, field measurements, name of sampler(s), names of contractor/subcontractor personnel, and other site-specific observations including any deviations from the project work plan. HDR will periodically record the precipitation, temperature, and barometric pressure from a nearby weather station during and 72 hours prior to sample collection.

5.4 Closeout

5.4.1 Field

Following sample collection, the following procedures will be performed by on-site personnel:

- Decontaminate all field equipment.

- Ensure all field documentation is completely filled out. This includes the COC and Summa sampling tag. Unless revised by the project manager, standard turn-around time (15 days) will be used. Retain copy of COC for the project file.
- Package Summa canisters in sturdy cardboard boxes with packing material to prevent any potential puncture of the canister. In most cases, the boxes and packing material used by the laboratory to ship the Summa canisters to the site can be reused.
- Affix a custody seal across the top taped seam of the canister shipping carton and elsewhere as necessary to ensure security.
- Ship Summa canisters to laboratory for analysis. Ensure copy of COC is included in shipment.
- Complete logbook, making notations as to site conditions, anomalous readings, etc.
- Ensure that equipment and associated supplies have been shipped back to the office or supplier.
- Ensure that all IDW/trash has been disposed in accordance with the project work plan and QAPP.

5.4.2 Office

Upon return to the office, field personnel will perform the following:

- Submit logbook and any original forms to Project/Task Manager for review
- Completion of the Field Event Closeout Report (Attachment 1-3) is not required.

6 Data and Records Management

All field forms and log book entries will be scanned and copied to the project folder on the “Z” drive within one week of the field event completion. All photographs taken during the field event will be uploaded along with a typed photograph log (date, project and subject) to the “Z” drive. All original forms will be stored on site in Memphis in the filing cabinet in the proper folder labeled for the project. The PM and PC will be sent a link for the data.

7 Quality Control and Quality Assurance

All work will be performed in accordance with the Quality Assurance Project Plan (HDR, 2017a), the project work plan, and applicable SOPs. All field activities will be recorded in the log books in sufficient detail to reconstruct the events. No erasures or mark outs will be made on field forms or log books. A single line will be used to strike out errors and will be annotated with the initials and date of the editor.

8 References

HDR, 2017a. *2017 Uniform Federal Policy-Quality Assurance Project Plan, Environmental Restoration Support at Former Defense Depot Memphis, Tennessee, Revision 0*. Prepared for the U.S. Army Corps of Engineers, Mobile District. May 2017.

New Jersey Department of Environmental Protection (NJDEP), 2016. *Vapor Intrusion Technical Guidance,* August 2016.

STANDARD OPERATING PROCEDURE 7 – SAMPLE CONTROL AND DOCUMENTATION

Lead Organization: Department of the Army (DA)
Preparing Organization: HDR
SOP Approved by: Field Team Leader: Eric North
Project QA Officer: Lynn Lutz
Project Manager: Tom Holmes

1 Purpose and Summary

This Standard Operating Procedure (SOP) provides guidance for sample control and identification, data recording, and proper completion of Chain-of-Custody (COC) forms.

2 Health and Safety

General Information on health and safety requirements are provided in SOP 1. Each individual is required to have read and understood the Site Safety and Health Plan for the specific project activity and signed the acknowledgement sheet confirming their review.

Health and safety concerns for sample handling include potential for exposure to contaminants, sample container preservatives, and injury from breakage of sample containers. Contamination levels at Defense Depot Memphis, Tennessee (DDMT) are relatively low but care should be taken to avoid exposure. Sample containers should be handled carefully; nitrile gloves and safety glasses should be used.

3 Personnel Qualifications and Responsibilities

Sample control activities will be directed by the Field Team Leader (FTL), an environmental professional (engineer, geologist or scientist) with experience in sampling activities. The field staff, environmental professionals or technicians, are responsible for proper sample handling and documentation of the sample collection.

4 Equipment and Supplies

The field staff will use a pen with blue or black waterproof ink to record field activities and document sample handling in a field logbook and on field data sheets. A laptop computer with laboratory-provided software may also be used for sample documentation.

5 Procedure

Proper field sampling and documentation help ensure sample authenticity and data integrity. These procedures describe sample collection documentation and sample handling, tracking, and custody procedures to ensure that sample integrity and custody are maintained.

If the computer is being used to scan the samples as they are collected the data recorded by the computer should be checked for correctness. The date and time on the computer should be checked prior to scanning of any samples. The sample label should be completed when the sample is collected. If a hand written COC will be used, all information should be recorded in a log book as to the type of sample, date and time collected and number of sample containers. The COC can then be filled out back at the field office in a quiet environment with out disturbances to avoid errors.

Corrections to the COC, field logbook or field data forms will be made by a single line to strike out errors annotated with the initials and date of the editor; the correct information will be inserted as appropriate.

The number of sample containers on the COC should be physically checked against the number of containers collected. Once this is confirmed the sample crew can properly store the samples for shipment.

5.1 Start-Up Activities

5.1.1 Office

The FTL will work with the project chemist (PC) to:

- Prepare the sampling plan detail (Attachment 7-1).
- Coordinate with the analytical laboratory and ensure that sample forms including chain of custody forms and custody seals are shipped to the site.

5.1.2 Field

After arrival on site, but prior to commencement of operations, the FTL will confirm that required documentation and equipment for field activities are on site.

5.2 Field Operations

5.2.1 Sample Identification

Individual samples will be identified by a unique alphanumeric code (also referred to as a sample ID number or field number) which will be written on the sample label and recorded on the COC form. The sample ID will include the location and sampling event as described in Worksheet #17of the Quality Assurance Project Plan (QAPP). Additional information to be written on the label includes sample ID, time and date of sample, sampler’s initials, and the analytical methods to be performed, as described in Section 5.2.3 of this SOP.

Field Quality Control (QC) samples to be collected at DDMT include trip blanks, rinsate blanks, field (ambient) blanks, and field duplicates. The ID for trip blanks, rinsate blanks and field blanks will consist of the prefix TB, RB or FB, respectively, followed by a number, followed by the sampling event, as shown below:

TB-1-ODPM-9	first Trip Blank for event ODPM-9
TB-2-ODPM-9	second Trip Blank for event ODPM-9
RB-1-ODPM-9	Rinsate Blank for event ODPM-9
FB-1-ODPM-9	Field Blank for event ODPM-9

Matrix spike and matrix spike duplicate samples will also be collected. The ID for these samples will consist of the location ID, followed by the sampling event, followed by the suffix matrix spike (MS) or matrix spike duplicate (MSD), as shown below:

MW-164-ODPM-9-MS	Matrix Spike sample for well MW-164
MW-164-ODPM-9-MSD	Matrix Spike Duplicate sample for well MW-164

The identity of field duplicate samples will be concealed from the laboratory by using a consecutively numbered duplicate identifier, followed by the sampling event, as shown below:

DUP-1-ODPM-9	first field duplicate for event ODPM-9
DUP-2-ODPM-9	second field duplicate for event ODPM-9

The location of field duplicates will be recorded on the sampling plan detail (SPD) and field notebook. The final SPDs will be maintained in the project file and copies will be kept at the on-site field office. At the end of the sampling event, the FTL will send the PM and PC the final SPD with changes to field duplicate or MS/MSD sample IDs, additional blanks collected, and any other changes.

5.2.2 Field Documentation

5.2.2.1 Logbook

The logbook is a written record of sampling activities to be completed in the field during sampling. The purpose is to document field conditions or procedural exceptions that may aid in the analysis of data generated from sampling activities. The log book will have with sequentially numbered pages and information will be recorded in blue or black waterproof ink. The recorder will sign and date each entry.

Information pertaining to environmental conditions at the site during the field investigation will be noted in the field log book for each day. The following information will be recorded for each activity:

1. Activity
2. Location
3. Date and time
4. Weather conditions

For field sampling activities, the following information will be recorded, if a sampling form is not used:

1. Sample type and sampling method

2. The identity of each sample and the depth(s) from which it was collected
3. Sample description (e.g., color, odor, clarity)
4. Identification of sampling devices used
5. Identification of sampling conditions that might affect the representativeness of a sample (i.e., refueling operations, damaged casings)

5.2.2.2 Daily Field Reports

Each day the FTL will prepare a Daily Field Report (SOP 1, Attachment 1-2). The report will include daily weather, time and description of field activities, samples collected, and any problems or changes in scope that occurred that day. The report also lists field staff, subcontractors and site visitors.

5.2.2.3 Photographs

Photographs taken for the purpose of project documentation will be noted in the field logbook. The sequential number of the photograph, photographer, date, time, location, description, and orientation of the photograph will be recorded in the logbook as the photographs are taken. The photographs and documentation will be loaded on the HDR network project file.

5.2.3 Sample Labels/Tags

Sample labels will be filled out for each sample with an indelible pen. The label will be protected from water and solvents with clear label protection tape. Any change in the pre-prepared label information will be initialed by the sampler.

5.2.3.1 Labels for Groundwater Samples

Pre-printed labels from the laboratory for groundwater sampling events contain the following information:

- Sample ID
- Preservative
- Date the bottle was prepared
- Matrix
- Tests
- Laboratory name
- Bar code

The sample collector will write in the following information:

- Date of collection
- Time of collection
- Name or initials of collector

5.2.3.2 Sample Tags for Air Samples

Sample tags from the laboratory for air sampling events contain the following information:

- Laboratory name, address, phone number and fax number

The sample collector will write in the following information:

- Client name (HDR)
- Sample ID
- Analysis (TO-15)
- Date and time of sample collection
- Sampler's initials
- Comments

5.2.4 Sample Custody

Sample custody is a part of a quality field or laboratory operation. Custody of a sample is defined as:

1. Having physical possession
2. Being in view, after being in possession
3. Having possession, then being placed in a secure area
4. Being maintained in a secure area by the person who had possession last

These custody practices will be observed in the field. They will be performed according to the procedures described in the following subsections.

5.2.4.1 COC Records

A hand-written three-part COC will be fully completed, in triplicate. The first two pages will accompany the cooler to the laboratory, and the bottom copy will be retained in the files at the field office after it is scanned into the computer file.

A computer-generated COC will have one copy printed that will accompany the cooler to the laboratory. The data used to generate the COC will be transmitted via E-mail to the laboratory and a PDF copy of the COC will be saved on the computer in the sampling file.

The information specified on the COC record will contain the same level of detail found in the site log book, with the exception that on-site measurement data will not be recorded. The custody record will include at least the following information:

- Name of person collecting the samples
- Date samples were collected
- Type of sampling conducted (composite/grab)
- Location of sampling station (including the site location)
- Number and type of containers used

- Signature of the HDR person relinquishing samples to a non-HDR person (such as a FedEx agent), with the date and time of transfer noted, and the cooler designation
- Airbill Number

If samples will require rapid turnaround in the laboratory because of project time constraints or analytical concerns such as extraction time or sample retention period limitations, these constraints will be noted in the remarks section of the custody record. The FTL or designee will contact the laboratory to confirm the turnaround time can be achieved. The computer generated COC is for use with Microbac Laboratories only. Other laboratories will provide COCs for use.

It is not practicable to seal the sample coolers or cartons at a FedEx office; they will be sealed beforehand. The custody record will, therefore, have the signature of the relinquishing field technician with the date and time, but the “relinquished to” box will not be completed.

The duplicate custody record will then be placed in a plastic bag, taped to the underside of the cooler lid, and the cooler closed. COCs for air samples will be included in the carton. The container will be tightly bound with filament tape. Finally, custody seals will be signed by the individual relinquishing custody and affixed in such a way that the cooler or carton cannot be opened without breaking the seals.

The original and duplicate custody records and the airway bill or delivery note together constitute a complete record. The FTL will email a copy of the airbill and the COC to the PC, who will maintain the custody records as part of the analytical data file.

Custody Seals: Custody seals will be preprinted, adhesive-backed seals designed to break if disturbed. For groundwater samples, affix custody seals on the sample shipping containers (coolers) in as many places as necessary to ensure security. For vapor samples affix a custody seal across the top, taped seam of the canister shipping carton and additional locations as necessary. Seals will be signed and dated before application.

Laboratory custody procedures are described in the laboratory sample handling and storage SOPs L8 and L104, included in Appendix C of the QAPP.

5.3 Closeout

Before leaving the site daily, the following procedures will be performed by on-site personnel:

- Maintain custody of samples, maintaining them as specified for the analyses to be performed.
- Prepare samples for shipment to the laboratory.
- Complete the COC forms.
- Contact the laboratory to inform them that samples will be shipped and also remind them of any special requirements for the sample analyses.
- Verify completion of logbook, ensuring that required information has been recorded.

Upon the completion of sample collection and shipment, copies of the COCs will be scanned and sent to interested parties to include the PM and PC. The FedEx tracking numbers will be checked

each day to confirm the samples were delivered and the laboratory will be contacted to check on problems with the samples or COCs.

6 Data and Records Management

All field forms, COCs, and log book entries will be scanned and copied project folder on the HDR network project file within one week of the field event completion. All original forms will be stored on site in Memphis in the filing cabinet in the proper folder labeled for the project. The PM and PC will be sent a link for the data.

7 Quality Control and Quality Assurance

Work will be performed in accordance with the QAPP, the specific work plan, and applicable SOPs. Field activities will be recorded in the log books in sufficient detail to reconstruct the events and forms provided with the SOP will be completed. No erasures or mark outs will be made on field forms or log books. A single line will be used to strike out errors and will be annotated with the initials and date of the editor; the correct information will be inserted as appropriate.

8 References

HDR, 2017a. *2017 Uniform Federal Policy-Quality Assurance Project Plan, Environmental Restoration Support at Former Defense Depot Memphis, Tennessee, Revision 0*. Prepared for the U.S. Army Corps of Engineers, Mobile District. May 2017.

SESDPROC-209-R2, *Operating Procedure: Packing, Marking, Labeling and Shipping of Environmental and Waste Samples,* 2011.

Attachment 7-1

EXAMPLE: Sample Plan Detail

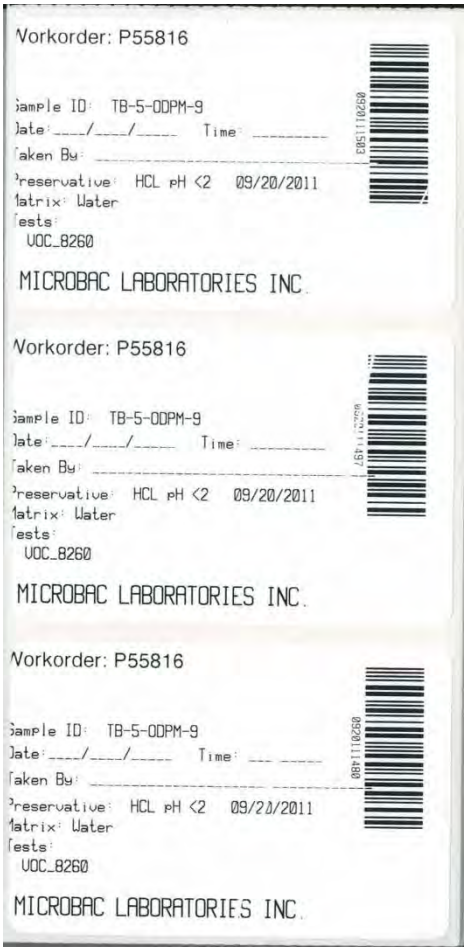
SAMPLING PLAN DETAIL (OFF DEPOT PM WELLS September 2011) - ODPM-9

Parameter	**VOCs**
Method	**8260B**
Container	**40 mL VOA**
Preservative	**HCl to pH<2**
	Cool to 4°C

#	Well ID	Sample ID	Additional	No. of Containers
1	MW-54	MW-54-ODPM-9		3
2	MW-70	MW-70-ODPM-9		3
3	MW-76	MW-76-ODPM-9		3
4	MW-77	MW-77-ODPM-9		3
5	MW-79	MW-79-ODPM-9	DUP-1	3
6	MW-148	MW-148-ODPM-9		3
7	MW-149	MW-149-ODPM-9		3
8	MW-150	MW-150-ODPM-9		3
9	MW-151	MW-151-ODPM-9		3
10	MW-152	MW-152-ODPM-9		3
11	MW-155	MW-155-ODPM-9		3
12	MW-157	MW-157-ODPM-9		3
13	MW-158	MW-158-ODPM-9		3
14	MW-158A	MW-158A-ODPM-9		3
15	MW-159	MW-159-ODPM-9	DUP-2	3
16	MW-160	MW-160-ODPM-9		3
17	MW-161	MW-161-ODPM-9		3
18	MW-162	MW-162-ODPM-9		3
19	MW-163	MW-163-ODPM-9		3
20	MW-164	MW-164-ODPM-9		3
20	MW-164	MW-164-ODPM-9-MS	MS	3
20	MW-164	MW-164-ODPM-9-MSD	MSD	3
21	MW-165	MW-165-ODPM-9		3
22	MW-165A	MW-165A-ODPM-9		3
23	MW-166	MW-166-ODPM-9		3
24	MW-166A	MW-166A-ODPM-9		3
25	MW-241	MW-241-ODPM-9		3
26	MW-242	MW-242-ODPM-9		3
27	MW-243	MW-243-ODPM-9		3
28	MW-244	MW-244-ODPM-9		3
29	MW-245	MW-245-ODPM-9		3
30	MW-246	MW-246-ODPM-9		3
31	RB	RB-ODPM-9		3
32	DUP-1	DUP-1-ODPM-9		3
33	DUP-2	DUP-2-ODPM-9		3
34	TB-1	TB-1-ODPM-9		3
35	TB-2	TB-2-ODPM-9		3

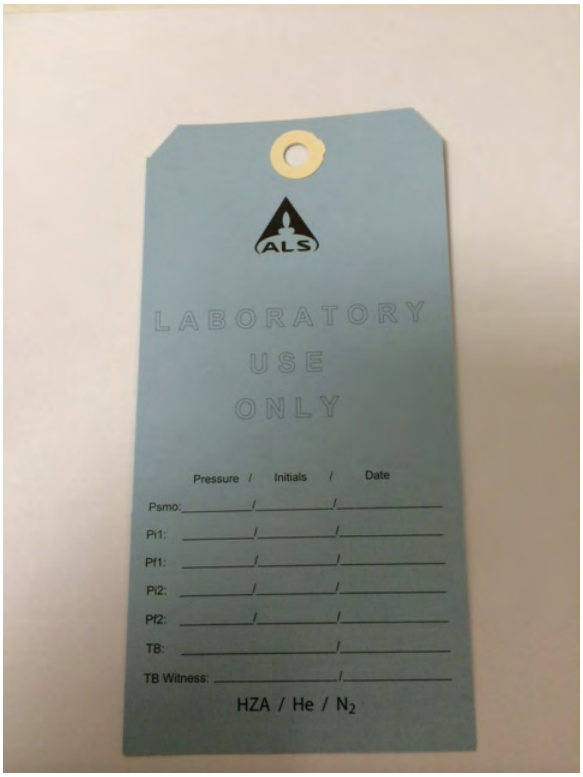
Attachment 7-2

EXAMPLE: Sample Labels for Groundwater Samples



Attachment 7-3

EXAMPLE: Sample Labels for Air Samples



Attachment 7-4

EXAMPLE: Microbac Chain-of-Custody Form (Computer)

Microbac

Chain of Custody
Chain #: 1001
Printed at : 04/26/2011 08:46

Barcode	Client ID	Tests	Collect Date	Beg. Depth	End. Depth	Notes
~~0420111~~	~~MW-91-ODLB-3~~	~~VOC_8260~~	~~04/25/2011 10:09~~			
~~0420112~~	~~MW-91-ODLB-3~~	~~VOC_8260~~	~~04/25/2011 10:09~~			
~~0420113~~	~~MW-91-ODLB-3~~	~~VOC_8260~~	~~04/25/2011 10:09~~			
0420111	04/25/11-TB-1-ODPM-8	VOC_8260	04/25/2011 10:09			
0420112	04/25/11-TB-1-ODPM-8	VOC_8260	04/25/2011 10:09			
0420113	04/25/11-TB-1-ODPM-8	VOC_8260	04/25/2011 10:09			
0420114	DUP-1-ODPM-8	VOC_8260	04/25/2011 11:32			
0420115	DUP-1-ODPM-8	VOC_8260	04/25/2011 11:32			
0420116	DUP-1-ODPM-8	VOC_8260	04/25/2011 11:32			
0420117	MW-250-ODPM-8	VOC_8260	04/25/2011 10:30			
0420118	MW-250-ODPM-8	VOC_8260	04/25/2011 10:30			
0420119	MW-250-ODPM-8	VOC_8260	04/25/2011 10:30			
04201110	MW-251-ODPM-8	VOC_8260	04/25/2011 10:42			
04201111	MW-251-ODPM-8	VOC_8260	04/25/2011 10:42			
04201112	MW-251-ODPM-8	VOC_8260	04/25/2011 10:42			
04201113	MW-54-ODPM-8	VOC_8260	04/25/2011 11:32			
04201114	MW-54-ODPM-8	VOC_8260	04/25/2011 11:32			
04201115	MW-54-ODPM-8	VOC_8260	04/25/2011 11:32			
04201116	MW-70-ODPM-8	VOC_8260	04/25/2011 13:23			
04201117	MW-70-ODPM-8	VOC_8260	04/25/2011 13:23			
04201118	MW-70-ODPM-8-MS	VOC_8260	04/25/2011 13:23			
04201120	MW-70-ODPM-8-MS	VOC_8260	04/25/2011 13:23			
04201122	MW-70-ODPM-8-MSD	VOC_8260	04/25/2011 13:23			
~~04201123~~	~~MW-70-ODPM-8-MSD~~	~~VOC_8260~~	~~04/25/2011 13:23~~			
04201125	MW-76-ODPM-8	VOC_8260	04/25/2011 13:07			
04201126	MW-76-ODPM-8	VOC_8260	04/25/2011 13:07			
04201127	MW-76-ODPM-8	VOC_8260	04/25/2011 13:07			
04201128	MW-77-ODPM-8	VOC_8260	04/25/2011 13:14			
04201129	MW-77-ODPM-8	VOC_8260	04/25/2011 13:14			
04201130	MW-77-ODPM-8	VOC_8260	04/25/2011 13:14			
04201131	MW-79-ODPM-8	VOC_8260	04/25/2011 11:17			
04201132	MW-79-ODPM-8	VOC_8260	04/25/2011 11:17			

Microbac OVD 221003014398
Received: 04/27/2011 12:56
By: BRENDA GREENWALT
(signed)

Barcode	Client ID	Tests	Collect Date	Beg. Depth	End. Depth	Notes
04201133	MW-79-ODPM-8	VOC_8260	04/25/2011 11:17			

Samples Collected on: 04/25/2011 by jbsperry
(signed)

Attachment 7-4

<u>EXAMPLE:</u> Microbac Chain-of-Custody Form (Hand)

COC No. A 23953

158 Starlite Drive
Marietta, OH 45750

Microbac
CHAIN-OF-CUSTODY RECORD

Phone: 740-373-4071
Fax: 740-373-4835

Company Name:	
Project Contact:	Contact Phone #:
Turn Around Requirements:	Location:
Project ID:	
Sampler (print):	Signature:

Program
- ☐ CWA
- ☐ RCRA
- ☐ DOD
- ☐ AFCEE
- ☐ Other ____________

Sample I.D. No.	Comp	Grab	Date	Time	Matrix*	NUMBER OF CONTAINERS	Hold															TOTAL # (LAB USE)	ADDITIONAL REQUIREMENTS

Relinquished by: (Signature)	Date	Time	Received by: (Signature)	Relinquished by: (Signature)	Date	Time	Received by: (Signature)
Relinquished by: (Signature)	Date	Time	Received for Laboratory by: (Signature)	Date	Time	Remarks:	

*Water (W), Soil (S), Solid Waste (SD), Unknown (X)

Page ________ of ________

Attachment 7-5

EXAMPLE: ALS Chain-of-Custody Form

Air - Chain of Custody Record & Analytical Service Request

Page _____ of _____

2655 Park Center Drive, Suite A
Simi Valley, California 93065
Phone (805) 526-7161
Fax (805) 526-7270

Requested Turnaround Time in Business Days (Surcharges) please circle
1 Day (100%) 2 Day (75%) 3 Day (50%) 4 Day (35%) 5 Day (25%) 10 Day-Standard

ALS Project No.

Company Name & Address (Reporting Information)	Project Name	ALS Contact:	Analysis Method		Comments e.g. Actual Preservative or specific instructions
	Project Number				
Project Manager	P.O. # / Billing Information				
Phone / Fax					
Email Address for Result Reporting	Sampler (Print & Sign)				

Client Sample ID	Laboratory ID Number	Date Collected	Time Collected	Canister ID (Bar code # - AC, SC, etc.)	Flow Controller ID (Bar code # - FC #)	Canister Start Pressure "Hg	Canister End Pressure "Hg/psig	Sample Volume			

Report Tier Levels - please select

Tier I - Results (Default if not specified) ____ Tier III (Results + QC & Calibration Summaries) ____
Tier II (Results + QC Summaries) ____ Tier IV (Data Validation Package) 10% Surcharge ____

EDD required Yes / No
Type: _____ Units: _____

Chain of Custody Seal: (Circle)
INTACT BROKEN ABSENT

Project Requirements (MRLs, QAPP)

Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:
Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:

Cooler / Blank Temperature ____ °C

STANDARD OPERATING PROCEDURE 8 – SAMPLE PACKING AND SHIPPING

Lead Organization: Department of the Army (DA)
Preparing Organization: HDR
SOP Approved by: Field Team Leader: Eric North
Project QA Officer: Lynn Lutz
Project Manager: Tom Holmes

1 Purpose and Summary

The purpose of this Standard Operating Procedure (SOP) is to provide guidance for packing and shipping environmental samples to the laboratory for analysis. The goals for sample packing and shipping are that: 1) the integrity of the sample is maintained, and 2) no exposure to the sample contents occurs during transit. These goals should be met regardless of the method by which the samples were shipped.

Samples will usually be shipped as either environmental samples or as hazardous materials based on the expected contaminant concentrations. While the concentration of constituents in the sample is not generally known prior to shipment of the sample, inferences can be made based on the site location and knowledge of past activities, observations during collection, and past sample results. Hazardous materials are generally considered to be samples of highly contaminated media collected at or near an observed release and can consist of pure product or a mixture. Environmental samples are generally media with low-level contamination.

Relevant regulations include Department of Transportation (DOT) regulations for ground transportation (49 Code of Federal Regulations [CFR]) and the International Air Transport Association (IATA) regulations for air transportation. Common carriers (e.g., FedEx, and UPS etc.) must abide by these regulations. This SOP provides specific guidance on how to package and ship samples to achieve the stated objectives and remain in compliance with shipping regulations. If field personnel are unsure regarding current shipping regulations, they will immediately contact the selected carrier (e.g., FedEx, UPS, etc.) for guidance.

2 Health and Safety

General Information on Health and Safety requirements are provided in SOP 1. Each individual is required to have read and understood the Health and Safety Plan (HASP) for the specific project activity and signed the acknowledgement sheet confirming their review.

Health and safety concerns for sample shipment include potential for exposure to contaminants, sample container preservatives, and injury from breakage of sample containers. Contamination levels at Defense Depot Memphis, Tennessee (DDMT) are relatively low but care should be taken to avoid exposure. Sample containers should be handled carefully; nitrile gloves and safety glasses should be used.

3 Personnel Qualifications and Responsibilities

Sample packing and shipping activities will be directed by the Field Team Leader (FTL), a mid- or senior level environmental professional (engineer, geologist or scientist) with experience in sampling activities. Field staff, environmental professionals or technicians, are responsible for proper sample handling and compliance with these guidelines.

4 Equipment and Supplies

The required equipment and supplies will consist of ice chests from the laboratory, clear tape, filament tape, gallon size Ziploc bags, trash bags, custody seals, bubble bags, cushion for bottom of cooler, and FedEx handle label hangers.

5 Procedure

5.1 Start-Up Activities

5.1.1 Office

The FTL will work with the project chemist (PC) to:

- Ensure that sufficient sample containers, shipping containers/coolers and packing material are shipped to the site based on the analytical parameters, total number of samples and average number of samples to be collected per day.
- Develop guidelines on the number/type of samples per shipper based on sample type and past analytical results (i.e. volatile organic compounds [VOCs] in one cooler to limit the number of trip blanks needed and samples from high concentration wells packed in separate cooler to prevent cross contamination)
- Coordinate sample shipments to ensure laboratory personnel will be available to receive the samples if weekend or holiday shipments are planned.

5.1.2 Field

After arrival on site, but prior to commencement of operations, the FTL will confirm that the required sample containers, sample coolers, packing material and ice are available on-site.

5.2 Field Operations

On specific projects, protocols for sample shipment will be specified in the work plan. This SOP provides general guidelines for sample packing and shipping.

- Samples will be shipped to the laboratory by an overnight courier service.
- Samples will not remain on site for more than 24 hours after collection, unless samples were collected on a weekend or there were not enough samples to make a shipment. These

samples will be stored in the refrigerator at 4 degrees Celsius (°C) in a locked office until the next shipment.

- Glass sample containers will be placed inside sealed plastic bubble wrap bags or wrapped in bubble wrap and placed in plastic bags as a precaution against cross-contamination due to leakage or breakage.
- Sample bottles will be placed in coolers in a manner to limit the breakage and/or leakage during shipment. All coolers will have a bottom cushion/absorbent placed in prior to placing the samples in the cooler.
- Coolers will be lined with a heavy duty plastic garbage bag.
- Segregate highly contaminated samples, if known, by placement in a separate cooler or in separate plastic zip-lock bags.
- All coolers will have the drain plug taped closed, if present.
- Sufficient ice in plastic bags (double-bagged) will be placed in the coolers to keep the samples at 4°C throughout shipment.
- The top of the garbage bag, lining the cooler and containing the samples and ice, will be tied or adequately sealed as to prevent leakage.
- Chain-of-Custody (COC) documents will be placed in zip-lock bags and taped to the inside lid of each cooler.
- Cooler lids will be secured by wrapping with filament tape.
- The air bill will be secured to the handle of the cooler for the shipment label.
- Place Fragile and perishable stickers on all coolers. If shipping for Saturday delivery, place multiple Saturday Delivery stickers on each cooler and contact the laboratory to confirm receiving staff will be present.
- Confirm arrangements with the laboratory point-of-contact for Saturday delivery samples so that hold times and/or sample preservation are not compromised.

Custody seals will be used for sample shipments in accordance with SOP 7, Sample Control and Documentation. Custody seals are adhesive labels that are placed in such a manner that they will be visibly disturbed upon opening the shipping container or cooler. The seals will be initialed and dated upon placement. Upon receipt at the laboratory, the sample custodian will note the condition of custody seals and will also check the sample temperature, recording these items on the laboratory receipt form.

5.3 Closeout

Before leaving the site daily, the following procedures will be performed by the FTL or designated field staff:

- Ensure that the sample transport containers are properly packed and are in compliance with DOT and IATA regulations.
- Complete the Sample Handling, Packing & Shipping Checklist (Attachment 8-1).

6 Data and Records Management

All field forms and log book entries will be scanned and copied project folder on the “Z” drive within one week of the field event completion.

7 Quality Control and Quality Assurance

Work will be performed in accordance with the Quality Assurance Project Plan (QAPP), the specific work plan, and applicable SOPs. The Sample Handling, Packing & Shipping Checklist will be completed each day that samples are shipped. No erasures or mark outs will be made on the checklist. A single line will be used to strike out errors and will be annotated with the initials and date of the editor.

8 References

MACTEC *RA SAP Volume I: Field Sampling Plan, Defense Depot Memphis, Tennessee, Revision 1*, *WTP-9 Sample Packaging and Shipping*. November, 2005.

SESDPROC-209-R2, *Operating Procedure: Packing, Marking, Labeling and Shipping of Environmental and Waste Samples,* 2011.

Attachment 8-1

Sample Handling, Packing & Shipping Checklist

When preparing samples for shipment to the laboratory, complete this checklist to ensure that samples, documents, and materials are properly packed in the sample shipper.

Sample Event: _____

Date: _____

PROJECT SAMPLES

□ All samples, duplicates, MS/MSDs, equipment blanks, ambient blanks, and trip blanks should be included in the cooler that are listed on the COC.

□ Verify that the proper number of bottles with appropriate preservative(s) were collected for each sample

□ Verify that samples were checked for pH (except volatile samples)

DOCUMENTS

□ **Chain-of-Custody (COC)** generated for *each* cooler

□ COC reviewed for completeness, including appropriate signature(s) and date(s), and include the **courier tracking/shipping number** on the COC

□ **COC** placed in a Ziploc bag and taped to the underside of the cooler lid

□ **Custody seals** placed on the front and back of each cooler, or across the sealing tape for Summa canister cartons.

□ Coolers for Saturday delivery have “Saturday Delivery” stickers and “Saturday Delivery” box checked on the airbill

□ Shipments are insured

PACKING MATERIALS

□ Ice is “double-bagged” and is sufficient to maintain a temperature of 4°C

□ Glass bottles placed in a bubble bag to prevent breakage and leakage

□ All coolers have a bottom cushion in place prior to placing samples in the cooler.

□ Highly contaminated samples (if known) placed together

□ **Trip blank** placed in each cooler that contains samples for VOC analyses at beginning of day

□ All VOC samples placed in same cooler to minimize the number of trip blanks,

□ Each cooler contains a **temperature blank**

Comments: (special handling or delivery requirements, highly contaminated samples, etc.)

_____

_____

Number of coolers shipped: _____

Checklist Completed By: _____ Date: _____

Note: Place the completed checklist in the project file with the associated COCs and airbill.

STANDARD OPERATING PROCEDURE 9 – EQUIPMENT DECONTAMINATION

Lead Organization: Department of the Army (DA)
Preparing Organization: HDR
SOP Approved by: Field Team Leader: Eric North
Project QA Officer: Lynn Lutz
Project Manager: Tom Holmes

1 Purpose and Summary

This Standard Operation Procedure (SOP) provides guidance for proper decontamination of equipment used in sampling and collection of equipment rinsates to evaluate effectiveness of decontamination procedures.

2 Health and Safety

General Information on Health and Safety requirements is provided in SOP 1. Each individual is required to have read and understood the Site Safety and Health Plan for the project and signed the acknowledgement sheet confirming their review.

Health and safety concerns for equipment decontamination include exposure to contaminants from sampling equipment. Nitrile gloves and safety glasses should be used during decontamination.

3 Personnel Qualifications and Responsibilities

Sampling equipment decontamination and rinsate sample collection will be directed by the Field Team Leader (FTL), an environmental professional (engineer, geologist or scientist) with experience in equipment decontamination and sampling activities. The field staff, environmental professionals or technicians, are responsible for following these procedures and seeking direction from the FTL when questions or problems arise.

4 Equipment and Supplies

The required equipment and supplies will consist of Alconox soap, deionized water (DI), tap water, paper towels, foil, and sample containers.

5 Procedure

Proper equipment decontamination will prevent cross-contamination of samples due to residual contamination from previous sample locations and spread of contamination via sampling equipment. Proper decontamination also supports the legal defensibility of data generated during site activities.

Decontamination procedures will be evaluated by the collection of equipment rinsate samples. These samples consist of reagent water collected from final rinse of sampling equipment after the decontamination procedure has been performed. The samples are analyzed with the environmental sample to assess the adequacy of the decontamination performed.

5.1 Start-Up Activities

5.1.1 Office

The FTL will confirm that sufficient equipment and supplies are available at the site based on the number of samples and estimated field days.

5.1.2 Field

After arrival on site, but prior to commencement of operations, the FTL will confirm that decontamination supplies and equipment are available on site and review procedures with field staff.

5.2 Field Operations

5.2.1 Decontamination Area

The location of the decontamination area, used primarily for larger pieces of equipment, will be determined in consultation with subcontractor personnel. The decontamination pad will include a sump lined with 6-mil polyethylene sheeting to collect the decontamination water. The sump will be constructed by either excavating a small area to create a depression or by elevating the edges of the sheeting. Existing concrete pads with containment areas can be used for large equipment like drill rigs. Small handheld equipment will be decontaminated in 5-gallon buckets in order to contain the water.

5.2.2 Decontamination Water Source

Potable water from the municipal water system will be used as a rinse in the decontamination procedure. The FTL will be responsible for coordinating with the subcontractor personnel to secure an adequate supply of potable water for decontamination procedures. If large quantities of water are to be used, the subcontractor will rent a water meter from Memphis Light Gas and Water (MLGW). For smaller amounts, the field office water supply can be used.

5.2.3 Decontamination Procedures

The required decontamination procedure for large pieces of equipment such as drill rigs, auger flights, and drilling and well casing is:

1. Wash the external surface of equipment or materials with high pressure hot water and Alconox or equivalent, and scrub with brushes if necessary until all visible dirt, grime, grease, oil, loose paint, rust flakes, etc., have been removed from the equipment.
2. Air dry.
3. Decontamination waste water will be stored at the site and analyzed prior to disposal.

The required decontamination procedure for sampling equipment except the water level indicator probe is:

1. Wash and scrub with Alconox solution (or equivalent) and nylon brushes.
2. Double tap water rinse.
3. Rinse with American Society for Testing and Materials (ASTM) Type II Reagent - Grade Water
4. Wrap in oil free aluminum foil for transport.
5. Collect all decontamination rinse water in 5 gallon buckets. Rinse water will be combined with other wastewater generated during sampling activities and disposed of according to the work plan.

During water level sweeps and measurements in low-flow sampling, the water level tape and indicator in contact with groundwater will be decontaminated before initial use and before moving to a new location. The decontamination procedure for the water level indicator is:

1. Hand wash the calibrated tape and probe with Alconox solution (or equivalent).
2. Rinse with deionized (Reagent Grade II) water.

5.2.4 Equipment Rinsate Collection

When non-dedicated sampling equipment is used, the equipment will be decontaminated before initial use and after each sample is collected. An equipment rinsate sample will be collected for equipment type (bladder pump or bailer). At least one equipment rinsate will be collected for each sampling protocol (i.e. soil sampling, bladder pumps used for groundwater sampling) during each week of sampling. Equipment rinsate samples will be collected to be representative of field decontamination procedures.

Sampling Equipment: Equipment rinsate samples will be obtained from decontaminated bladder pumps, bailers, stainless steel split-spoons, hand augers, and stainless steel bowls with ASTM Type II water or better.

The equipment rinsate protocol will be as follows:

a. Label Sample Container - Label the sample container as outlined in SOP 7 – Sample Control and Documentation.

b. Collect Sample - After sample collection and equipment has been decontaminated as described above, an equipment rinsate will be collected. ASTM Type II water (or better) will be poured over and through the sampling equipment into a cleaned stainless steel bowl (preferably the equipment and bowl to be used on a specifically identifiable sample location). The collected water will be poured into the appropriate sample container. Repeat the process as necessary to fill each container to the required volume. Vials for volatile analysis and bottles for total organic carbon (TOC) analysis will be completely filled, leaving no air space above the liquid portion (to minimize volatilization). Check that the Teflon on the Teflon- lined silicone septum is toward the sample in the caps and secure the cap tightly. If semi-volatile compounds are to be sampled for, collect these samples next. Proceed to the collection of

samples for the remaining analyses. Be careful of all pre-preserved bottles. If acids are present, open the bottle downwind and away from the body.

c. Custody, Handling and Shipping - Complete the procedures as outlined in SOP 7 – Sample Control and Documentation and SOP 8 - Sample Packing and Shipping.

5.3 Closeout

Before leaving the site daily, the following procedures will be performed by the FTL or designated field staff:

- Confirm all equipment is decontaminated and properly stored all equipment.
- Add decontamination rinse water to the wastewater storage tank
- Note equipment decontamination activities and rinsate sample collection on the Daily Field Report (SOP 1, Attachment 1-2).

6 Data and Records Management

All field forms and log book entries will be scanned and copied project folder on the “Z” drive within one week of the field event completion.

7 Quality Control and Quality Assurance

Work will be performed in accordance with the Quality Assurance Project Plan (QAPP), the specific work plan, and applicable SOPs.

8 References

HDR, 2017a. *2017 Uniform Federal Policy-Quality Assurance Project Plan, Environmental Restoration Support at Former Defense Depot Memphis, Tennessee, Revision 0*. Prepared for the U.S. Army Corps of Engineers, Mobile District. May 2017.

SESDPROC-205-R2, *Operating Procedure Field Equipment Cleaning and Decontamination,* 2011.

STANDARD OPERATING PROCEDURE 10 – DATA VERIFICATION, VALIDATION, QUALIFICATION AND USABILITY ASSESSMENT

Lead Organization: Department of the Army (DA)
Preparing Organization: HDR
SOP Approved by: Project Chemist: Lynn Lutz
Project Manager: Tom Holmes

1 Purpose and Summary

This Standard Operating Procedure (SOP) provides guidance for the data verification, validation and usability assessment (hereafter called “data review” to denote all three stages) performed for analytical data generated for groundwater and vapor samples collected at Defense Depot Memphis, Tennessee (DDMT).

2 Health and Safety

There are no health and safety issues associated with the activities described in this SOP.

3 Personnel Qualifications and Responsibilities

Initial data review will be performed by a subcontractor, Diane Short & Associates (DSA), a professional data review company with expertise in reviewing data for Department of Defense (DoD) projects.

Final data review will be performed by the DDMT Project Chemist (PC), who will be familiar with the sampling areas and data requirements at DDMT and experienced in data review.

4 Equipment and Supplies

A computer loaded with Microsoft Excel, Microsoft Word and Adobe Acrobat (reader level or higher) is required.

5 Procedure

This section describes the data qualifiers that will be applied to the data during the verification and validation steps of the data review, and how the determination of usability will be performed. General guidelines for final qualification are provided; individual circumstances for data packages or specific samples may result in different qualification.

To maintain comparability among data sets for the entire DDMT project, the data validation guidelines in Appendix E, Data Quality Evaluation SOPs, of the previous version of the DDMT Quality Assurance Project Plan (QAPP) (MACTEC, 2005), the United States Environmental

Protection Agency (USEPA) National Functional Guidelines (USEPA, 2016), and the Quality Systems Manual (QSM) 5.1 have been incorporated herein.

Refer to Sections 1 and 2 and Worksheets #12, #19, #24 and #28 of the QAPP for the quality control limits to be used for data validation.

Final qualifiers will be:

- No qualification
- Non-detect (U)
- Detected and estimated (J)
- Detected and estimated with possible low bias (J-)
- Detected and estimated with possible high bias (J+)
- Non-detect and estimated (UJ)
- Rejected (R)

5.1 Chain-of-Custody

If the chain-of-custody (COC) form was not received by the laboratory with the sample, was not signed with date and time by the sampler in the “relinquished by” box, and/or was not signed with date and time by the lab’s sample receipt personnel in the “received by” box, the legal trail of custody may be compromised. A copy of the COC will be sent to the lab and the PC by the Field Team Leader (FTL) following sample shipment. The PC will examine sample receipt documentation and call or email the lab when discrepancies are identified. Custody seals should be noted as unbroken.

5.2 Sample Receipt

5.2.1 Water Samples

Water samples should arrive at the lab between 0 degrees Celsius (ºC) and 6ºC. If water samples were received warm, the lab will contact the PC immediately. The PC and Project Manager (PM) will determine whether samples should be analyzed or re-collected. If samples are analyzed and reported, generally all results will be qualified as estimated (J), estimated with possible low bias (J-), or non-detect estimated (UJ).

5.2.2 Air Samples

Air samples have no temperature requirements.

5.3 Holding Times and Preservation

For samples analyzed past their holding time, generally all results will be qualified as estimated with possible low bias (J-) or non-detect estimated (UJ) unless holding times are grossly exceeded.

5.4 Method Identification, Analyte List, and RLs/MDLs

The correct methods (SW-846 8260B, SW-846 9060 modified, SW-846 6010, RSK-175 and 830-MBA for waters, TO-15 for air samples) used for analysis must be identified on the sample result pages. If an incorrect method was used, the lab may be instructed to reanalyze samples using the correct method.

If the list of reported analytes is incorrect, or incorrect reporting limits (RLs) and methods detection limits (MDLs) are reported, the lab will be requested to report the correct analyte list or the correct RLs and MDLs.

5.5 Gas Chromatography/Mass Spectrometry Tuning and Analytical Sequence

If tuning requirements were not met, the lab should not have proceeded with sample analysis. If samples were analyzed and reported after an unacceptable tune with 4-bromofluorobenzene (BFB), this will be brought to the attention of the lab PM, and it should have been mentioned in the Case Narrative.

For volatile organic compounds (VOCs) in water and air the critical ion abundance criteria for BFB are the m/z 95/96, 174/175, 174/176, and 176/177 ratios. The relative abundances of m/z 50 and 75 are of lower importance. Samples reported after an unacceptable tune may be rejected (R), or qualified as estimated (J) and non-detect estimated (UJ), according to the reviewer's judgment.

Analysis of all field and QC samples must begin within 12 hours (for waters) or within 24 hours (for air samples) of a valid BFB tune. If sample analysis began later than required, sample results will be qualified as estimated (J) or non-detect estimated (UJ). If analysis began only a short time (within 15 minutes) after the required interval, the results need not be qualified.

5.6 Initial Calibration

Initial calibration Relative Response Factors (RRFs) and % Relative Standard Deviations (RSDs) will be examined to determine whether they met required control limits.

5.6.1 Water Samples

VOC analytes with a %RSD greater than 15% should have had a linear curve fit with an r value of at least 0.995 or a quadratic curve fit with an r^2 value of at least 0.990, or the average %RSD of all analytes in the calibration curve must be 15% or less. Calibration check compounds (CCCs) must have %RSDs less than or equal to 30%. Analytes outside these limits will be qualified as estimated (J) or non-detect estimated (UJ).

A number of VOC analytes (shown below) are considered poor responders and have less stringent requirements for minimum RRF.

Poor Responders		
Acetone	Chloroethane	1,2-Dibromoethane (EDB)
2-Butanone	Chloromethane	1,2-Dibromo-3-chloropropane

Poor Responders		
2-Hexanone	Dichlorodifluoromethane	cis-1,2-Dichloroethene
4-Methyl-2-pentanone	Trichlorofluoromethane	trans-1,2-Dichloroethene
Carbon disulfide	Methyl tert-butyl ether (MTBE)	1,2-Dichloropropane
	Isopropylbenzene	Methylene chloride

All VOC analytes except the poor responders should have an RRF of at least 0.05. The poor responders should have an RRF of at least 0.01. System performance check compounds (SPCCs) must have RRFs of at least 0.1 or 0.3 as required by the method. Analytes outside these limits will be qualified as estimated (J) or non-detect estimated (UJ).

Initial calibrations for other analytes that do not meet requirements will be qualified in a similar manner as VOCs.

5.6.2 Air Samples

Analytes with a %RSD greater than 30% will be qualified as estimated (J) or non-detect estimated (UJ).

5.7 Initial Calibration Verification (Second Source Standard)

A second source standard must be analyzed after every initial calibration. An LCS can serve as a second source standard for VOCs or dissolved gases as long as it can be determined from the standard prep sheets of instrument run logs that a different standard than those used for the calibration curve was used.

5.7.1 Water Samples

Any analyte with a %D (difference or drift) greater than the control limit compared to the initial calibration will be qualified as estimated (J) or non-detect estimated (UJ).

5.7.2 Air Samples

Any analyte with a %D (difference or drift) greater than 30% compared to the initial calibration will be qualified as estimated (J) or non-detect estimated (UJ).

5.8 Continuing Calibrations

5.8.1 Water Samples

VOC CCCs must have %D values less than or equal to 20%. Other analytes should have %D values less than or equal to 20%. Analytes outside these limits with lower responses than the initial calibration will be qualified as estimated with possible low bias (J-) or non-detect estimated (UJ). Detected analytes outside these limits with higher responses than the initial calibration will be qualified as estimated with possible high bias (J+).

All VOC analytes except the poor responders should have an RRF of at least 0.05. The poor responders should have an RRF of at least 0.01. SPCCs must have RRFs of at least 0.1 or 0.3 as

required by the method. Analytes outside these limits will be qualified as estimated (J) or non-detect estimated (UJ).

Any other analyte with a %D (difference or drift) greater than the control limit compared to the initial calibration will be qualified as estimated with possible low bias (J-) , estimated with possible high bias (J+), or non-detect estimated (UJ), as in Section 5.8.1 above.

5.8.2 Air Samples

All analytes must have %D values less than or equal to 30%. Analytes outside these limits with lower responses than the initial calibration will be qualified as estimated with possible low bias (J-) or non-detect estimated (UJ). Detected analytes outside these limits with higher responses than the initial calibration will be qualified as estimated with possible high bias (J+).

5.9 Blanks

5.9.1 Method Blank

There must be a method blank associated with each sample. Method blanks should contain no COCs above one-half the RL. Analytes detected above the RL should be discussed in the Case Narrative.

Analytes detected in the samples as well as the method blank will be qualified as non-detect (U) or will not be qualified, in accordance with the qualification as discussed in the applicable National Functional Guidelines (USEPA, 2016).

5.9.2 Trip Blank

A trip blank must accompany all VOC water samples during sampling and shipment, in the same cooler. Trip blanks are not required for air samples.

Analytes detected in the samples as well as the trip blank will be qualified as for a method blank.

5.9.3 Rinsate Blank

A rinsate blank must be collected periodically when non-dedicated sampling equipment is used to collect water samples. Rinsate blanks are not required for air samples.

Analytes detected in the associated samples as well as the rinsate blank will be qualified as for a method blank.

5.10 Laboratory Control Sample and Duplicate

There must be a laboratory control sample (LCS) associated with each sample. There may also be a laboratory control sample duplicate (LCSD), although this is not required. LCSs must be spiked with all COCs.

Analytes with recoveries above the control limits may be biased high and will be qualified as estimated with possible high bias (J+) when detected; non-detect results will not be qualified. Analytes with recoveries below the control limits may be biased low and will be qualified as estimated with possible low bias (J-) or non-detect estimated (UJ). If an LCSD is also analyzed, analytes with relative percent difference (RPD) values greater than 20% (30% for VOCs in air) will be qualified as estimated (J) when detected; non-detect results will not be qualified. All samples associated with the LCS will be qualified.

5.11 Matrix Spike and Matrix Spike Duplicate

MS/MSD samples will be indicated on the COC. Matrix spike/matrix spike duplicate (MS/MSD) samples must be spiked with all COCs.

5.11.1 Water Samples

One set of MS/MSD samples will be collected for every 20 field samples. Recovery limits are the lab's in-house control limits. Analytes with higher recoveries may be biased high and will be qualified as estimated (J) when detected; non-detect results will not be qualified. Analytes with lower recoveries may be biased low and will be qualified as estimated with possible high bias (J+) or non-detect estimated (UJ). Analytes with RPD values greater than 20% will be qualified as estimated (J) when detected; non-detect results will not be qualified. Only the parent sample will be qualified.

5.11.2 Air Samples

MS/MSD samples are not collected for air samples.

5.12 Field Duplicates

Field duplicate samples will be sent blind to the laboratory. They will be designated on the COC but not identified with a specific sample location. One field duplicate sample will be collected for every 10 field samples.

Analytes detected above the RL should agree within the RPD control limit. Sample results outside this control limit will be qualified as estimated (J). Results detected below the RL will not be assessed. If one result is above the RL and the other result is below the RL, both results will be qualified as estimated (J). If one result is above the RL and the other result is non-detect, the detected result will be qualified as estimated (J) and the non-detect result will be qualified as non-detect estimated (UJ).

5.13 Laboratory Duplicates

5.13.1 Water Samples

Laboratory duplicates may be analyzed for metals in water samples. Control limits and qualification are the same as for a field duplicate.

5.13.2 Air Samples

A laboratory duplicate of an air sample must be analyzed daily. Laboratory duplicate results are assessed only if the duplicate was on a DDMT sample. Control limits and qualification are the same as for a field duplicate.

5.14 Surrogates

Surrogates are spiked into every field sample, quality control (QC) sample, and standard for VOCs in water and air.

Surrogates with recoveries above the control limits may indicate a high bias in detected sample results; all detected analytes in the sample will be qualified as estimated with possible high bias (J+). Surrogates with recoveries below control limits may indicate a low bias in sample results; all analytes in the sample will be qualified as estimated with possible low bias (J-) or non-detect estimated (UJ). Samples will not be qualified if only one surrogate out of three or four has a high or low recovery.

5.15 Internal Standards

Internal Standards are spiked into every field sample, QC sample, and standard for VOCs in water and air.

Internal standards with recoveries or retention times outside control limits may indicate interferences in the sample matrix or poor purging.

All analytes associated with an internal standard that has an area outside control limits will be qualified as estimated (J) or non-detect estimated (UJ).

If an internal standard has a retention time outside control limits, the chromatogram and quantitation report will be examined to determine possible impact on the detected or non-detected sample results. Retention times outside control limits may lead to false positive or false negative results for other analytes.

5.16 Usability Assessment

The HDR PC will assess the Precision, Accuracy/bias, Representativeness, Comparability, Completeness, and Sensitivity (PARCCS) parameters and determine overall usability of the data. In general, non-rejected data will be considered usable. Measurement error will be deemed within acceptable limits when project data quality objectives (DQOs) as assessed by PARCCS parameters are met. The PC will write a brief assessment of data usability for each data package.

6 Data and Records Management

This section details the distribution of data files from the laboratories to HDR, DSA, and the project files.

6.1 Data Files from Laboratories

The laboratories will email to the HDR PC the Level IV data package in PDF format, an electronic data deliverable (EDD) file in Excel, and the ERPIMS files (Sample, Test and Result) to the PC. The PC will save these files to the appropriate folders on the HDR network drive, to be retained in perpetuity. The laboratories will also send PDF Level IV data packages directly to DSA.

Hardcopy (paper) data files are not required for this project.

6.2 Data Files from HDR

The HDR PC will email the PDF Level IV data package and the Excel EDD to the HDR Project Manager (PM). The PC will email the Excel EDDs to DSA.

6.3 Data Files at DSA

DSA will perform data review (the three steps described in Section 3 of this SOP) and write a report that summarizes what was reviewed, what decisions were made, and what qualifiers were applied to the data. DSA's report format (redacted for confidentiality and to omit sections related to inorganic samples) also serves as their SOP and is located in Attachments 10-1 through 10-4 of this SOP.

DSA will add a field to the Excel EDD file and apply qualifiers as discussed in their report.

6.4 Data Files from DSA to HDR

When DSA has completed the data review, they will email the report and the EDD file to the HDR PC.

6.5 Data Files at HDR

The PC will save the report and EDD file to the appropriate folders on the HDR network drive. The PC will make a copy of the EDD file, with the same name plus "-final".

The PC will review the DSA report and prepare a final validation report noting any changes to the DSA report with a discussion of why the changes were made. An example of this report is included as Attachment 10-5 to this SOP.

The PC will open the "final" file and add a column before the analytes column, called "Report?"; all entries will default to "Y(es)", and will be changed to "N(o)" in the event of multiple runs and/or dilution runs of a sample, so that there is only one reportable result per analyte in all samples. The PC will also add a field named "Final Qualifier" at the end of the fields, and record final qualifiers as described in Section 5.6.1 of this SOP. Qualifiers for undetected results (U) do not all need to be copied to this field, unless the final qualifier is UJ or R, which will be entered into this field.

When all the EDDs have been completed for a sampling event, the PC will email the database manager the location of the files, or the files themselves, to create result tables to be used in reports.

7 Quality Control and Quality Assurance

All work will be performed in accordance with the QAPP, the specific work plan, the specific sampling plan details (SPD) and applicable SOPs.

8 References

MACTEC Engineering and Consulting, Inc. (MACTEC), 2005. *Remedial Action Sampling and Analysis Plan, Volume II: Quality Assurance Project Plan, Defense Depot Memphis, Tennessee, Revision 1*. November 2005.

United States Environmental Protection Agency (USEPA), 2016. *National Functional Guidelines for Superfund Organic Methods Data Review (EPA-540-R-2016-002).* September 2016.

USEPA, 2016. *National Functional Guidelines for Inorganic Superfund Methods Data Review (EPA-540-R-2016-001).* September 2016.

Attachment 10-1

EXAMPLE: DSA Organic Data Quality Review Report – Water Samples

Volatile Organics by SW-846 Method 8260B (GC/MS)

SDG	
PROJECT	Memphis Defense Depot, LS-13 Sampling for HDR, Denver
LABORATORY	
SAMPLE MATRIX	Water SAMPLING DATE
NUMBER OF SAMPLES	77, including 6 trip blanks, 1 rinse blank, and 7 field duplicates
ANALYSES REQUESTED	SW-846 8260B (VOA by GC/MS)
SAMPLE NUMBER	See result forms attached and associated EDD

DATA REVIEWER: _____

QA REVIEWER: Diane Short & Associates, Inc. INITIALS/DATE: _____

Telephone Logs included Yes____ No __X_

Contractual Violations Yes____ No __X_

The project QAPP (11/05), the EPA Contract Laboratory Program National Functional Guidelines for Organic Review, 1999 and 2001, and the SW-846 Method 8260B have been referenced by the reviewer to perform this data validation review. The EPA qualifiers have been expanded to include a descriptor code and value to define QC violations and their values, per the approval of the Project Manager. Per the Scope of Work, the review of these samples includes Level III validation of all chains of custody, calibrations and QC forms referencing the QC limits in the above documents.

Attachment 10-1

I. DELIVERABLES

A. All deliverables were present as specified in the Statement of Work (SOW), SW-846, or in the project contract.

Yes __X__ No _____

This report has been requested to include the following review: QC, hold times, sample integrity (Chains-of-Custody [COCs], sample login), and summary calibrations.

B. COC documentation was complete and accurate.

Yes __X__ No _____

No qualifiers have been added for COC issues, and the Project Manager will update COC per the following notes to complete the project record. These chains were complete.

C. Samples were received at the required temperature, preservation and intact with no bubbles.

Yes __X__ No _____

EPA regulations (see Federal Register, March 12, 2007, 40CFR Part 122) require only that the temperature of samples delivered to the laboratory be equal to or less than 6°C. The sample temperature conditions are fully compliant with applicable regulations.

SDG L110xxxxx: The laboratory notes that TB-5 samples had headspace > 6 mm.

Analyses were performed on samples without headspace. As these are trip blanks, the client data are not considered to be impacted.

II. ANALYTICAL REPORT FORMS

A. The Analytical Report or Data Sheets are present and complete for all requested analyses.

Yes __X__ No _____

B. Holding Times

1. The contract holding times were met for all analyses [time of sample receipt to time of analysis (VOA) or time of extraction to time of analysis].

Yes __X__ No _____

2. The Clean Water Act (40 CFR 136) or method holding times were met for all analyses (14 days from time of sample collection to analysis or extraction).

Yes __X__ No _____

Attachment 10-1

III. INSTRUMENT CALIBRATION – GC/MS

A. Initial Calibration

1. The Response (RF) and Relative Response Factors (RRF) and average RRF for all compounds for all analyses met the contract criteria of > 0.05 or > 0.01 for poor responding volatiles.

Yes __X__ No _____ NA _____

Method 8260: Per the Project Manager, the 2001 EPA CLP validation guidance has been applied to the common “poor responders.” Calibration response factors below 0.05 have historically been observed for Acetone, 2-Butanone, and 4-Methyl-2-pentanone. The validation guidance used for this project allows for a response of 0.01 for these compounds if spectral integrity can be verified at low concentrations. These spectra are not commonly provided and are not part of the deliverable for this data set. The laboratory has been tasked with providing to the client verification that the 0.01 RF is valid. If the spectral verification is available, data are not qualified for response factors greater than 0.01 and less than 0.05 for “poor responders.” No data have been qualified.

Most of the low-responding compounds are highly water-soluble and capable of hydrogen bonding with water. This decreases their purge efficiency and results in the relatively low response. The implication of this low purge efficiency is that a relatively low absolute recovery of such compounds is achieved in the purge step of the analysis. If this recovery is consistent, reasonable accuracy and precision can be achieved in a given matrix, which is indicated for the lab matrix by acceptable recoveries in LCS and calibration checks. However, this causes these targets to be more sensitive to matrix variations that impact purge efficiency (such as ionic strength or the presence of varying levels of soluble non-target organic material) than are the more hydrophobic compounds typically analyzed by this method, and as a result they are more likely to exhibit matrix bias.

2a. The relative standard deviation (RSD) for the five point calibration was within the 30% limit for the CCCs.

Yes __X__ No _____ NA _____

This is a method requirement and indicates that the analytical system is in control.

2b. The RSD for the five point calibration was within the 30% limit for all other compounds, the average % RSD was < 15%, or a linear curve was used.

Yes __X__ No _____ NA _____

3. The 12 hour system Performance Check was performed as required in SW-846.

Yes __X__ No _____ NA _____

B. Continuing Calibrations

1. The midpoint standard was analyzed for each analysis at the required frequency, and the QC criterion of > 0.05 (> 0.01 for CLP 2001 VOA) was met.

Yes __X__ No _____ NA _____

Attachment 10-1

2. The percent difference (% D) limits of ± 25% were met. The 2001 NFG also allow for 40% D for the "poor responders." For other compounds, the QAPP notes rejection of detected compounds with % D > 40%.

Yes _____ No __X__ NA _____

See the table below. When there are no detections, no qualification is required unless the % D is biased low and so large as to indicate a significant probability of false negatives. Qualification is required for a % D biased high for detected compounds only. This requires that the RF is acceptable to verify the non-detect status, which is the case here. Data are qualified "JC#", where # is the % D. There could be variability to the data as there is variability to the response.

The QAPP indicates that compounds in a run should be rejected if the % D is > 40%. We interpret this to mean that non-detects should be rejected and that detected compounds should be "J" qualified, which is the normal validation process for rejection. Professional judgment is that high bias CCVs with a % D greater than 40% should not be rejected for non-detects if the response factors are sufficient to ensure verification of the non-detect. Non-detects with % D values > 40% are qualified "RC#", where # is the % D.

The table below shows the outliers observed in CCVs for this report.

SDG	Batch	Analyte	% D	Bias	Qualifier
L110xxxxx	WGxxx	Bromomethane	27.5	high	None, ND
	WGyyy	Bromomethane	33.6	high	None, ND

IV. GC/MS INSTRUMENT PERFORMANCE CHECK

The BFB (VOA) performance check was injected once at the beginning of each 12-hour period and relative abundance criteria for the ions were met.

Yes __X__ No _____ NA _____

V. INTERNAL STANDARDS

The Internal Standards met the 100% upper and -50% lower limits criteria, and the Retention times were within the required windows.

Yes __X__ No _____ NA _____

VI. SURROGATE STANDARDS

Surrogate spikes were analyzed with every sample.

Yes __X__ No _____

And met the recovery limits defined in the QAPP of 70 – 130% for VOA water or 75 – 125% for soil samples.

Attachment 10-1

Yes __X__ No _____

VII. MATRIX SPIKE/MATRIX SPIKE DUPLICATE

A. Matrix Spike (MS) and Matrix Spike Duplicate (MSD) were analyzed for every analysis performed and for every 20 samples or for every matrix whichever is more frequent.

Yes __X__ No _____

There are eight MS/MSD samples associated with the 77 non-QC client samples. This meets the recommendation of 1 per 20 field samples. As this is an ongoing project an overall adherence to the 1:20 frequency is monitored by the Project Manager.

B. The MS and MSD percent recoveries were within the limits defined in the QAPP of VOA at 70 – 130% with five compounds allowed to be within 60 – 140%.

Yes _____ No __X__ NA _____

The full target list has been spiked. Qualifiers are added for all outliers as described here. Please refer to the project EDD for a detailed list of qualifiers added.

The table below shows the outliers observed in MS/MSD samples for this report. Results have been qualified per the QAPP as “JMS#”, where # is the % recovery. Data could be biased high or low in approximate proportion to the spike recovery. Only detected data are qualified for high recovery. Data are not qualified if four times (4x noted in table) =the spike amount is less than the amount in the parent sample. Only the parent sample is qualified.

SDG	Parent Sample	Analyte	MS	MSD	Bias	Qualifier
L110xxxxx	XXX	Carbon disulfide	124	132	high	None, ND

C. The MSD relative percent differences (RPD) were within the defined contract limits for VOA of 30% water or 40% soil, with five compounds allowed to be > 40% RPD.

Yes __X__ No _____ NA _____

Qualifiers are added only when the MS or MSD recovery is also out of limits. Data are qualified “JD#”, where # is the RPD. As the RPD increases, the matrix precision decreases.

D. The MS/MSD were client samples.

Yes __X__ No _____ NA _____

VIII. LABORATORY CONTROL SAMPLE

A. Laboratory Control Sample (LCS) was analyzed for every analysis performed and for every 20 samples.

Yes __X__ No _____

Attachment 10-1

B. The LCS percent recoveries were within the limits defined in the QAPP for VOA of 80 – 120% for water or 75 – 125% for soil. Five compounds are allowed to be 60 – 140%. If an LCS and LCSD are analyzed, both samples must have the same compounds out for data to be qualified.

Yes _____ No __X__

The full target list has been spiked. When a high LCS recovery is associated with a non-detect in samples, no qualifier is added since the indicated bias is high. When the target is detected, data are qualified "JL#", where # is the recovery. Data could be biased high proportional to the LCS percent recovery. All results associated with low recoveries are qualified, and data could be biased low.

The table below shows the outliers and the limits applied per the QAPP. The limits are specified based on the matrix. Qualifiers are added for all outliers as described here, but the Project Manager may consider reversing some of these when the limits fall within the marginal exceedance limits (60 – 140%). Please refer to the project EDD for a detailed list of qualifiers added.

SDG	Batch	Analyte	% Recovery (LCS/LCSD)	Bias	Qualifier
L110xxxxx/ L110xxxxy	WGxxx	Dichlorodifluoromethane	129	high	None, ND

IX. BLANKS

A. Method Blanks were analyzed at the required frequency and for each matrix and analysis.

Yes __X__ No _____

B. No blank contamination was found in the Method Blank.

Yes __X__ No _____

C. If Field Blanks were identified, no blank contamination was found.

Yes _____ No __X__ NA ____

When analytes are present in both the Field Blank and the associated samples, the results in the samples are qualified in the same manner as for Method Blanks. For clarity, the qualifiers used in this case are "UTB#" for Trip Blanks or "UFB#" for Rinse Blanks, where # is the associated blank value. Results added are shown in the table below. No qualifiers have been applied.

SDG	Sample	Analyte	Result	Qualifier
L110xxxxx	XXX	Methylene Chloride	0.538	None, ND

X. FIELD QC

If Field Duplicates were identified, they met guidance for VOA of RPD of < 30% for water or < 50% for soil. For values reported at < 5 × the reporting limit (RL), a difference of 2 × RL for water or 3.5 × RL for soil samples is used as guidance. This is referred to as the CRDL Rule. Data are not qualified for Field Duplicates as these are evaluated for the total project by the Project Manager.

Attachment 10-1

Yes _____ No __X__ NA _____

There are xxx identified field duplicates as described in the following table.

SDG	Parent Sample	Field Duplicate	Observations
L110xxxxx	XXXMW-21-LS-13	DUP-1	OK

XI. SYSTEM PERFORMANCE

A. The RICs, chromatograms, tunes and general system performance were acceptable for all instruments and analytical systems.

Yes _____ No _____ NA __X__

Not part of this review level.

B. The suggested EQLs for the sample matrices in this set were met.

Yes __X__ No _____ NA _____

XII. TCL COMPOUNDS

A. The identification is accurate and all retention times, library spectra and reconstructed ion chromatograms (RIC) were evaluated for all detected compounds.

Yes _____ No _____ NA __X__

Not part of this review level.

B. Quantitation was checked to determine the accuracy of calculations for representative compounds in each internal standards quantitation set.

Yes _____ No _____ NA __X__

Not part of this review level.

XIII. TENTATIVELY IDENTIFIED COMPOUNDS

TICs were properly identified and met the library identification criteria.

Yes _____ No _____ NA __X__

Not part of this review level.

XIV. OVERALL ASSESSMENT OF THE CASE

The laboratory has complied with the requested method. Data are fully usable after consideration of qualifiers. The following is noted:

Sample Preservation

Attachment 10-1

EPA regulations (see Federal Register, March 12, 2007, 40CFR Part 122) require only that the temperature of samples delivered to the laboratory be equal to or less than 6°C. The sample temperature conditions are fully compliant with applicable regulations.

SDG L110xxxxx: The laboratory notes that XXX samples had headspace > 6 mm.

Analyses were performed on samples without headspace. As these are trip blanks, the client data are not considered to be impacted.

Continuing Calibrations

See the table within the body of this report. When there are no detections, no qualification is required unless the % D is biased low and so large as to indicate a significant probability of false negatives. Qualification is required for a % D biased high for detected compounds only. This requires that the RF is acceptable to verify the non-detect status, which is the case here. No qualifiers have been applied.

Matrix Spikes

There are eight MS/MSD samples associated with the 77 non-QC client samples.

See the table within the body of this report. Results have been qualified per the QAPP as “JMS#”, where # is the % recovery. Data could be biased high or low in approximate proportion to the spike recovery. Only detected data are qualified for high recovery. Data are not qualified if four times the spike amount is less than the amount in the parent sample. Only the parent sample is qualified. Data have been qualified with a low bias only for cis-1,2-dichloroethene in samples XXXX.

Laboratory Control Samples

See the table within the body of this report. When a high LCS recovery is associated with a non-detect in samples, no qualifier is added since the indicated bias is high. When the target is detected, data are qualified “JL#”, where # is the recovery. Data could be biased high proportional to the LCS percent recovery. Data for vinyl chloride and carbon tetrachloride have been qualified for slightly high recoveries.

Field Blanks

See the table within the body of this report. When analytes are present in both the Field Blank and the associated samples, the results in the samples are qualified in the same manner as for Method Blanks. For clarity, the qualifiers used in this case are “UTB#” for Trip Blanks or “UFB#” for Rinse Blanks, where # is the associated blank value. No data have required qualification.

Field QC

There are seven identified Field Duplicates. See the table within the body of this report. No data have been qualified.

Attachment 10-2

EXAMPLE: DSA Organic Data Quality Review Report – Air Samples

ORGANIC AIR QUALITY REPORT

METHOD TO-15

SDG: _____

PROJECT: Memphis Defense Depot DDMT Fluvial Soil Vapor Extraction for HDR Environmental, Operations and Construction Inc. (formerly e2m)

LABORATORY: Microbac, subcontracted to Columbia Analytical Services Laboratories, CA_____

SAMPLE MATRIX: Air_____

SAMPLING DATE (Month/Year): March, 2011_____

NUMBER OF SAMPLES: 9 samples including one field duplicate_____

ANALYSES REQUESTED: Summa Canister VOA TO-15_____

SAMPLE NO.: See project EDD for sample IDs_____

DATA REVIEWER: Diane Short_____

QA REVIEWER: Diane Short & Associates, Inc. INITIALS/DATE: _____

Telephone Logs included Yes____ No __X_

Contractual Violations Yes____ No __X_

The EPA CLP National Functional Guidelines for Organic Data Review, 2001 (SOP), EPA Method TO-15 current updates have been referenced by the reviewer to perform this data validation review. The EPA qualifiers have been expanded to include a descriptor code and value to define QC violations and their values, per the approval of the HDR/e2m Project Manager. Per the Scope of Work, the review of these samples includes validation of all QC forms and submitted calibrations referencing the QC limits in the above documents.

Attachment 10-2

I. DELIVERABLES

All deliverables were present as specified in the Statement of Work (SOW) or in the project contract.

Yes _X__ No ____

Note an extended list of volatile compounds was reported. Level III data packages were submitted and Level III validation was performed for holding times, chain-of-custody, calibrations and QC.

II. ANALYTICAL REPORT FORMS

A. The Analytical Report or Data Sheets are present and complete for all requested analyses.

Yes __X__ No___

B. Holding Times

The contract holding times were met for all analyses (Time of sample receipt to time of analysis (VOA) or extraction and from extraction to analysis). Contract holding times for TO-15 canisters is 30 days from date of collection.

Yes ___X__ No____

C. Chains of Custody

Chains of Custody were present and were complete with signatures, sign-offs and complete entry of data. Canisters were properly sampled and received.

Yes __X___ No ____

The project manager is informed of the following and the project record is being updated.

There are gaps from relinquishment to sample receipt. The courier is identified as FedEx and there is no airbill number on the chain or log-in. The client notes that the coolers are often sealed before the airbill number is known.

D. Canister Pressure

Canister pressures were measured and recorded for initial vacuum check, initial field vacuum, final field reading, lab initial pressure and final pressure.

Yes ___X___ No _____ NA _____

Pressures were reported for the field initial and final pressures and the laboratory final pressure. The pressure changes are not as large as for some other samplings, but these are highly contaminated samples most of which have required some dilutions even at the reported volume.

All readings met the limits or exceptions were noted and pressure corrected

Yes ___X__ No ____ NA ____

Attachment 10-2

III. INSTRUMENT CALIBRATION

A. Initial Calibration – GC/MS

1. The Relative Response Factors (RRF) and average RRF for all compounds for all analyses met the required criteria.

Yes __X__ No____ NA____

Minimum response factors are not defined by the method but meet routine Method 8260 limits. This method does not involve purging water samples. Consequently, all targets, including the typically poor-purging compounds, normally have response factors that are acceptable per validation criteria for volatiles.

The relative standard deviation (RSD) for the five-point calibration was within the 30% limit.

Yes ___X__ No ____

B. Continuing Calibration – GC/MS

1. The RRF standard was analyzed for each analysis at the required frequency and the QC criteria were met

Yes __X__ No____ NA____

Minimum response factors are not defined by the method, but met validation guidance. There were 3 calibrations as there were 2 days of analysis for dilutions.

2. The percent difference (%D) limits of 30% were met.

Yes __X__ No ____

Outliers were not client compounds.

IV. GC/MS INSTRUMENT PERFORMANCE CHECK

A. The BFB performance check was injected once at the beginning of each 12-hour period and relative abundance criteria for the ions were met.

Yes ___X__ No____ NA ____

Tunes were provided and were acceptable.

V. INTERNAL STANDARDS

A. Area Limits

The Internal Standards met the 100% upper and -50% lower limits criteria and the Retention times were within the required windows.

Yes __X___ No____ NA ____

Attachment 10-2

B. Retention Times

The relative retention times of the internal standards and sample compounds met the ± 0.06 RRT units limit.

Yes __ X ___ No____ NA ____

VI. SURROGATE

Surrogate spikes were analyzed with every sample.

Yes __X___ No ____

Note that three surrogates are used. Method 8260 requires 3 surrogates, but one is acceptable for TO-15.

And met the recovery limits defined in the current contract

Yes __ X _ No ____

VII. MATRIX SPIKE/MATRIX SPIKE DUPLICATE

A. Matrix spike (MS) and matrix spike duplicates (MSD) were analyzed for every analysis performed and for every 20 samples or for every matrix whichever is more frequent.

Yes____ No ____NA ___X__

Spikes are not amenable to canister analysis and are not required. Laboratory duplicates are required and are provided by the laboratory. See below.

B. The laboratory duplicate relative percent differences (RPD) were within the defined contract limits. Method requirements are 25% maximum RPD.

Yes ____ No ____ NA__X_

For validation purposes, only results > 5x PQL are qualified for RPD outliers. For results < 5x PQL, results are qualified if the absolute difference is greater than 2x PQL. The qualifier added is JD#, where # is the RPD or the absolute difference observed, as appropriate.

The laboratory duplicates was sample FSVE-SVEC-2Q11-NS. RPDs were reported above the limit, but these were low level results that met the 2 x PQL criteria.

VIII. DUPLICATE CONTROL SAMPLES

A. Duplicate Control and Duplicate Control Sample Duplicates similar to Laboratory Control Samples (LCS) were performed for every set.

Yes _X_ No ___

B. And percent recoveries were acceptable at 70 – 130%.

Attachment 10-2

Yes _X___ No ____

For air data, the laboratory limits are used as there are no air limits defined in the QAPP. There was an LCS reported for all 3 days of analysis.

C. And Relative Percent Differences were within lab limits.

Yes ___ No____ NA__X__

LCSDs have not been performed, and are not required by the method.

IX. SHIFT CHECKS

Shift checks were performed and were within time limits.

Yes __X__ No____

X. BLANKS

A. Method Blanks were analyzed at the required frequency and for each matrix and analysis.

Yes __X___ No____

This is a nitrogen blank run with each set.

B. The method blank was free of contamination.

Yes _____ No ___X__

Methylene chloride was reported in all 3 blanks

4/26/11 at 0.14 ug/m3; 4/28/11 at 0.16 ug/m3; 4/29 at 0.15 ug/m3.

The samples have been diluted for most reported results. The blank is multiplied by a factor determined from the reported MRL for each sample/compound. See the EDD and table at the end of the report for the final blank value as data are qualified BMB#, where # is the blank corrected value. Only data that are less than 10 x the blank (corrected for dilution) are qualified.

C. If Field Blanks were identified, they were free of contamination.

Yes _____ No ____ NA ___X___

There were no field blanks identified.

D. Contamination level was less than 0.03 mg/cubic meter before samples were analyzed per the method.

Yes __X__ No____ NA _____

Reporting units include both ppbv and ug/m3.

Attachment 10-2

XI. FIELD QC

A. If Field duplicates or Performance Check Compounds were identified, they met the RPD or % recovery criteria for the project.

Yes ___ No __X__ NA___

One field duplicate pair is reported: FSVE-SVEG-2Q11: -NS and DUP. These are regularly sampled locations and the precision is built in as the sites are sampled routinely. Qualifiers of JFD#, where # is the RPD (or the difference for low level values) are added for field duplicate differences. When results are > 5x the reporting limit, a 35% RPD is used to identify potential deviations. When results are < 5x the reporting limit, an absolute difference between the results that is < 2x PQL is considered to be acceptable reproducibility. (The laboratory uses MRL not PQL for these reports). Note that the times of collection indicate that these samples are not collected at the same time, but are sequential samples. An RPD of 50% is recommended for a precision limit for these samples. The QAPP does not define air QC.

Parent		DUP	RPD	Qualifier
Cis-1,2-dichlorothene	32	59 ug/m3	59	JFD59
Chloroform	1000	1800 ug/m3	57	JFD57
Carbon tetrachloride	190	360 ug/m3	62	JFD62
Trichloroethene	580 (not diluted)	960 ug/m3 (diluted)	49.3	JFD49
Tetrachloroethane	80	150 ug/m3	61	JFD61
1,1,2,2-tetrachloroethane	560	910 ug/m3	48	JFD48

XII. TCL COMPOUNDS

A. The identification is accurate and all retention times, library spectra and reconstructed ion chromatograms (RIC) were evaluated for all detected compounds:

Yes __X_ No____ NA___

It is noted that there are high dilutions for tetrachloroethane; 1,1,2,2-tetrachloroethane, trichloroethene and chloroform and some other compounds. No dilution factors are noted on the results forms but dilutions have been estimated from the MDLs. For compounds that are reported from a re-analysis at the higher dilution, the lab has added a “D” flag. All the samples are at least 1.5 times the MRL of the blanks indicating a general overall lower volume or dilution. Sample SVEF has MRLs 15 times higher than the baseline (method blank), SVEA at 4 times, EFF at 2.4 times and SVEG and DUP at 7.5 times.

B. Quantitation was checked to determine the accuracy of calculations for representative compounds in each internal standard set

Yes___ No ____ NA___X__

OVERALL ASSESSMENT

Data are considered to be usable for project purposes after consideration of qualifiers or comments. Points of significance are summarized below:

Attachment 10-2

Note an extended list of volatile compounds was reported. Level III data packages were submitted and Level III validation was performed for holding times, chain of custody, calibrations and QC.

Calibration

Minimum response factors are not defined by the method, but met validation guidance. There were 3 calibrations as there were 2 days of analysis for dilutions.

Laboratory Duplicate

The laboratory duplicates was sample FSVE-SVEC-2Q11-NS. RPDs were reported above the limit, but these were low level results that met the 2 x PQL criteria.

Method Blanks

Methylene chloride was reported in all 3 blanks

4/26/11 at 0.041 ppbv; 4/28/11 at 0.046 ppbv; 4/29 at 0.042 ppbv.

The samples have been diluted for most reported results. The blank is multiplied by a factor determined from the reported MRL for each sample/compound. See the EDD and table at the end of the report for the final blank value as data are qualified UMB#, where # is the blank corrected value. All qualifiers are from the 4/26/11 analysis. Only data that are less than 10 x the blank (corrected for dilution) are qualified.

Field Duplicates

One field duplicate pair is reported: FSVE-SVEG-2Q11: -NS and DUP. These are regularly sampled locations and the precision is built in as the sites are sampled routinely. Qualifiers of JFD#, where # is the RPD (or the difference for low level values) are added for field duplicate differences. When results are > 5x the reporting limit, a 35% RPD is used to identify potential deviations. When results are < 5x the reporting limit, an absolute difference between the results that is < 2x PQL is considered to be acceptable reproducibility. (The laboratory uses MRL not PQL for these reports). Note that the times of collection indicate that these samples are not collected at the same time, but are sequential samples. An RPD of 50% is recommended for a precision limit for these samples. The QAPP does not define air QC.

Parent		DUP	RPD	Qualifier
Cis-1,2-dichlorothene	32	59 ug/m3	59	JFD59
Chloroform	1000	1800 ug/m3	57	JFD57
Carbon tetrachloride	190	360 ug/m3	62	JFD62
Trichloroethene	580 (not diluted)	960 ug/m3 (diluted)	49.3	JFD49
Tetrachloroethane	80	150 ug/m3	61	JFD61
1,1,2,2-tetrachloroethane	560	910 ug/m3	48	JFD48

Reporting Limits

It is noted that there are high dilutions for tetrachloroethane; 1,1,2,2-tetrachloroethane, trichloroethene and chloroform and some other compounds. No dilution factors are noted on the results forms but dilutions have been estimated from the MDLs. For compounds that are reported from a re-analysis at

Attachment 10-2

the higher dilution, the lab has added a “D” flag. All the samples are at least 1.5 times the MRL of the blanks indicating a general overall lower volume or dilution. Sample SVEF has MRLs 15 times higher than the baseline (method blank), SVEA at 4 times, EFF at 2.4 times and SVEG and DUP at 7.5 times.

TABLE OF QUALIFIED DATA

Lab ID	Client ID	Compound	Result (ppbv)	Qualifier
P1101503-001	FSVE-SVEA-2Q11-NS	Methylene Chloride	0.75	BMB.17
P1101503-002	FSVE-SVEB-2Q11-NS	Methylene Chloride	0.1	BMB.063
P1101503-003	FSVE-SVEC-2Q11-NS	Methylene Chloride	0.6	BMB.062
P1101503-004	FSVE-SVED-2Q11-NS	Methylene Chloride	0.095	BMB.062
P1101503-005	FSVE-SVEE-2Q11-NS	Methylene Chloride	0.084	BMB.063
P1101503-006	FSVE-SVEF-2Q11-NS	Methylene Chloride	3.4	BMB.61
P1101503-007	FSVE-SVEG-2Q11-NS	Methylene Chloride	2.4	BMB.31
P1101503-003DUP	FSVE-SVEC-2Q11-NS	Methylene Chloride	0.576	BMB.06
P1101503-007	FSVE-SVEG-2Q11-NS	cis-1,2-Dichloroethene	8	JFD59
P1101503-007	FSVE-SVEG-2Q11-NS	Chloroform	210	JFD57
P1101503-007	FSVE-SVEG-2Q11-NS	Carbon Tetrachloride	30	JFD62
P1101503-007	FSVE-SVEG-2Q11-NS	Trichloroethene	110	JFD49
P1101503-007	FSVE-SVEG-2Q11-NS	Tetrachloroethene	12	JFD61
P1101503-007	FSVE-SVEG-2Q11-NS	1,1,2,2-Tetrachloroethane	81	JFD48
P1101503-009	FSVE-SVEG-2Q11-DUP	cis-1,2-Dichloroethene	15	JFD59
P1101503-009	FSVE-SVEG-2Q11-DUP	Chloroform	370	JFD57
P1101503-009	FSVE-SVEG-2Q11-DUP	Carbon Tetrachloride	58	JFD62
P1101503-009	FSVE-SVEG-2Q11-DUP	Trichloroethene	180	JFD49
P1101503-009	FSVE-SVEG-2Q11-DUP	Tetrachloroethene	22	JFD61
P1101503-009	FSVE-SVEG-2Q11-DUP	1,1,2,2-Tetrachloroethane	130	JFD48

Attachment 10-3

DSA Proprietary List of Data Validation Qualifiers

General to all:

JD# duplicate precision, # = value of the Relative Percent Difference (RPD)

JH# holding time exceeded, # = number of days (hours for some wet chem. analytes)

JL# laboratory control sample recovery, # = value of the percent recovery of the LCS

JMS# matrix spike recovery, # = value of the percent recovery of the spike

JT# temperature exceedence, # = temperature in degree C. exceeding holding time

R_# rejected data for associated reason noted in this list or below (R replaces 'J')

UB# blank contamination for the following contaminant sources, the qualifier can be expanded:

UEB equipment blank

UFB field blank

UPB laboratory preparation blank

Organic Data:

JC# calibration accuracy, # = a) a whole number for initial and continuing calibration % RSD or %D, b) a decimal number for response factors, or c) 0.9xx for linear curve

JCCAL or JICAL– denotes continuing or initial calibration if that level of specificity is required on the project

JI# internal standard recovery, # = value of the percent recovery of the internal standard for the specific sample

JN tentatively identified compound

JP# second column confirmation when 2 column difference > 25%, # = RPD of 2 results (for low level values, < 5 x RL, just JP, no number)

JS# surrogate recovery, # = percent recovery of surrogate spike, can be further specified as JSUR#

JQ identification issue, usually poor spectra or interferences

UB# blank contamination, # = highest concentration of method blank affecting data

UTB# trip blank contamination, # = value of TB compound (x dilution)

Attachment 10-4

DIANE SHORT & ASSOCIATES, INC.

PROPRIETARY DATA VALIDATION USABILITY SUMMARY

GENERAL TO ALL ANALYSES

Data are qualified referencing the USEPA Contract Laboratory Program (CLP) data validation guidance with the usability modifications defined by the Data Validator. Data validation qualification is noted by a “J” or “R” qualifier next to the reported data value. The “J” indicates that one or more of the method quality control (QC) limits have been violated and the data may be estimated. The “R” qualifier indicates that the data are considered to be rejected due to significant deviations from the acceptance limits. In order for the qualifiers to be useful in determining the effect of the violation on the data, codes for the violation(s) and a numeric value of the violation are appended to the qualifier.

The USEPA CLP laboratories ‘flag’ data on the Form I in the “Q” field. These ‘flags’ are not to be confused with the data validation qualifiers. ‘Flags’ are notations of laboratory procedures and/or QC alerts and are not necessarily indicative of data qualification. They are not to be used as qualifiers of data. The only code used by the laboratory that must be transferred over with lab data is the “U”, undetected, code indicating that the value reported is the project reporting limit and that the analyte was not detected in the sample above the method detection limit.

Holding Times or sample integrity

JH#, where # is the number of days exceeding the holding time. Some wet chemistry methods have holding times in hours which will be noted in the report. Data could possibly be biased low as the number of days (hours) exceeds the allowed holding time due to loss of the compound.

JT#, where # is the degrees in temperature over the method temperature limit. Data could be biased low due to analyte degradation or other metabolic conversion.

JP, denotes a preservation issue, usually improper pH for inorganics. If there is a preservation issue for organics, a new qualifier may need to be defined in order to avoid confusing it with the 2 column JP qualifier.

Matrix Spike Accuracy

JMS#, where # is the value of the percent recovery of the matrix spike. The QC limit is usually defined by the laboratory using historic control charts for the particular matrix of the sample. For inorganic data a set limit of 75-125% is often used. Given that a matrix spike is also a duplicate sample, the limit for soil samples is extended for some projects. Data whose percent recoveries are less than the established limit could possibly be biased low with respect to the extent of the recovery. Data whose percent recoveries are greater than the limit could possibly be biased high with respect to the extent of the recovery. Undetected data are not qualified for high spike recoveries. For organics, only the parent sample is qualified. For inorganics, the entire set associated with the QC sample is qualified. The %R can indicate:

- the accuracy of the laboratory analytical procedures and instrumentation,
- the geochemical interaction of the specific analyte with the sample matrix, or

Attachment 10-4

- the homogeneity or reproducibility of the sample aliquot.

Laboratory Control Sample (LCS) Accuracy

JL#, where # is the value of the percent recovery of the LCS from the true value or established laboratory limits. The LCS is a measure of laboratory method accuracy. All data associated with the LCS are qualified. If the solid LCS is outside of the defined range, the reviewer calculates a percent recovery from the mean of the range and reports that recovery. Undetected data are not qualified for high LCS recoveries. Organic data may only be qualified for the LCS when the MS/MSD is also out of control for the same compounds. The percent recovery of each analyte from the LCS standard can indicate:

- the percent efficiency of the sample digestion procedure in the laboratory, or accuracy of the analytical method and instrumentation.

ORGANIC ANALYSES

BLANK CONTAMINATION

UB#, where # is the value of the highest blank affecting the data. These data are considered to be fully usable as undetected values. The reported value is considered to be due to contamination from preparation, general laboratory contamination or solvents, or from field contamination. The extent of the contamination is reflected in the value of the blank. Any positive value reported above the MDL is considered to be contamination. When the blank value has not been reported and the determination has been made from a direct comparison of the raw data blank to the raw data sample, no number follows the qualifier. For the common laboratory contaminants (e.g., methylene chloride, acetone, and phthalates), only values less than 10 times the blank value are qualified. For other contaminants, values less than 5 times the blank value are qualified. If there are field blanks, the qualifier may be modified to UMB for method blank contamination and UFB for field blanks or UTB for trip blanks.

MATRIX SPIKE DUPLICATE PRECISION

JD#, where # is the value of the relative percent difference (RPD) between the %Rs of the spikes. The RPD is an absolute number and a high or low bias cannot be determined. The larger the number is over the limit, the greater the difference between the two reported sample values. This may indicate the non-homogeneity of the sample matrix or inadequate sample preparation. Data should be used knowing that there is a range of values around the reported value. Only the parent sample is qualified, and usually only if there is also a spike out of control.

SURROGATE SPIKE ACCURACY

JS#, where # is the surrogate spike recovery. See matrix spike as application is the same.

Calibration Accuracy

JC#, where # is a whole number for initial and continuing calibration curve data or a decimal number (0.0xx) for response factors or correlation coefficient (.9xx). JICAL may also be used for initial

Attachment 10-4

calibration and JCCAL for continuing calibration if the distinction is determined to add to the usability of the data.

For initial calibration, the number indicates the deviation from the relative response factor per the relative standard deviation (RSD). The RSD limit is usually 20-30 percent. For continuing calibration, # is the percent difference (%D) of the continuing calibration standard from the initial calibration average response factor. The limit is usually from 15-25 percent. As the value increases, the range of the quantitation could possibly increase, therefore increasing the estimation of the reported value. Undetected data are not qualified for these calibration violations if the response factors indicate that the compound can be detected if present.

When # is a decimal value, usually less than 0.05, it is the value of the average initial response factors or daily continuing calibration response factor. These data could be false non-detects or biased low due to the lack of sensitivity of the method for the qualified compound.

When # is a decimal value, usually less than 0.995, the value is the correlation coefficient of the linear curve used for initial calibration. As the value decreases, the range of the quantitation could possibly increase, therefore increasing the estimation of the reported value. Undetected data are not qualified for these calibration violations if the response factors indicate that the compound can be detected if present.

Internal Standards

JI#, where # is the value of the percent recovery of the internal standard (IS) area counts for the specific sample. The %R is calculated from the 12-hour average. Values greater than 100 percent indicate quantitation could be biased high. Values less than 50 percent indicate quantitation could be biased low. The raw data should be evaluated, however, to determine if there is an overall suppression of the results. Evaluation of the surrogate and matrix spike recoveries can sometimes give an indication of the impact of the internal standard on the data calculation. Data associated with the given IS are qualified. If there are several IS compounds out of limits and there is a trend to the recoveries, all data will be qualified per the average recovery.

LINEAR RANGE

JE indicates that the value exceeded the linear range of the instrument. A numeric bias cannot be determined, but the bias would be low. These compounds are routinely re-analyzed at a dilution and that value should be used for project decisions. For all other compounds, the lowest dilution should be reported.

Tentatively Identified Compounds (TICs)

JN. The laboratory flags TICs that are not on the method list of standard compounds, but which are reported from electronically stored library spectra. Identification may be accurate, and quantitation is estimated due to lack of daily standard. The JN may also be applied when second column confirmation has not been performed for identified compounds.

Attachment 10-4

Second Column Confirmation

JP indicates that the reported number has a percent difference greater than 25 percent from the second column (quantitation column or confirmation column). When the difference between the two values is greater than 25 percent, it is possible that the higher of the two values was due to interferences. The # after the 'P' is the value of the % difference between the values. Most laboratories report the lower of the two column results in their analytical report. However, data usability reviews for those compounds with greater than 25% RPD will be completed and the more accurate of the two column results will be reported. If no distinction can be made between the two results, the higher of the two will be included in the final report.

DDT/Endrin Breakdown

JX#, where # is the value of the DDT/endrin breakdown. When this number is >20%, it is possible that DDT is being broken down due to column or instrument problems and the data could be biased low due to loss of the compound.

IDENTIFICATION ISSUES

JQ is usually used to denote that an identification is questioned due to poor spectra (missing masses, or mass ratios that do not match reference) or chromatographic interferences.

Attachment 10-5

Final Data Qualification and Usability Report

Project:	Defense Depot Memphis, TN (DDMT) Main Installation Long Term Monitoring
Sampling Event:	LA-26
Project / Task Number:	10021503-228-235772-005
Sample Data Package(s):	L17100514, L17100636, L17100637, L17100710, L17100839
Data Validation Performed by:	Diane Short & Associates (DSA)
Final Data Qualification and Usability Report Prepared by:	Lynn K. Lutz, HDR Inc.

Data Validation Report Review and Comments
The validation report was acceptable except as noted below for final qualifiers.

The validator, in discussing relative response factors, noted that "[t]he laboratory has been tasked with providing to the client verification that the 0.01 RF is valid. If the spectral verification is available, data are not qualified for response factors greater than 0.01 and less than 0.05 for 'poor responders.' The Project Chemist reviewed the area counts for the lowest standard included in the initial calibration for the low responders and found the areas to be adequate, and no qualifiers were applied.

The validator did not qualify a number of analytes based on low or high responses in the CCV; the PC evaluated the %D values and qualified associated results as estimated "J" for high or low results or non-detect estimated "UJ" for non-detect low results, as both the project QAPP and the method require CCV results be within 20%D:

- Chloromethane - workgroup # WG633719: %D = -20.2% (UJ)
- Dichlorodifluoromethane - workgroup # WG634168: %D = +22.8% (none, all ND)
- Dichlorodifluoromethane - workgroup # WG634400: %D = +25.9% (none, all ND)
- 2,2-Dichloropropane - workgroup # WG634436: %D = +32.1% (none, all ND)
- Acetone - workgroup # WG634436: %D = -21.6% (1 UJ, 4 J-)
- Dichlorodifluoromethane - workgroup # WG634436: %D = +23.1% (none, all ND)
- Dichlorodifluoromethane - workgroup # WG634505: %D = +21.4% (none, all ND)
- Hexachlorobutadiene - workgroup # WG634606: %D = +20.4% (none, all ND)
- 2,2-Dichloropropane - workgroup # WG634645: %D = +55.1% (none, all ND)
- Dichlorodifluoromethane - workgroup # WG634775: %D = +26.8% (none, all ND)
- 2-Chlorotoluene - workgroup # WG635230: %D = -20.7% (1 UJ)

The validator noted that all three vials of samples DUP6-LA-26 and LTM-TB3-LA-26 had headspace, and were "considered to be possibly biased low or contaminated." This is actually only true for the trip blank. For the field duplicate, the HDR chemist instructed the lab to use one of the vials from the parent sample, MW-100B-LA-26, and analyze it as the FD. The parent sample vial did not have headspace and no qualification was required for the FD.

Attachment 10-5

Final Data Qualifiers
The final qualifiers were UJ or J- where DSA had not qualified for calibration issues as noted above, for these analytes:
Chloromethane – 10 samples (UJ)
Acetone – 4 samples (UJ)
Acetone – 1 sample (J-)
2-Chlorotoluene - 1 sample (UJ)

The final qualifier for all analytes in sample DUP6-LA-26 was “no qualifier” where DSA had qualified as JP to indicate integrity issues due to sample vial headspace, as noted in the section above.

The final qualifier for all analytes in sample LTM-TB3-LA-26 was UJ where DSA had qualified as JP to indicate integrity issues due to sample vial headspace, as noted in the section above.

The final qualifier for acetone in seven samples was 10.0 U where DSA had qualified as BTB2.87 for acetone in the trip blank. Sample results were less than the reporting limit (RL) and qualified as non-detect at the reporting limit.

The final qualifier for acetone in one sample was 10.0 U where DSA had not qualified for acetone in the trip blank. The sample result was less than the reporting limit (RL) and qualified as non-detect at the RL.

The final qualifier for acetone in four samples was “no qualification” where DSA had qualified as BTB2.87 for acetone in the trip blank. Sample results were greater than two times the blank concentration.

The final qualifier was J where the lab had qualified as F.

Data Usability
There were no rejected sample results. All results are usable as qualified.

(signature) 20 November 2017

Lynn K. Lutz, HDR Inc.

STANDARD OPERATING PROCEDURE 11 – FIELD SAMPLING TECHNICAL SYSTEMS AUDIT

Lead Organization: Department of the Army (DA)
Preparing Organization: HDR
SOP Approved by: Field Team Leader: Eric North
Project Chemist: Lynn Lutz
Project Manager: Tom Holmes

1 Purpose and Summary

This Standard Operating Procedure (SOP) provides guidance and a checklist for the performance of a field sampling technical systems audit (field audit). Activities to be audited include field sample collection, sample handling, documentation, and field analysis of samples collected at Defense Depot Memphis, Tennessee (DDMT). Review of this SOP and other applicable field and sampling SOPs is mandatory prior to the start of each field audit.

2 Health and Safety

Each individual is required to have read and understood the Health and Safety Plan (HASP) for the specific project activity. Upon arrival at the site, each person shall sign the acknowledgement sheet confirming their review of the HASP. Personal protective equipment and other provisions for site safety requirements are discussed in the HASP. Any questions should be addressed to the Field Team Leader (FTL).

3 Personnel Qualifications and Responsibilities

The field audit will be conducted by the project quality assurance (QA) Officer or a designated representative familiar with field sampling techniques and requirements, field analyses, and sample handling and documentation requirements.

4 Equipment and Supplies

The auditor will use the field audit checklist (Attachment 11-1 to this SOP) and a pen with blue or black waterproof ink to record field activities and sample handling at the field office. The audit report will be prepared in Microsoft Word.

5 Procedure

The auditor will first read and become familiar with the applicable SOPs, specifically those dealing with sample collection, documentation, handling, and analysis of field parameters.

The auditor will then accompany the sampling team to the field location and observe the activities, comparing activities performed against the SOP requirements, and note discrepancies.

The auditor will use the checklist (Attachment 11-1) to ensure that all necessary activities are performed by the field sampling team and observed by the auditor. If the auditor notes discrepancies, he or she will make notations on the checklist and discuss the issues with the FTL at the end of the audit.

If the auditor notes serious discrepancies that would adversely impact the data quality, these issues will be immediately brought to the attention of the FTL and the project manager (PM). Discussion may result in changes to be implemented immediately in the field, and these changes along with the date and time of implementation will be noted on the audit checklist. The field sampling team will also record the changes along with the date and time of change in the field notes. Such changes would likely result in changes to the applicable SOP(s).

5.1 Using the Field Audit Checklist

The sections below describe the general areas of evaluation as listed on the checklist (Attachment 11-1).

5.1.1 Part 1: Sampling and Analysis Plan

Part 1 asks questions to ensure the existence and adequacy of a Sampling Analysis Plan (SAP) and relevant SOPs. The auditor will review these documents before going into the field.

5.1.2 Part 2: Organization, Management& Personnel

Part 2 asks questions to determine whether sampling staff have been adequately and appropriately trained for the field activities. The auditor will review these records before going into the field.

5.1.3 Part 3: Equipment

Part 3 asks questions to determine whether the sampling equipment, including sample containers, are the appropriate equipment, are ready to use, and have a routine maintenance plan to ensure performance. The auditor will review these records and will also interview the FTL before going into the field.

5.1.4 Part 4: Sampling Event Information

Part 4 asks questions to ensure that the sampling event and sample identifications have been adequately planned and that samples are clearly identified and handled properly. The auditor will review these records before and after going into the field for sample collection.

5.1.5 Part 5: Sample Management

Part 5 asks questions to ensure that samples are clearly identified and handled properly. The auditor will observe these activities in the field during sample collection, storage, and shipment.

5.1.6 Part 6: Field Analyses

Part 6 asks questions to ensure field analyses are performed according to the SOPs and recorded accurately and adequately. The auditor will make these observations in the field.

5.2 Field Sampling Technical Systems Audit Report

The auditor will prepare a Field Sampling Technical Systems Audit Report for delivery to the PM and the FTL within one month of the completion of the on-site audit. As noted in Section 5.0, any changes to procedures that were made in the field will be documented in the report, and these changes must also be added to the relevant SOP, if applicable. The SOP changes will be made prior to the next field sampling event, and the FTL will be responsible for ensuring that all field sampling personnel have read the revised SOP and are aware of and familiar with the changes.

6 Data and Records Management

The field auditor will complete all sections of the audit checklist, scan the checklist into a PDF file, and save the file to the appropriate location on the HDR network drive.

The field auditor will prepare a summary report of audit results and save the file to the appropriate location on the HDR network drive.

7 Quality Control and Quality Assurance

All work will be performed in accordance with the Quality Assurance Project Plan (QAPP), the HASP, the specific work plan, the specific sampling plan details (SPD) and applicable SOPs. No erasures or mark outs will be made on the checklist or other field notes. A single line will be used to strike out errors and will be annotated with the initials and date of the editor.

8 References

Field audit checklist: Attachment 11-1

Attachment 11-1

Checklist for Field Sampling Audit

Sampling Organization	Date of Evaluation	Name and Affiliation of Evaluator
Field Sampling Event		

Item to be Evaluated	Y	N	N/A	Comments
Part 1: Sampling and Analysis Plan (SAP)				
1.1 General Information				
Is there a sampling plan?				
Are the procedures for the transportation, handling, protection, storage, retention and/or disposal of samples, including all provisions necessary to protect the integrity of the sample?				
Is there a documented system for uniquely identifying all samples and subsamples, to ensure that there can be no confusion regarding the identity of such samples at any time?				
Does the sampling process address the factors to be controlled to ensure the validity of the environmental test and calibration results?				
Is there a process for documenting corrective actions taken in the field?				
1.2 Standard Operating Procedures (SOPs)				
Are there SOPs for field activities available at the location where sampling is taking place and are they accessible to all sample collectors?				
Are the SOPs official documents (e.g., signed and dated)?				
Have the SOPs been approved for the project?				
Part 2: Organization, Management and Personnel (not checked on site)				
Are the sampling personnel's qualifications and/or training certifications adequate for the tasks performed?				
Are the names of all sampling personnel recorded?				
Do sampling personnel meet minimum qualifications specified in the contract?				
If the sampling organization is supporting a larger organization, are there any arrangements that could cause a conflict of interest?				

Attachment 11-1

Item to be Evaluated	Y	N	N/A	Comments
Does the sampling agency have policies or procedures to ensure the client confidentiality and propriety rights?				
Are staff training records maintained and up to date?				
Part 3: Equipment				
3.1 General Equipment Information				
Is the type of equipment sufficient for the sampling project?				
Is the quantity of equipment sufficient for the sampling project?				
Is the following information recorded for each piece of equipment that will be used for the sampling project?				
Maintenance and repair procedures for equipment of instrument?				
Routine cleaning procedures?				
Filling solution replacement for the probes?				
Parts replacement for instruments or probes?				
Calendar date for each procedure performed?				
Names of personnel performing maintenance and repair tasks?				
Description of malfunctions associated with any maintenance and repair?				
Vendor service records?				
Inclusive rental dates, types and unique descriptions of rental equipment?				
Is the equipment storage procedure acceptable?				
Is there an existing QC check on sampling equipment?				
3.2 Field Instrument Calibration				
Is information about all calibration standards and reagents used for field testing linked to the calibration information associated with the field testing measurements for the project?				
Are field instruments properly calibrated and calibrations recorded in a bound field log book?				
For each instrument unit used for the sampling project, is the following information recorded for all calibrations?				
Unique identification (designation code) for the instrument?				
Date and time of each calibration and calibration verification?				
Instrument reading or result (display value) for all calibration verifications, with appropriate measurement units?				
Names of analyst performing each calibration or verification?				

Attachment 11-1

Item to be Evaluated	Y	N	N/A	Comments
Designation of each calibration standard used linked to the associated records for the calibration standard?				
The acceptance criteria for each calibration and verification standard used?				
The assay specifications or acceptance criteria for any QC standard or sample used to independently verify the calibration of the standard?				
Are all corrective actions performed on the instrument prior to attempting reverification or recalibration of the instrument linked to the records required for preventive maintenance?				
Does the field instrumentation documentation include the standard concentrations used for calibration?				
Did all field-testing equipment and instrumentation brought to the field appear to function properly?				
Are the manufacturer's suggested maintenance activities and any repairs performed and documented for all applicable equipment and instruments?				
3.3 Containers				
Are sample containers well organized, properly prepared, protected from contamination and ready to use?				
Are proper sample containers and sizes used for each type of sample?				
Are certificates of analysis for pre-cleaned bottles maintained on file?				
Are all containers and container caps free of cracks, chips, discolorations and other features that might affect the integrity of the collected samples?				
3.4 Sampling Equipment				
Is the appropriate equipment used for the sampling project? Check all relevant equipment used for sample collection, handling, storage and transport.				
Is equipment constructed of materials appropriate for the analytes of interest?				
Is equipment brought to the field pre-cleaned?				
For equipment decontaminated on site in the field, are the date and time of the cleaning procedure recorded in the field records or referenced in an internal SOP?				
Are cleaning steps in all procedures used for decontamination documented either by description or reference to an SOP?				
Are there current maintenance records for all field equipment?				
Part 4: Sampling Event Information				
For all samples, is the following information recorded and transmitted to the client?				
Site name and address?				
Date and time of sample collection?				

Attachment 11-1

Item to be Evaluated	Y	N	N/A	Comments
Name of sampler responsible for sample transmittal?				
Unique field identification code for each sample container or group of containers?				
Total number of samples collected?				
Required analyses for each sample container or group of containers?				
Sample preservation used for each container or group of containers?				
Comments about samples, sample sources or other relevant field conditions?				
Identification of common carrier used to transport the samples, when applicable?				
Are shipping invoices and related records from common carriers archived with the field records, when applicable?				
Are sampling locations adequately documented in a bound field log book using indelible ink?				
Are photos taken and is a photo log maintained?				
4.1 Field QC				
Are trip blanks and/or field blanks collected as specified in the approved sampling plan?				
Are field blanks collected after equipment is decontaminated in the field?				
Are field blanks collected if no equipment was cleaned by the sampling organization?				
Are additional samples for matrix spike/matrix spike duplicate analyses collected?				
Are all QC samples collected in the same manner as the routine field samples?				
Part 5: Sample Management				
5.1 Collection				
Are the samples taken from a representative point of the source?				
Are the samples homogeneous where appropriate?				
When possible, does sampling originate from the suspected least contaminated location first and progress to the suspected most contaminated location?				
Are samples for different analyte groups collected in the appropriate order?				
Are samples co9llected for all required analyses?				
Are samples to be tested for dissolved metals filtered prior to preservation?				
Is every effort made to prevent cross-contamination of samples?				
Are gloves worn by all samplers handling purging equipment, sampling equipment, measurement equipment and sample containers?				

Attachment 11-1

Item to be Evaluated	Y	N	N/A	Comments
Are new, clean, unpowered gloves used for each glove change?				
Is care taken to avoid contact with sample and sample container interiors?				
Are VOC sample containers protected from any fuel sources and fuel-powered equipment?				
Do VOC sample containers remain capped until just prior to sample collection and do they remain capped after sample collection?				
Where applicable, are samples collected for measurement of dissolved components, filtered, preserved with acid and placed on ice within 15 minutes of collection?				
5.2 Collection Devices				
Is sample collected using an intermediate collection device?				
Are intermediate collection devices rinsed with ample amounts of site water prior to collecting the sample?				
Is rinse water from intermediate collection devices discarded away from and downstream of the sampling location?				
Is the use of intermediate collection devices avoided when sampling for VOCs, oil and grease, or microbiologicals, where practical?				
Are any intermediate collection devices constructed of material appropriate for the analytes to be measured?				
Are sample containers submerged neck first, inverted into the oncoming direction of flow where applicable, slowly filled, and returned to the surface for preservation, if applicable?				
5.3 Sample Labeling				
Is each sample container or group of containers tagged or labeled with a unique field identification code that distinguishes the sample from all other samples?				
Are the unique identification codes for samples recorded in a manner that links the codes to all other field records associated with the samples?				
Is waterproof indelible ink used to label containers?				
5.4 Storage				
Are samples for different parameters segregated during storage?				
Are samples stored on ice?				
Is the cooler clearly labeled?				
Are samples properly preserved (if applicable)?				
5.5 Preservation				
Do all sample preservation techniques conform to SOP or method requirements?				
Are all samples properly preserved within 15 minutes, as applicable?				

Attachment 11-1

Item to be Evaluated	Y	N	N/A	Comments
Are the preparation and dispensing of preservatives documented and traceable?				
Is preservation information and verification recorded for each sample, as applicable?				
Are samples placed on ice immediately after collection, if applicable?				
If the samples are found to contain cyanide, are they NOT acidified?				
5.6 Delivery				
Are samples protected during delivery to prevent breakage?				
Are samples shipped in a timely manner?				
5.7 Disposal				
Are wastes generated as a result of the sampling project containerized and stored for proper disposal according to applicable local, state and federal regulations?				
Are all sampling-derived waste containers properly labeled?				
Is all sampling-derived waste properly disposed of?				
5.8 Documentation				
Is waterproof ink used for all paper documentation?				
Are the date and time of sample collection recorded for all samples?				
Are the ambient field conditions recorded for all samples?				
Is a specific description of each sampling location (source) recorded?				
Does the chain of custody/traffic report include the following: date, time, sample numbers, sampler names, shipping method, number of samples, matrix, and comments?				
Is preservation information recorded on the chain of custody/traffic report?				
Are copies of traffic reports or COCs sent to the proper recipients?				
Are deviations, additions, or exclusions from the documented sampling procedure recorded in detail with the associated sampling information?				
Are these deviations included in all documents containing environmental test and/or calibration results?				
Are these deviations communicated to the appropriate personnel?				
Are all errors in documentation (if applicable) corrected and initialed without obliteration?				
5.9 Field Reagents				
Are the concentration (or other assay value), the vendor catalog number and the description of the standard or reagent recorded for all preformulated solutions, neat liquids, powders, and blank water?				
Are certifications of assay, grade and other vendor specifications for all standards and reagents retained and recorded for the standards and reagents?				

Attachment 11-1

Item to be Evaluated	Y	N	N/A	Comments
Are the lot numbers and inclusive dates of use recorded toe all reagents, detergents, solvents, and other chemical used for decontamination and preservation of samples?				
Are the expiration dates for all calibration standards and reagents recorded?				
Are expired standards and reagents verified prior to use during sample collection?				
Are all steps used for preparation of standards or reagents in-house documented either by description or reference to an SOP?				
Part 6: Field Analyses				
6.1 General Field Test Information				
Are all field measurement tests and related data recorded and linked to the project, the date and the sample source?				
Are all field measurements recorded with the appropriate units, the value of the test result, the parameter measured, the name of the analyst performing the test, the time of the measurement and the unique identification for the test instrument used?				
6.2 pH				
Are all samples requiring pH adjustment tested for proper pH preservation?				
Is at least one sample per analyte group requiring pH adjustment tested for proper preservation during repeat sampling?				
Is pH paper or a pH electrode inserted into sample containers?				
Do the pH meter and electrode system meet SOP specifications for accuracy, reproducibility and design?				
Are all measurements corrected for temperature (manual or automatic)?				
Is a pH 7 buffer used as the first calibration standard?				
For pH, do all calibration verifications meet the acceptance criteria?				
If the calibration and/or calibration verifications did not meet the acceptance criteria, is the calibration or verification identified as a failure and was this documented in the calibration log?				
Are all sample measurements associated with acceptable calibration verifications?				
Is the pH meter system checked on a weekly basis to ensure >90% theoretical electrode slope?				
Are the field instrument probes rinsed with deionized or distilled water between standard solutions and between sample measurements?				
Are instrument pH readings allowed to stabilize before pH values are recorded?				
6.3 Filtration				
Are samples collected for analysis of dissolved components filtered within 15 minutes of collection and before addition of chemical preservatives where appropriate?				

Attachment 11-1

Item to be Evaluated	Y	N	N/A	Comments
Unless otherwise specified, are applicable samples filtered using a 0.45-µm pore size?				
6.4 Temperature				
Do the temperature measurement devices meet SOP and/or sampling event specifications for design and measurement resolution?				
Are all sample measurements associated with calibration verifications of the temperature measurement device at a minimum of two temperatures using a NIST-traceable thermometer?				
If the calibration and/or calibration verifications did not meet the acceptance criteria, is the calibration or verification identified as a failure and was this documented in the calibration log?				
Are all temperature measurements chronologically associated with acceptable calibration verifications?				
Are the temperature readings allowed to stabilize before measurement values were recorded?				
6.5 Conductivity				
Do the specific conductance meter and electrode system meet the SOP and/or sampling event specifications for accuracy and reproducibility?				
Do all calibration verifications meet the acceptance criteria?				
If the calibration and/or calibration verifications did not meet the acceptance criteria, is the calibration or verification identified as a failure and was this documented in the calibration log?				
Are all conductivity measurements chronologically associated with acceptable calibration verifications?				
Are all conductivity measurements corrected for temperature (manual or automatic)?				
Is the instrument allowed to stabilize before e measurement values are recorded?				
6.6 Turbidity				
Does the turbidimeter meet the SOP and/or sampling event specifications for accuracy and reproducibility?				
Are all sample measurements associated with acceptable calibration verifications?				
If the calibration and/or calibration verifications did not meet the acceptance criteria, is the calibration or verification identified as a failure and was this documented in the calibration log?				
Are the sample cells (optical cuvettes) inspected for scratches and discarded or coated with a silicone oil mask, as necessary?				
Are the sample cells (optical cuvettes) optically matched for calibrations and sample measurements?				

Attachment 11-1

Item to be Evaluated	Y	N	N/A	Comments
Are the sample cells (optical cuvettes) cleaned with detergent and deionized or distilled water between standard solutions and between sample measurements, as applicable?				
Are the sample cells (optical cuvettes) rinsed with sample prior to filling with sample for measurement?				
Is the exterior of the sample cell (optical cuvette) kept free of fingerprints and dried with a lint-free wipe prior to insertion in the turbidimeter?				
6.7 Dissolved Oxygen				
Do the dissolved oxygen meter and electrode system meet the SOP and/or sampling event specifications for accuracy and reproducibility?				
Are all sample measurements associated with acceptable calibration verifications?				
If the calibration and/or calibration verifications did not meet the acceptance criteria, is the calibration or verification identified as a failure and was this documented in the calibration log?				
Are all measurements corrected for temperature (manual or automatic)?				
Are all measurements corrected for salinity, where applicable (manual or automatic)?				
Is the salinity (conductivity) sensor calibration verified?				
Is the dissolved oxygen electrode stored in a water-saturated air environment when not in use?				
Are the dissolved oxygen readings allowed to stabilize before measurement values are recorded?				

Other Comments or Issues:

GEOEXPLORER 6000 SERIES
QUICK START GUIDE

Four simple steps to get started:

This guide provides an overview of the important features and instructions for how to set up and operate your Trimble® GeoExplorer® 6000 series handheld.

1. Insert the battery in the handheld and then charge it fully.
2. Turn on the handheld.
3. Connect the handheld to a PC and install your GNSS field software. If required, transfer files to the handheld.
4. In the field, use the handheld's integrated GNSS receiver with your field software to collect GNSS data.

Additional information:

For further details regarding the features and operation of the handheld, refer to the *GeoExplorer 6000 Series User Guide*, provided on the Microsoft® Getting Started Disc.

To access the latest information, including release notes, visit:

GeoXT handheld:
www.trimble.com/geoxt.shtml

GeoXH handheld:
www.trimble.com/geoxh.shtml

Trimble

Parts of the handheld:

Included accessories:

AC Power Supply & International Adapter Kit	USB Data Cable	Spare Stylus & Lanyard
Carry Pouch	Hand Strap	Screen Protectors (x2)
Software CD	Device ID labels	Battery Pack

Charge the battery first:

⚠ ***WARNING:*** – For safety information, refer to the "Safety" section of the *GeoExplorer 6000 Series User Guide.*

A rechargeable Lithium-ion battery is provided with the GeoExplorer handheld. Charge the battery using the AC Power Adapter and the international adaptor kit. The battery can be charged inside or outside the handheld.

The battery should be charged for at least **five hours** before using it the first time. If the battery has been stored for more than six months, charge it before use.

Installing and removing the battery pack:

To install the battery pack:
Insert the battery pack into the battery opening and then push the battery firmly into the handheld, ensuring that both battery latches click into place fully.

To remove the battery pack:
Pinch the latches together until the latches disengage from the handheld, and then slide the battery out.

Fitting the hand strap:

1. Align the upper hand strap bracket with the two mounting holes and tighten with a screwdriver or coin.
2. Stretch the hand strap to align the bottom hand strap bracket with the mounting hole and tighten the screw with a screwdriver or coin.

Installing screen protectors:

To protect the screen from pressure and abrasive objects, Trimble recommends that you apply one of the anti-reflective screen protectors provided with your GeoExplorer 6000 series handheld to the touch screen.

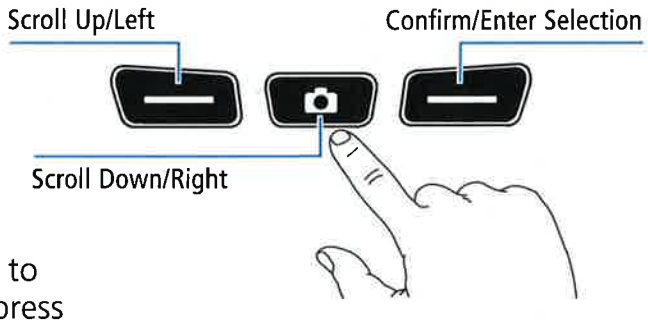
Starting the handheld for the first time:

The first time you turn on your handheld, you must select the language used by the Windows Mobile® operating system and align the touch screen. Press the **Power** key ⏻ to start the handheld.

> ⚠ *CAUTION:* – You can only select the language once. To change the language you must return the handheld to an authorized Trimble service provider.

1. Select the language you want to install: Press the **Left** application key to scroll up the list, or the **Camera** key to move down the list. Press the **Right** application key to make your selection.

2. In the confirmation screen that appears:
   - To go back and choose a different language:
     Make sure the **<Back** button is highlighted, then press the **Right** application key to go back.
   - To confirm the selected language:



   a. Press the **Left** application key once to highlight the check box, and then press the **Right** application key to confirm the selection.
   b. Use the **Camera** key to scroll the selection highlight to the **Next>** button.
   c. Press the **Right** application key to install the selected language onto the handheld.

> **TIP:** You must use the keypad to select the language, this cannot be performed using the touchscreen.

Calibrate the touch screen:

Once the language is confirmed, the handheld restarts. Follow the on-screen instructions to calibrate the touch screen.

Interacting with the display:

Use the stylus or a finger to operate the touch panel display.

- Tap the screen **once** to open items and select options.
- **Tap and hold** on an item to see a pop-up menu of available actions. Then tap the action you want to perform.
- **Flick or swipe** the screen to scroll through lists and menus.
- To enter text, use the text input panels.

TIP: To enter special characters, tap **[123]** to display a keyboard containing numbers and symbols. To switch back to the main keyboard, tap **[123]** again.

Connecting to an office computer:

To transfer files to the handheld or to install software, you must connect the handheld to a computer. If your computer is running:

Windows® 7 or Windows Vista®	Use Windows Mobile Device Center to manage the connection.
Windows XP or Windows 2000	Use Microsoft ActiveSync® technology to manage the connection.

Install ActiveSync or Windows Mobile Device Center to your office computer. Connect your handheld to a USB port on the office computer using the supplied USB data cable.

TIP: You must install the Windows Mobile Device Center or ActiveSync technology onto the computer **before** you connect the handheld. To download the latest version of Windows Mobile Device Center or ActiveSync technology go to www.microsoft.com/windowsmobile.

Configuring the GNSS receiver connection:

To collect GNSS data, install your preferred field software application onto the GeoExplorer handheld and—if required—configure the software to connect to the internal GNSS receiver. To configure:

Trimble TerraSync™ software	**No configuration or setup is required.** All Trimble Mapping & GIS field software is configured to correctly connect to the internal GNSS receiver out-of-the-box and configure GNSS settings for optimum GNSS data collection productivity and accuracy.
Trimble GPS Controller software	
Trimble GPScorrect™ extension for ArcPad	
ArcPad (if not using GPScorrect)	In ArcPad, tap the GPS button drop-down menu, select GPS preferences, then tap the GPS tab. In the protocol field, select NMEA 0183, then select COM2 from the Port field. Tap OK. To connect to the receiver, tap the GPS button, then tap Yes.
Other NMEA GNSS field applications	Configure the software to connect on COM 2.

TIP - To configure output settings when using NMEA applications use the GPS Controller software, available for download from www.trimble.com/support.html.

Configuring a modem connection:

Some configurations of the GeoExplorer 6000 series include an integrated cellular modem. To operate the modem you must install a SIM card and configure a modem connection.

1. Turn off the handheld. Open the SIM card door and insert the SIM card as illustrated. Close the SIM card door.
2. Turn on the handheld. The SIM card is automatically detected. Follow the instructions to configure connection settings for your SIM card provider.

TIP: Your cellular service provider may require you to use a custom username, password and/or other configuration settings. Contact your service provider and confirm which settings are required when connecting.

Title bar status indicators:

Status indicators are displayed in the title bar at the top of the display.

Wi-Fi Network Detected
Indicates one or more Wi-Fi networks are available

Connectivity Status
Indicates USB and wireless data connectivity status

Cellular Status
Indicates cellular modem status and current network connection type

Battery Status
Display the current level or charging status of the battery

Audio Status
Display the current status of the system speaker

LED status indicators:

Three LEDs on the keypad of the handheld indicate battery charge status, GNSS status, and wireless radio status:

Battery Status

	Battery charging is complete
	Battery is charging
	Battery level is low (< 10% remaining)
	Battery fault

GNSS Receiver Status

	Receiver is on, and GNSS positions are available.
	Receiver is on, but GNSS positions are not available.
	Receiver firmware loading / updating
	GNSS fault

Wireless Status

	A wireless radio is turned on

TIP - Leaving wireless radios activated when they are not in use reduces battery run time. If they are not being used, turn off Bluetooth, Wi-Fi, and the cellular modem using the Wireless Manager. To access the Wireless Manager, Tap **Start > Settings > Connections > Wireless Manager.**

GEOEXPLORER 6000 SERIES

Power menu:

To access the Power Menu, press and hold the **Power** key ⏻ when the handheld is operating.

The Power Menu is a convenient way to:

- View the remaining battery run time
- Access Power and Backlight settings
- Swap batteries without turning off the device
- Reset or turn off the handheld
- Calibrate the touch panel
- Access the Start Menu from applications running in full screen mode.

Troubleshooting:

If the screen on the handheld is blank, try one of the following:

- The backlight is off: tap the screen with the stylus or press a key to turn on the backlight.
- The handheld is off, or has suspended: press the **Power** ⏻ key to turn on the handheld.

If the handheld stops responding to the stylus, or if it does not respond when you press any keypad button, you may need to reset it. To reset your handheld, press and hold the **Power** ⏻ key until the Trimble boot screen appears.

⚠ *CAUTION:* Resetting your handheld may cause unsaved data to be lost. Always try to save your data before resetting your handheld.

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Appendix B
Analytical Standard Operating Procedures

MICROBAC®

STANDARD OPERATING PROCEDURES
ANALYSIS OF VOLATILE ORGANIC ANALYTES
BY METHOD 8260

Issue/Implementation Date: 10 January 2017

Last Review Date: 10 January 2017

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TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 Microbac SOP MSV01 pertains to the determination of volatile organic compounds in solid and liquid matrices using purge and trap GC/MS. This method references USEPA SW846 Methods 8000D (July 2014), 8260B (December 1996), 5030C (May 2003), and 5035A (July 2002); AFCEE QAPP's 1998, 2001, and 2005. SOP MSV01 applies to all volatile mass spectral analyses except where client specific Quality Assurance Project Plan's (QAPP) override this method's quality assurance plan.

1.2 Table 1 contains the target compound list for this method.

1.3 Appendix I contains suggested primary and secondary quantitation ions. Section 11.13 contains information regarding the analysis of wipe samples. Appendix II contains requirements for South Carolina.

1.4 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

BFB	Bromofluorobenzene
BS	Blank Spike
BSD	Blank Spike Duplicate
CCV	Continuing calibration verification
DI water	Deionized water
GC	Gas Chromatography
GC/MS	Gas Chromatograph/Mass Spectrometer
HCl	Hydrochloric acid
ICAL	Initial calibration
ICV	Initial calibration verification
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
LIMS	Laboratory Information System
LOD	Limit of Detection
LOQ	Limit of Quantitation
MB	Method blank
MDL	Method detection limit
MS	Mass Spectrometer
MS	Matrix spike
MSD	Matrix spike duplicate
NCR	Nonconformance report

PFTBA	Perfluorotributylamine
PPE	Personal Protective Equipment
QC	Quality control
RGT	Reagent
RL/LLOQ	Reporting limit/lower limit of quantitation
RT	Retention time
SDS	Safety Data Sheets
SOP	Standard Operating Procedure
STD	Standard
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compounds

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

2.0 SAFTY PRECAUTIONS

2.1 Standard laboratory safety procedures must be followed when working with unknown samples. Gloves must be worn while handling any chemicals, standards, or samples. Other required PPE includes lab coats and safety glasses with sideshields.

2.2 **WARNING:** The following VOC's have been tentatively classified as known or suspected human or mammalian carcinogens:

benzene	chloroform
carbon tetrachloride	vinyl chloride

The toxicity or carcinogenicity of the other reagents and analytes used in this method have not been precisely defined, therefore, each chemical and sample must be treated as a potential health hazard and exposure reduced to the lowest possible level. Procedures involving primary standards and sample preparation shall be performed in a fume hood.

2.3 SDS for each analyte and reagent used within the laboratory are available to all employees. Consult SDSs prior to handling chemicals.

2.4 Thermal Hazards

Several zones on the GC are heated to high temperatures. Care must be exercised when working around these areas to avoid severe burns to the skin.

2.5 Broken Glassware Hazards
All dilutions that require the use of glassware must be made with care to reduce the possibility of cuts from broken glass. All broken or defective glassware must be disposed of in the broken glass container located in the laboratory.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Pre-cleaned 40 mL glass screw-cap VOA vials with Teflon-faced silicone septa must be used for both liquid and solid matrices utilizing methods 5030 and 5035. Soil samples not utilizing Method 5035 must be collected in 125 mL pre-cleaned glass screw cap jars with teflon-lined lids. Soil samples collected via 5035 must also be collected in Encore (or equivalent) containers then transferred to 40 mL VOA vials for analysis. Refer to Microbac SOP PAT01 for additional requirements.

3.2 Water samples preserved with HCl (pH < 2) must be analyzed within 14 days of sample collection. Unpreserved water samples (pH ≥ 2) must be analyzed within 7 days of sample collection. Waste, soil, and sludge samples do not require the addition of preservative but must be stored at ≤ 6° C. Solid samples utilizing Method 5035 require preservation if analysis cannot be performed within 48 hours of collection. Waste, soil, oil, and sludge samples have a holding time of fourteen days from the date of collection. Soil samples collected in wide mouth bottles, are stored at ≤ 6° C. Samples collected via 5035 must be stored at -10° C to -20° C. Concentrated waste, oil, soil, sludge, or any other matrix can be stored in an ambient location segregated from low-level environmental samples.

3.3 Sample hold time is defined as time elapsed from sample collection date and time to sample analysis date and time.

3.4 Samples are stored in assigned locations until expiration of hold times. After hold-time expiration, samples are removed from storage refrigerators and returned to sample archive. Samples requiring internal chain-of-custody are returned to the sample receiving custodian.

3.5 Temperature logs are maintained for all refrigerator and freezer storage units. Refer to Microbac SOP GP-TEMP-SSU for temperature monitoring of sample storage units.

3.6 Sample preservation should be <6°C. Samples exceeding the upper temperature limit are to be flagged CT1.

4.0 METHOD PERFORMANCE

4.1 Table 2 summarizes the performance data for water analysis; Table 3 summarizes performance data for soil/solid waste analysis. These tables include the analyte list, ranges for accuracy and precision, current laboratory MDL, nominal laboratory RLs/LLOQs, true values, and suggested calibration range.

4.2 The laboratory performed an initial assessment of the MDL using the procedures outlined in 40 CFR Part 136. Results are filed electronically at H:\DATA\COMMON\MDL.

4.3 The LOD, or verified MDL, are presented in Tables 2 and 3 were established using verification procedures outlined in Microbac SOP 45.

4.4 The LOQ are the nominal laboratory RLs/LLOQs and were established per Microbac SOP 45. Actual project RLs/LLOQs may be higher. The LLOQ is verified quarterly by analysis of duplicate samples spiked at 0.5-2 times the established LLOQ. The verification is performed per instrument and in both solid and aqueous matrices. The recovery of target analytes should be within the LCS criteria ±20%.

4.5 Precision and accuracy data were derived from an initial demonstration of capability using spiked control samples. The laboratory uses results from LCS to assess precision/accuracy and to annually evaluate the associated control limits.

4.6 AFCEE and other specific QA objectives may be found in the appropriate Statement-of-Work or QAPP.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Samples for volatile organics analyses are susceptible to laboratory contaminants (e.g.: methylene chloride, acetone, n-hexane). To eliminate the

potential for interferences from other areas of the laboratory, the Volatiles Laboratory has an independent air intake system and positive air pressure is maintained in the laboratory.

5.2 Samples preserved with HCl or sodium bisulfate may result in the loss of 2-chloroethylvinylether as a target or spiked analyte due to its reactivity with the preservatives.

5.3 Soil analyses may result in low internal and/or surrogate standard recovery due to the poor purging efficiencies of some matrices. Reanalysis must be performed to confirm matrix interference.

5.4 Carry-over contamination may occur when a sample containing low levels of VOC's is analyzed immediately following a sample containing high levels of VOC's. If this situation occurs during a non-monitored analysis, the sample containing the low concentration VOC's may require reanalysis.

5.5 Samples may become contaminated by diffusion of volatile organics through the septum seal into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling, handling, and analysis steps serves as a check on such contamination.

5.6 Storage blanks are placed in refrigerator and freezer units used for the storage of samples for volatiles analysis. Refer to Section 13.0 for storage blank procedures.

6.0 EQUIPMENT AND SUPPLIES

6.1 GC/MS: Hewlett-Packard (HP), Agilent 6890 Gas Chromatograph equipped with HP, Agilent 5973 Mass Spectrometer, Agilent 7890 GC equipped with Agilent 5977 MSD.

Chemstation: HP, Agilent Eviroquant; Agilent Mass Hunter

6.2 Purge-and-trap: Tekmar liquid sample concentrator (LSC), 3000, Stratum; Varian, Archon auto-sampler; Tekmar Atomx liquid sample concentrator/ autosampler.

6.3 Top loading balance: Ohaus Navigator, Mettler PE600,

6.4 Capillary column: Restek 502.2: 60 m, 0.32 mm ID, 1.8 μm film thickness. Restek RTX-VMS: 20 m, 0.18 mm, 1 mm film thinkness.

6.5 Trap: Supelco Vocarb 3000; Tekmar trap #9.

6.6 Volumetric flask: Class A; 1 mL to 200 mL

6.7 Mininert vial with septum valve: 1 mL to 10 mL

6.8 40 mL VOA vial: I-Chem, Thermo Scientific

6.9 Syringe: Hamilton Gas tight with Luer lock tip: 25 mL, 5 mL; Gas tight with fixed needle: 10 uL, 25 uL, 50 uL, 100 uL, 250 uL, 500 uL, 1000 uL (Hamilton syringes accuracy: ±1% at or above 10% of syringe volume)

6.10 Steel and wooden spatulas

6.11 Disposable Pasteur pipets

6.12 Equivalent equipment and supplies may be used.

6.13 Refer to Table 4 for suggested GC/MS and purge-and-trap operating parameters.

6.14 Computer, software, hardware:

Instrument	Operation System	Computer Name	Connection Type	Instrument Software
HPMS6	Windows XP Professional	C10028	1 Gbps	Enviroquant Chemstation C.00.00
HPMS8	Windows 7	HPMS8	10/100 Mbps	Enviroquant Chemstation C.00.00
HPMS9	Windows XP Professional	HPMS9	10/100 Mbps	Enviroquant Chemstation C.00.00
HPMS11	Windows XP Professional	HPMS11	10/100 Mbps	Enviroquant Chemstation C.00.00
HPMS17	Windows 7 Professional	Microbac-HP	1 Gbps	Masshunter B.07.00; MSD Chemstation F.01.00.1903

7.0 STANDARDS AND REAGENTS

All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and

working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.1 Primary calibration standards:

STANDARD	VENDOR	PART NUMBER	CONCENTRATION
502.2 Mega Mix with MTBE	Ultra	DWM-596	2000 ug/mL
Custom VOC Mix 3	Restek	567801	2000 ug/mL
2-Chloroethyl vinyl ether (2-CVE)	Ultra	EPA-1016	5000 ug/mL
Acrolein-Acrylonitrile Mix	Ultra	AMN-623	2000 ug/mL
Custom Concentrated Ketones #2	Restek	567523	2000 ug/mL
Vinyl Acetate	Restek	30216	2000 ug/mL
Custom 8260 VMS CCV ADDS Standard	Restek	569679	2000-4000 ug/mL
502.2 Mix #1	Restek	30042	2000 ug/mL
Freon 113	Restek	30462	2000 ug/mL
Mass. Oxygenates Standard	Supelco	21624806	2000-4000 ug/mL
1,3-Butadiene	Accustandard	S-406A-10X	2000 ug/mL
1-Bromopropane	Ultra	CUS-12711	10000 ug/mL

7.2 Primary internal and surrogate standard mixtures:

STANDARD	VENDOR	PART NUMBER	CONCENTRATION
Method 8260 Internal Standards	Ultra	STM-520	2500 ug/mL (fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4)
Method 8260 Surrogate Standard Mixture	Ultra	STM-530	2500 ug/mL (dibromofluorobenzene, 1,2-dichloroethane-d4, toluene-d8, 4-bromofluorobenzene)

7.3 Primary laboratory control sample (LCS) / matrix spike (MS), alternate source (ICV) standards:

STANDARD	VENDOR	PART NUMBER	CONCENTRATION
Volatile Organic Compound (VOC) Mixture	Accustandard	M-502A-R-PAK	200 ug/mL

Volatile Organic Compounds (VOC) Additional Mixture 8260 Calibration Mix 2	Supelco	21678315	200 ug/mL
Ma. Oxygenates Standard	CPI	Z-G34-120696-02	2000/4000 ug/mL
Gas Mix	CPI	Z-120313-02	2000 ug/mL
Vinyl Acetate	Accustandard	APP-9-211-20X	2000 ug/mL
Acrolein	Restek	30645	5000 ug/mL
1,3-Butadiene	Supelco	21696443	200 ug/mL
1-Bromopropane	Sigma-Aldrich	B78106-5mL	99%
Custom VMS LCS ADDS	Supelco	2195749	200-2000 ug/mL

NOTE: Source of LCS/MS/ICV standards different then primary calibration standards.

7.4 Primary 4-bromofluorobenzene (BFB) standard: Ultra STS-112, 2500 ug/mL

7.5 Intermediate calibration standards: Primary calibration standards diluted to prepare intermediate standards as follows:

INTERMEDIATE STANDARD	PRIMARY STANDARD	CONCENTRATION (ugmL)	VOLUME (uL)	FINAL VOLUME (mL methanol)	FINAL CONCENTRATION (ugmL)
VOA Mix 1	502.2 Mega Mix with MTBE	2000	500	5	200
	502.2 Calibration Mix #1	2000	500		
	Freon 113	2000	500		
VOA Mix 2	Custom Concentrated Ketones #2	2000	500	5	200
	2-Chlorethylvinyl ether (2-CVE)	5000	200		
VOA Mix 3	1,3-Butadiene	2000	500	5	200
	Custom VOC Mix 3	2000	500		
	1-Bromopropane	10000	100		
VOA Mix 4	Acrolein-Acrylonitrile Mix	2000	500	10	100-400
	MA Oxygentates Standard	2000-4000	1000		
Vinyl Acetate	Vinyl Acetate	2000	500	5	200

VMS Column

INTERMEDIATE STANDARD	PRIMARY STANDARD	CONCENTRATION (ugmL)	VOLUME (uL)	FINAL VOLUME (mL methanol)	FINAL CONCENTRATION (ugmL)
VOA Mix 1	502.2 Mega Mix with MTBE	2000	500	5	200
	Custom Standard (Gases)	2000	500		
VOA Mix 2	Custom Concentrated Ketones #2	2000	500	5	200
	2-Chlorethylvinyl ether (2-CVE)	5000	200		
VMS CCV STD	1,3-Butadiene	2000	500	5	200-400
	Acrolein-Acrylonitrile Mix	2000	500		
	Custom RTX-VMS Standard	200-400	500		
Vinyl Acetate	Vinyl Acetate	2000	500	5	200

7.6 Intermediate internal and surrogate standards preparation:

PRIMARY STANDARD	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL METHANOL)	FINAL CONCENTRATION (ug/mL)
Intermediate internal and surrogate standards	2500	1000	10	250

7.7 Intermediate LCS / MS / ICV standards prepared as follows:

INTERMEDIATE STANDARD	PRIMARY STANDARD	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL MEHTANOL)	FINAL CONCENTRATION (ug/mL)
Gases & Adds	Custom Gases	250	1800	10	20
	VOC Adds Mix	200	1000	10	20
Mega	VOC Mega Mix	200	1000	10	20
	1-Bromopropane	1000	20	10	20

	Intermediate*				
1,3-Butadiene and Vinyl Acetate Mix	Vinyl acetate	2000	100	10	20
	1,3-Butadiene	200	1000	10	20
Oxygenates LCS Mix	MA Oxygenates	2000-4000	500	10	100-200
	Acrolein	5000	200	10	100

1-Bromopropane Intermediate solution is prepared by diluting 75uL of 99% 1-Bromopropane into 10mL of methanol.

VMS Column

INTERMEDIATE STANDARD	PRIMARY STANDARD	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL MEHTANOL)	FINAL CONCENTRATION (ug/mL)
8260 LCS Mix	VOC Mix	200	1000	10	20
	Custom Gases	250	800	10	20
VMS ADDS Mix	Custom VMS LCS ADDS	200-2000	500	5	20-200
1,3-Butadiene and Vinyl Acetate Mix	Vinyl acetate	2000	100	10	20
	1,3-Butadiene	200	1000	10	20
Acrolein Alt	Acrolein	5000	100	5	100

7.8 50 ug/mL BFB intermediate solution preparation:

7.9 Working standards preparation

7.9.1 Working standards used for initial calibration and calibration verification are prepared by diluting intermediate standards in DI water as follows:

Soil Initial Calibration Standards, ug/Kg
(suggested preparation)

STOCK STANDARD, CONCENTRATION	WORKING STANDARDS CONCENTRATIONS (ug/Kg)								
	0.5	1 [5]	2 [25]	5 [50]	20 [80]	50* [100]	100 [200]	200	300 [300]
VOA Mix 1 (200 ug/mL)	N/A	N/A	N/A	N/A	5 uL	12.5uL	25 uL	50 uL	75 uL
VOA Mix 2 (200 ug/mL)	N/A	N/A	N/A	N/A	5 uL	12.5uL	25 uL	50 uL	75 uL
VOA Mix 3 (200 ug/mL)	N/A	N/A	N/A	N/A	5 uL	12.5uL	25 uL	50 uL	75 uL
VOA Mix 4 (200-400 ug/mL)	N/A	2.5 uL	6.25 uL	12.5uL	20 uL	25 uL	50 uL	N/A	75 uL
20 ppm mix 1+2+3 Intermediate Std	2.5 uL	5 uL	5 uL	12.5uL	N/A	N/A	N/A	N/A	N/A
Surrogate Standard (20 ug/mL)	N/A	5 uL	5 uL	12.5uL	N/A	N/A	N/A	N/A	N/A
Surrogate Standard (200 ug/mL)	N/A	N/A	N/A	N/A	5 uL	12.5uL	25 uL	50 uL	75 uL
Final Volume, DI Water (mL)	100	100	50	50	50	50	50	50	50

20 ppm intermediate = Mix 1 + Mix 2 + Mix 3

$$(20ppm\ intermediate = 50uL\ Mix1 + 50uL\ Mix2 + 50uL\ Mix3 + 350uL\ MeOH)$$

* Denotes CCV

[ ] Denotes Oxygenates

Water Initial Calibration Standards, ug/L
(suggested preparation)

STOCK STANDARD, CONCENTRATION	WORKING STANDARDS CONCENTRATIONS (ug/L)									
	0.3	**0.4**	**1 [5]**	**2 [25]**	**5 [50]**	**20 [80]**	**50* [100]**	**100 [200]**	**200**	**300 [300]**
VOA Mix 1 (200 ug/mL)	N/A	N/A	N/A	N/A	N/A	5 uL	12.5 uL	25 uL	50 uL	75 uL
VOA Mix 2 (200 ug/mL)	N/A	N/A	N/A	N/A	N/A	5 uL	12.5uL	25 uL	50 uL	75 uL
VOA Mix 3 (200 ug/mL)	N/A	N/A	N/A	N/A	N/A	5 uL	12.5uL	25 uL	50 uL	75 uL
VOA Mix 4 (200-400 ug/mL)	N/A	N/A	2.5 uL	6.25 uL	12.5uL	20 uL	25 uL	50 uL	N/A	75 uL
20 ppm mix 1+2+3 Intermediate Std	3 uL	2 uL	5 uL	5 uL	12.5uL	N/A	N/A	N/A	N/A	N/A
Surrogate Standard (10 ug/mL)	N/A	N/A	5 uL	5 uL	12.5uL	N/A	N/A	N/A	N/A	N/A
Surrogate Standard (100 ug/mL)	N/A	N/A	N/A	N/A	N/A	5 uL	12.5uL	25 uL	50 uL	75 uL
Final Volume, DI Water (mL)	200	100	100	50	50	50	50	50	50	50

20 ppm intermediate = Mix 1 + Mix 2 + Mix 3

$$(20ppm\ intermediate = 50uL\ Mix1 + 50uL\ Mix2 + 50uL\ Mix3 + 350uL\ MeOH)$$

* Denotes CCV
[ ] Denotes Oxygenates

7.9.2 Procedure for preparing working standard in volumetric flask: The appropriate volume of intermediate standard is injected into the expanded area of a volumetric flask containing DI water. The flask is adjusted to volume then inverted three times. An aliquot is transferred to a 5 mL Luer lock syringe or 40 mL VOA vial and placed on the autosampler.

7.9.3 Procedure for preparing standard in 5 mL Luer lock syringe: The volume of stock standard is injected into a 5 mL Luer lock syringe containing DI water.

7.10 Working standards used for LCS/MS/ICV are prepared by diluting intermediate standards in DI water as follows:

Water Analyses

STOCK STANDARD, CONCENTRATION	LCS/MS VOLUME (uL)	ICV VOLUME (uL)	FINAL VOLUME DI Water (mL)	LCS/MS FINAL CONCENTRATION (ug/L)	ICV FINAL CONCENTRATION (ug/L)
Gases & Adds (20 ug/mL)	50	125	50	20	50
Mega (20 ug/mL)	50	125	50	20	50
1,3-Butadiene Vinyl acetate (20 ug/mL)	50	125	50	20	50
Oxygenates (50-100 ug/mL)	50	50	50	100-200	100-200

Soil Analyses

STOCK STANDARD, CONCENTRATION	LCS/MS VOLUME (uL)	ICV VOLUME (uL)	FINAL VOLUME DI Water (mL)	LCS/MS FINAL CONCENTRATION (ug/Kg)	ICV FINAL CONCENTRATION (ug/Kg)
Gases & Adds (20 ug/mL)	5	12.5	5	20	50
Mega (20 ug/mL)	5	12.5	5	20	50
1,3-Butadiene Vinyl acetate (20 ug/mL)	5	12.5	5	20	50
Oxygenates (50-100 ug/mL)	5	5	5	100-200	100-200

7.11 50ng BFB: Prepared by diluting 10 uL of BFB intermediate solution in 50 mL of DI water then purging 5 mL (else 1 uL of BFB intermediate solution is injected into the GC injection port).

7.12 Purge and trap grade methanol: (Fisher Scientific)

7.13 Reagent water (ASTM Type II DI water, UV treated)

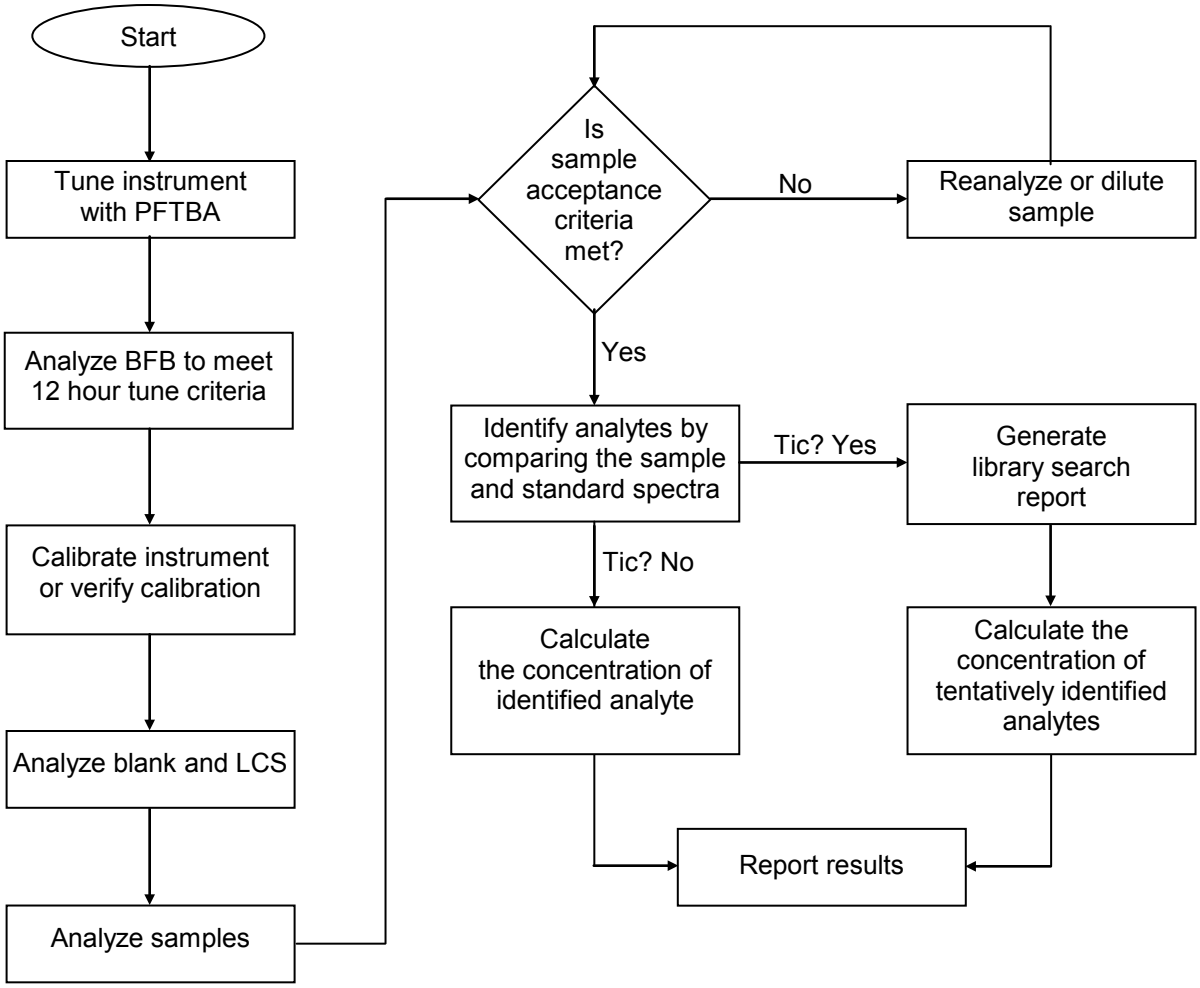
7.14 Purified Sand: J.T. Baker (Baked 150°C)

7.15 Concentration of calibration standards may vary depending on, but not limited to, availability, purity, and project requirements, therefore, recipes for standards preparation will be adjusted accordingly. Autosampler adds 1 uL of 250 ug/mL internal standards mixture.

7.16 Equivalent standards and reagents may be used.

7.17 Standards are stored at < 0°C or per manufacturer's instructions. Standards are stored in glass vials with Teflon-lined lids and/or mininert vials. Expiration dates for primary standards are per manufacturer's instructions; intermediate standards have a 30 day expiration date from the preparation date.

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



9.0 SAMPLE PREPARATION

9.1 Purge-and-trap procedures are found in Microbac SOP PAT01 for 5030C and 5035A.

10.0 CALIBRATION PROCEDURES

10.1 The GC/MS is hardware-tuned via auto-tune or manual tune.

10.2 50ng of BFB is analyzed via direct inject or purging and the mass spectrum is compared to acceptance criteria in Table 5. Evaluation is performed using the "Autofind" option of the Enviroquant software [the average of the apex, 1(-)apex, 1(+)apex is calculated and a background scan is then subtracted]. Once acceptance criteria is met, an initial calibration or calibration verification is performed. All standards, samples, and QC samples associated with a BFB analysis must use identical mass spectrometer instrument conditions.

10.3 For Initial calibration a minimum of five calibration levels containing target analytes and surrogate standards is required. The lowest calibration level must be equal to or below the required reporting limit/lower limit of quantitation for each analyte.

10.4 Standards used for soil calibration are loaded into 40 mL VOA vials containing 5.00 g (±0.1g) of oven baked reagent sand and utilize a heated purge (40° C).

10.5 Following analysis of the initial calibration, relative response factors (RRF) and average RRF for each surrogate and target analyte are calculated.

10.6 Five analytes designated as system performance check compounds (SPCC) must meet minimum average response factor criteria ($\overline{RRF}$) as follows:

COMPOUND	MINIMUM $\overline{RRF}$
chloromethane	0.10
1,1-dichloroethane	0.10
bromoform	0.10
chlorobenzene	0.30
1,1,2,2-tetrachloroethane	0.30

10.7 The percent relative standard deviation (%RSD) is calculated for all surrogates and target analytes. The %RSD for all target analytes must be less than 15%, however 6 analytes designated calibration check compounds (CCC) must have %RSD less than or equal to 30%. The CCC's are:

COMPOUND	ICAL MAX %RSD	CCV MAX %D
1,1-dichloroethene	± 30	± 20
chloroform	± 30	± 20
1,2-dichloropropane	± 30	± 20
toluene	± 30	± 20
ethyl benzene	± 30	± 20
vinyl chloride	± 30	± 20

10.8 Method 8260 Calibration Options

Linear – Using Average RF with RSD ≤ 15%

If the % RSD for all target analytes is less than or equal to 15%, then the response factor is assumed constant over the calibration range. Average response factor, therefore, may be used for quantitation. If the CCC's are not target analytes for a specific project, all required analytes must be ≤ 30% RSD.

If more than 5 calibration levels were analyzed, high and/or low points for poor responding and/or saturated compounds can be removed. The low calibration levels must be at or below the required reporting limit/lower limit of quantitation. The curve still must contain a minimum of 5 levels.

The average RF option is the preferred method of GC/MS calibration, since linearity may be assumed throughout the full calibration range. However, linear and quadratic models may be used under the conditions discussed in the following sections. If the % RSD for any target analyte is greater than 15%, one of the following procedures may be employed.

Linear Regression with Coefficient of Determination (COD) r^2 ≥ 0.99

Linear regression is an alternative to average RF, but has the potential for significant bias at the lower concentration levels .It should only be used when refitting the lowest calibration standard yields a maximum % error (12.7) of 30% (residual test). If a particular analyte exceeds 15% RSD, then linear regression may be utilized for that analyte. The fit for the equation (r^2) must be ≥ 0.99.

Quadratic Calibration with COD (r^2) ≥ 0.99

Several compounds on the 8260/8270 extended lists and the EPA Appendix IX list do not display consistently linear behavior. Quadratic calibration, employing at least six calibration points, may be used to improve accuracy for these analytes, particularly at the lower calibration levels, and is a better alternative than linear regression when linear fails the residual (% error) test. Quadratic calibration must never be used to compensate for a poorly maintained GC/MS system, and should not be used for analytes with a previous history of linear performance. Quadratic regression can be employed provided the COD (r^2) is ≥ 0.990. Those analytes utilizing first and/or second order calibration are noted on the initial calibration report.

NOTE: Origin not forced when using linear and quadratic regressions. Quadratic regression cannot be used to extend the calibration range.

For samples received from California, quadratic models for analytes that normally display linear responses in the calibration ranges will not be employed. Listed below are 8260 compounds that do not consistently exhibit linear behavior:

8260 Compounds	**Additional 8260 Compounds**	
vinyl acetate	t-butyl alcohol	paraldehyde
vinyl chloride	1,4-dioxane	1-bromopropane
2-chloroethylvinyl ether	propionitrile	isobutyl alcohol
naphthalene	tetrahydrofuran	1-butanol
acetone	acrolein	1-chlorohexane
	iodomethane	

10.9 Following the initial calibration an ICV is performed. Acceptance criteria is ± 20% drift. Criteria may be different depending on the sample's state of origin or a project specific QAPP.

10.10 The mid-point standard of the calibration curve must be used to establish the relative retention time window position for each analyte and surrogate.

10.11 A CCV is performed every 12 hours of analysis time following an acceptable BFB. Acceptance criteria:

10.11.1 SPCC's meet minimum $\overline{RRF}$ criteria in Section 10.6.

10.11.2 CCC's in Section 10.7 ≤ 20 % difference when using average response factor or ≤ 20 % drift when using regression fit. Non-CCC's must be ≤ 20% difference/drift. Some compounds are historically poor performing analytes and

may not meet ≤ 20% difference/drift criteria. Any compound not meeting criteria must be addressed in the case narrative. These compounds are listed below:

dichlorodifluoromethane	dimethyl disulfide
chloromethane	t-1,4-dichloro-2-butene
bromomethane	1,3-butadiene
chloroethane	acetonitrile
trichlorofluoromethane	2-chloro-1,3-butadiene
2-chloroethyl vinyl ether	ethyl acetate
acetone	methacrylonitrile
vinyl acetate	isobutyl alcohol
2-butanone	1-butanol
2-hexanone	methyl methacrylate
4-methyl-2-pentanone	2-nitropropoane
1,2-dibromo-3-chloropropane	cyclohexanone
bromoform	paraldehyde
acrolein	1-bromopropane
iodomethane	acrylonitrile

10.11.3 CCV internal standard response and retention times within –50% to +100% and ±30 seconds, respectively, compared to the same calibration standard in the initial calibration.

10.12 Single-point calibration may be performed for Appendix IX and F list analytes. Analytes detected above reporting limits/lower limit of quantitation require reanalysis using a multi-point calibration curve.

10.13 The separation of 2-chlorotoluene and 4-chlorotoluene will be evaluated for standards and QC samples using the "evaluation resolution" option in Chemstation. The resolution between 2-chlorotoluene and 4-chlorotoluene must be greater than 25% as evaluated by Chemstation.

10.14 Refer to Section 13.0 for quality control requirements and corrective action.

10.15 Calibration training materials are available on the intranet home page in the "General" links section, "Calibration Training". Review of "Calibration Models" and "The Effect that Saturation of the Detector has Upon Calibration" are recommended training for all new analysts. There are additional calibration training materials available through the same link on the homepage.

11.0 ANALYTICAL PROCEDURES

11.1 Prior to sample analysis, instruments must pass tuning and calibration criteria per Section 8.0.

11.2 Method blank: Analyzed prior to environmental samples. Method blanks are matrix specific.

11.2.1 Preparation of method blank

Water blank preparation: Fill a 40 mL VOA vial with UV-treated DI water (headspace not present). Place vial in Autosampler. Autosampler adds 1 uL of 250 ug/mL internal and surrogate standard mixtures.

Soil blank preparation: 5.00 g (±0.1g) of oven baked reagent sand is weighed into a tared 40 mL VOA vial containing a stir bar. 5 mL of UV-treated DI water is added to the vial. The vial is placed on the Autosampler. The Archon autosampler adds 5 mL of UV-treated DI water containing 1 uL of 250 ug/mL internal and surrogate standards mixtures. A 2 minute preheat (40° C) and heated purge (40° C) is utilized.

Middle-level extraction blank: 5.00 g (±0.1g) of oven baked reagent sand is weighed into a tared 40 mL VOA vial. 10 mL of methanol is added to the vial. The vial is shaken then allowed to settle. A 50x dilution is performed on the extract. The Autosampler adds 1 uL of 250 ug/mL internal standard mixture and surrogate standards mixtures.

11.3 Following the method blank a matrix specific LCS containing selected 8260 target analytes is analyzed. An LCS/LCS duplicate analyses is performed when the client does not provide sufficient volume for MS/MSD analyses.

Water and low-level soil LCS preparation: Refer to Section 7.0. **NOTE:** For low-level soil LCS, 2 minute preheat and heated purge (40° C) is utilized.

Middle-level extraction LCS: 5.00 g (±0.1g) of oven baked reagent sand is weighed into a tared 40 mL VOA vial. 8.5 mL of methanol and 0.5 mL of the LCS mixtures are added to the vial (**NOTE:** 8.5 mL methanol volume is dependent upon the number of LCS mixtures added; extract final volume is 10 mL). The vial is shaken then allowed to settle. A 50x dilution is performed on the extract. The dilution is loaded on the autosampler and analyzed.

11.4 MS/MSD are analyzed when the client provides appropriate sample volume.

11.4.1 Water MS/MSD preparation: Refer to Section 7.0 with the exception of sample used in place of DI water.

11.4.2 Low-level and mid-level soil MS/MSD preparation: Refer to Section 7.0 with the exception of 5.00 g (± 0.1 g) sample used in place of reagent sand.

11.5 Sample/sample duplicate analyses may be analyzed providing there is appropriate volume. Sample/sample duplicate analyses are generally associated with concentrated soil/waste samples and oils and are used to determine precision.

11.6 Samples are prepared per Microbac SOP PAT01.

11.7 Samples are analyzed within the 12 hour tune, which begins with the injection of BFB. At the end of tune time, a new BFB, blank, CCV, and LCS must be injected.

11.8 Once sample analysis is complete, a computer generated quantitation report containing all target analytes and their concentrations is generated. Also, detailed spectrum are generated for all target analytes detected above a nominal amount.

11.9 Qualitative analysis

An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). These standard reference spectra are obtained through analysis of the calibration standards. Two criteria must be satisfied to verify identification: (1) elution of the sample component at the same GC relative retention time (RRT) as the standard component; and (2) correspondence of the sample component and the standard component mass spectrum.

11.9.1 The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within the same 12 hours as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT must be assigned by using extracted ion current profiles for ions unique to the component of interest.

11.9.2 All ions present in the standard mass spectrum at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum. The relative intensities of the characteristic ions must agree within 30% of the relative intensities of these ions in the reference spectrum.

Analyst judgment is permitted even if these criteria are not met. The positive identification of a hit should not be made based solely on the criteria mentioned above.

11.10 If the response for any target analyte exceeds the initial calibration range, the sample must be diluted. Dilutions are prepared so that the majority of compounds above the calibration range fall near the midpoint of the calibration. Water and mid-level soil dilutions are prepared by using syringes or pipets to transfer aliquots of sample into a volumetric flask containing DI water. Examples of water dilutions are presented below.

DILUTION	SAMPLE VOLUME (mL)	DI WATER VOLUME (mL)	FINAL DILUTION VOLUME (mL)
10x	5	45	50
50x	1	49	50
100x	0.5	49.5	50
1000x	0.05	49.95	50

Low-level soil dilutions are prepared by weighing an aliquot less than 5.00 g in a 40 mL VOA vial. Examples of low-level soil dilutions:

DILUTION	SAMPLE AMOUNT (g)	DI WATER VOLUME (mL)
2x	2.5	5
2.5x	2.0	5
5x	1.0	5

11.10.1 Low level soils collected via 5035 must utilize the mid-level aliquot for dilutions.

Mid-level soil dilutions are prepared by diluting an aliquot of the methanol extract in a volumetric flask. Examples of mid-level soil dilutions:

DILUTION	METHANOL EXTRACT VOLUME (mL)	DI WATER VOLUME (mL)	FINAL VOLUME (mL)
50x	1	49	50
100x	0.5	49.5	50
500x	0.1	49.9	50
1000x	0.05	49.95	50

11.11 The raw data is processed using the chem station software and the data is uploaded into the LIMS. The laboratory then performs a primary and secondary review of the raw data and quality control forms.

11.12 Tentatively identified compounds (TIC): For samples containing components not associated with the calibration standards, a library search may be performed for the purpose of tentative identification. Guidelines for making tentative identification are:

Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) must be present in the sample spectrum.

The relative intensities of the major ions must agree within ± 40% for TIC's. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% -70%.)

Molecular ions present in the reference spectrum must be present in the sample spectrum.

Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of coeluting compounds.

Ions present in the reference spectrum but not in the sample spectrum must be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.13 Wipe Sample analysis: Wipes must be stored in 40 mL VOA vials containing 10 mL of methanol after collection. Prior to analysis, a glass rod (or similar) is inserted through the septum to completely immerse the wipe in methanol. Vortex the vial for 20 seconds. Remove ample volume of methanol by inserting the needle of a syringe (or similar) through the septum for a 50x dilution. Prepare as per the mid-level extraction procedures.

12.0 DETAILS OF CALCULATIONS

12.1 Relative response factor (RRF):

$$RRF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

where:

A_x = Area of the characteristic ion for the surrogate or compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard.
C_x = Concentration of the surrogate or compound being measured.

12.2 Average RRF $(\overline{RRF})$:

$$\overline{RRF} = \frac{\sum_{1}^{n} RRF}{n}$$

12.3 Standard deviation (s):

$$s = \sqrt{\frac{\sum (x - \bar{x})^2}{n-1}}$$

12.4 Percent relative standard deviation (%RSD):

$$\%RSD = \left(\frac{s}{\bar{x}}\right)100$$

where:

$\bar{x} = \overline{RRF}$: $\overline{RRF} = \frac{\sum_{1}^{n} RRF}{n}$

s = standard deviation(s): $s = \sqrt{\frac{\sum\left(x-\bar{x}\right)^2}{n-1}}$

12.5 Percent recovery (%R)

LCS, surrogate:

$$\%R = \left(\frac{C_x}{C_t}\right)100$$

where:

C_x = the concentration of the analyte in the LCS
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

MS/MSD:

$$\%R = \left[\frac{\left(C_{spk} - C_x\right)}{C_t}\right]100$$

where:

C_{spk} = the concentration of the analyte in the spiked sample
C_x = the concentration of the analyte in the reference (parent) sample
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

12.6 Relative percent difference (RPD):

$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2}\right]100$$

where:

C_1 = concentration of the first sample
C_2 = concentration of the second sample

12.7 Percent difference (%D), percent drift (% drift), percent error (% error):

$$\%D = \left[\frac{(C_t - C_x)}{C_t}\right]100$$

where:

C_t = True concentration of the analyte or surrogate in the standard
C_x = Measured concentration of analyte or surrogate in the standard 12.8

Coefficient of correlation (r):

$$\frac{\sum XY - \sum X \sum Y / n}{\sqrt{\left(\sum X^2 - \left(\sum X\right)^2 / n\right)\left(\sum Y^2 - \left(\sum Y\right)^2 / n\right)}}$$

where:

X = individual values of the independent variable, i.e. concentration
Y = individual values of the dependent variable, i.e. response
n = number of pairs of data

12.8 Coefficient of determination (COD):

$$\left[\frac{\sum XY - \sum X \sum Y / n}{\sqrt{\left(\sum X^2 - \left(\sum X\right)^2 / n\right)\left(\sum Y^2 - \left(\sum Y\right)^2 / n\right)}}\right]^2$$

12.9 Sample concentration using $\overline{RRF}$:

Water (ug/L):

$$ug/L = \frac{(A_x)(I_s)(DF)}{(A_{is})(\overline{RRF})(V_o)}$$

where :

A_x = area of characteristic ion for compound being measured
I_s = amount of internal standard injected (250ng)
A_{is} = area of characteristic ion for the internal standard
$\overline{RRF}$ = mean relative response factor for compound being measured
V_o = volume of water purged (10mL)

DF = dilution factor

Low-level soil/sediment:

$$ug/Kg = \frac{(A_x)(I_s)}{(A_{is})(\overline{RRF})(W_s)(D)}$$

where :

A_x, I_s, A_{is}, $\overline{RRF}$, = same as for water
W_s = weight of sample purged in grams
D = % dry weight of sample divided by 100, or 1 for a wet-weight basis

Medium-level soil/sediment:

$$ug/kg = \frac{(A_x)(I_s)(V_i)(DF)}{(A_{is})(\overline{RRF})(V_o)(V_t)(D)}$$

where :

A_x, I_s, A_{is}, $\overline{RRF}$, V_o, DF= same as for water
W_s = weight of sample extracted in grams
V_t = volume of total extract (mL) = $V_m + \left[(W_s)\left(\frac{100-D}{100}\right)\right]$
V_m = adjusted volume of solvent
V_i = volume of extract added (mL) for purging
D = % dry weight of sample (not applicable for a wet-weight basis)

12.10 Linear calibration calculations:

The response ratio is plotted vs. the concentration ratio giving a linear equation:

$$y = mx + b$$

where:

y = Response ratio = Response(x)/Response(istd) = R_x/R_{istd}
x = Concentration ratio = Conc(x)/Conc(istd) = C_x/C_{istd}
And m and b are the slope and intercept from the regression equation

For a given response ratio we can solve for C_x/C_{istd} :

$$C_x/C_{istd} = [R_x/R_{istd} - b]/m$$

Use equations 12.13 or 12.14 to calculate the unknown concentration, C_x.

12.11 Quadratic calibration calculations:

The response ratio is plotted vs. the concentration ratio giving a quadratic equation:

$$y = ax^2 + bx + c$$

OR

$$ax^2 + bx + (c - y) = 0$$

Solving for x using the quadratic equation:

$$x = \frac{b \pm \sqrt{(b^2 - 4a(c - y))}}{2a}$$

where:

y = Response ratio = Response(x)/Response(istd) = R_x/R_{istd}
x = Concentration ratio = Conc(x)/Conc(istd) = C_x/C_{istd}
a ,b, c are constants from the regression equation
Use equations 12.13 or 12.14 to calculate the unknown concentration, C_x

12.12 Solving for the concentration in water sample:

For a given concentration ratio, compute the unknown, C_x

$$C_x = (C_{is})(C_x/C_{istd})(V_f/V_i)(DF)(1000)$$

where:

C_{istd} = concentration of the internal standard (ug/mL)
V_f = final sample (extract) volume (mL)
V_i = initial sample volume (mL)
DF = dilution factor
C_x = concentration of the sample in ug/L

12.13 Solving for the concentration in soil sample:

$$C_x = (C_{is})(C_x / C_{istd})(V_f / W_i)(DF)(1000)$$

where:

C_{istd} = concentration of the internal standard (ug/mL)
V_f = final sample (extract) volume (mL)
W_i = initial sample volume (mL)
DF = dilution factor
C_x = concentration of the sample (ug/Kg) (as received)

12.14 Tentatively identified compounds (TIC) estimated concentration determination:

TIC water (ug/L):

$$ug/L = \frac{(A_x)(I_s)(DF)}{(A_{is})(\overline{RRF})(V_o)}$$

where :

A_x = total area of the peak from the total ion chromatogram
I_s = amount of internal standard injected (250ng)
A_{is} = total area of the internal standard from the total ion chromatogram
$\overline{RRF}$ = 1
V_o = volume of water purged (10mL)
DF = dilution factor

TIC low-level soil/sediment:

$$ug/Kg = \frac{(A_x)(I_s)}{(A_{is})(\overline{RRF})(W_s)(D)}$$

where :

A_x, I_s, A_{is}, $\overline{RRF}$, = same as for water
W_s = weight of sample purged in grams
D = % dry weight of sample divided by 100, or 1 for a wet-weight basis

TIC medium-level soil/sediment:

$$ug/Kg = \frac{(A_x)(I_s)(V_t)(DF)}{(A_{is})(\overline{RRF})(V_o)(W_s)(D)}$$

where :

A_x, I_s, A_{is}, $\overline{RRF}$, V_o = same as for water
W_s = weight of sample extracted in grams
V_t = volume of total extract (mL) = $V_m + \left[(W_s)\left(\frac{100 - D}{100}\right)\right]$
V_i = volume of extract added (mL) for purging
D = % dry weight of sample divided by 100, or 1 for a wet-weight basis
DF = dilution factor
V_m = volume of methanol added (mL)

12.15 Wipe:

$$ug/wipe = (C)(D_F)(E_V)$$

where:

C = extract concentration, ug/L
D_F = dilution factor
E_V = extract volume, L/wipe

NOTE: E_V assumed to be 0.01 L

13.0 QUALITY CONTROL REQUIREMENTS

13.1 The quality control procedures discussed in this section are intended to monitor and control the entire analytical process. Batch quality samples are specified for ICAL, MB, LCS, MS, MSD, laboratory duplicates (LD), and surrogate compounds. Additional procedures were defined in Section 8.0 for initial calibration, ICV using a second source, and CCV, and are included in the overall review process. The procedures, required frequency, acceptance criteria, and the required corrective action measures are outlined in Table 7.

13.2 Workgroups are analytical batches that contain instrument performance checks (BFB), calibration standards (ICAL, ICV, CCV), QC samples, and client samples.

13.3 Workgroups are comprised of:

- Instrument performance check: BFB tune evaluation to verify detector is working properly
- ICAL/CCV standards: used to calibrate instrument or verify accuracy of the calibration curve
- ICV: standard from an alternate source used to verify accuracy of the calibration curve
- Method blank: verify system is free of contaminants and interferences
- LCS/LCSD: verify precision and accuracy of the system
- MS/MSD: measure matrix effect of environmental sample on target analytes; measure precision
- Sample/sample duplicate: dual analysis of environmental system to measure precision
- Environmental sample: samples submitted for analysis

13.4 Method blank analyzed per method requirements. Target analytes must be less than ½ the RL/LLOQ. Exceptions may include common laboratory solvents which must be less than the RL/LLOQ or the exceptions of section 13.11.3. All blanks are evaluated down to the current MDL for the presence of target analytes. Any amount of target analytes found in the blank at a level greater than the current MDL are reported in the LIMS and these values will appear on the QC summary sheet for the batch.

13.5 The LCS must be evaluated using acceptance criteria listed in Tables 2 and 3, as well as any project specific criteria. Upon completion of a batch of samples, LCS summary reports are generated by the analyst, which compare the actual recoveries to the applicable acceptance ranges for the samples in the batch. The standard laboratory limits specified in Tables 2 and 3 are used in the absence of a project QAPP or program specified control limits. If more than 10% of the LCS analytes are out of the laboratory limits, the analyst must stop the analysis, prepare an NCR, and contact the department supervisor for the appropriate corrective action. If any of the identified project specific chemicals of concern (COC) are outside the control limits, the analyst must stop the analysis and prepare an NCR to be reviewed by the department supervisor.

13.6 The MS/MSD is analyzed per method requirements. MS/MSD results are included in the QC summary report and are used to monitor matrix accuracy and precision. For MS/MSD, Sample/sample duplicate, failure to meet surrogate and internal standard areas acceptance criteria does not necessarily warrant corrective action. Sample MS/MSD or sample/duplicate results can be used to confirm sample matrix interference. In obvious cases of error, reanalysis would be performed.

13.7 When ICAL acceptance criteria are not met, corrective action may include (but is not limited) to the following:

- Evaluate individual data points and reanalyze
- Evaluate calibration standards and reanalyze
- Prepare fresh calibration standards and reanalyze
- Perform instrument maintenance to include but not limited to:
  - Reanalyze calibration curve
  - Tune instrument
  - Replace trap
  - Bake analytical system
  - Replace column
  - Replace transfer line(s)
  - Service auto-sampler, sample concentrator, gas chromatograph, and/or mass spectrometer
- Qualify results and address in case narrative

13.8 When CCV acceptance criteria are not met, corrective action may include (but not, limited to) the following:

- Reanalyze CCV
- Prepare fresh standards
- Bake analytical system
- Perform instrument maintenance

13.9 Surrogate is added to all standards, QC samples, and environmental samples. Table 6 lists surrogate acceptance limits.

13.10 Storage blanks are placed in sample refrigerators and freezers to monitor potential cross contamination. Storage blanks consists of 40 mL of analyte free DI water or 5 mL DI water and 5.00 g of sand stored for 14 days in each VOA refrigerator/freezer. Analyses are performed via Method 8260 with results quantitated to the MDL. Storage blanks are prepared weekly and logged into the LIMS laboratory account. Weekly, (after storage blanks have been stored for two full weeks) storage blanks are analyzed via 8260 (storage blanks must be analyzed within the 12 hour tune time).

Target analytes must be less than ½ the reporting limit/lower limit of quantitation with the exception of common lab contaminants. Common lab contaminants must be less than the reporting limit/lower limit of quantitation. During primary review of the data, the analyst will review storage blank results to ensure acceptance criteria are met. If acceptance criteria are not met the analyst must

initiate corrective action. Corrective action begins with determining the non-compliant analyte(s) and recording any known reason for the failure then reanalysis of the duplicate vial of the storage blank. If reanalysis of the storage blank yields results within acceptance criteria then no further corrective action is required. If the reanalysis results confirm the initial analysis results or the reason for the initial failure is not evident then a Form NC02 is initiated. After primary review, results are uploaded to the LIMS. The laboratory will conduct an internal investigation and assess impact on associated samples if these criteria are not met with the next group of storage blanks analyzed. The laboratory will attempt to identify the source of contamination, and evaluate the impact on data reported for the contaminant during the period of storage. Clients may be contacted based on the investigation, if the QAO judges that to be necessary.

13.11 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.11.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Table 7 does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Table 7.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch Figure 1. A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.11.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RAC, corrections, corrective action(s) and evidence of effectiveness.

13.11.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

13.12 Table 7 contains method 8260B quality control criteria.

13.13 LCS control limits are reviewed annually.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data review:

14.1.1 All data undergoes a 100% primary review to ensure method and project specific compliance, reduce the data into reportable results, and generate appropriate QC forms. All items in Figure 1 (data review checklist) are reviewed and results are uploaded to the LIMS.

14.1.1.1 Data may be reviewed by an analyst other than the primary analyst provided the reviewing analyst's initials are recorded on the "Data Checklist".

14.1.2 Following the primary review the data undergoes a 100% peer review. All items in 14.1.1 are repeated by the peer. The peer review is performed by the supervisor or designee.

14.2 Data reporting:

14.2.1 Following peer review all uploaded results are reviewed, verified, and qualified.

14.2.2 Default reporting units are “ug/L” for water and “ug/Kg” for soil/sediments/oils.

14.2.3 All uploaded results are uploaded to a maximum number of significant figures dictated by the LIMS. The number of significant figures in the final report vary per project requirements.

14.2.4 Dilution and sample matrix confirmation analyses are uploaded into the LIMS and per the client’s request may be reported as separate analyses or combined (concatenated) into one set of results.

14.3 Quantitative results between the MDL and RL/LLOQ are qualified as “estimated” if requested by the client.

14.4 Refer to Microbac SOP 41 for acceptable procedure on manual integration if necessary.

14.5 Electronic run logs and preparation logs are reviewed electronically.

15.0 PREVENTIVE MAINTENANCE

15.1 Gas pressures are monitored daily. Other maintenance performed as needed. Laboratory maintenance log books maintained per instrument.

15.2 Instrument configuration and maintenance is recorded in the instrument maintenance log book.

15.3 Trouble-shooting involves, but is not limited to, direct injections, chromatography review, evaluating contamination, standards recoveries, injection port maintenance, and leak check.

15.4 Vendor instrument repair reports will be included in maintenance log.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2 The waste streams are as follows:

- Volatile Laboratory – non-halogenated solvents, solid waste (methanol)

16.3 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33, Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. Our procedures comply with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal hazardous waste that this is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

17.0 REFERENCES

17.1 *Test Methods for Evaluating Solid Waste,* SW-846, US-EPA, Office of Solid Waste, including updates I, II, III, IV, and V:

8000D	July 2014
8260B	December 1996
5030C	May 2003
5035A	July 2002

17.2 AFCEE 1998 QAPP, Version 3.0, March 1998

17.3 AFCEE 2001 QAPP, Version 3.1, August 2001

17.4 AFCEE 2005 QAPP, Version 4.0, February 2005

17.5 U.S. EPA, 40 CFR, Part 136, October 26, 1984

17.6 Microbac SOP PAT01 “Methods 5030 and 5035 Purge and Trap for Volatile ` Organics”

17.7 Microbac SOP 45 “Method Validation Procedures”

17.8 Microbac SOP 41 “Manual Integration of Chromatographic Peaks”

17.9 Microbac SOP 33 “Laboratory Waste Management”

17.10 Microbac SOP LQAP “Laboratory Quality Assurance Plan”

17.11 Microbac SOP GP-TEMP-SSU “Temperature control Systems for Sample Storage Units”

Appendix I
Suggested 8260B Quantitation Ions

COMPOUND NAME	SUGGESTED PRIMARY ION	SUGGESTED SECONDARY ION	MICROBAC'S PRIMARY ION
fluorobenzene (internal std)	**96**	**77**	**96**
dichlorodifluoromethane	85	87	85
chloromethane	50	52	50
vinyl chloride	62	64	62
1,3-butadiene	54	39.53	54
bromomethane	94	96	94
chloroethane	64	66	64
Trichlorofluoromethane	101	151, 153	101
diethyl ether	59	45, 74	59
isoprene	67	53	67
acrolein	56	55, 58	56
trichlorotrifluoromethane	101	151	101
Acetone	43	58	43
1,1-dichloroethene	96	61, 63	96
t-butyl alcohol	59	41, 47	59
dimethyl sulfide	62	47	62
iodomethane	142	127, 141	142
methyl acetate	43	74, 59	43
acetonitrile	41	40, 39	41
methylene chloride	84	86, 49	84
carbon disulfide	76	78	76
acrylonitrile	53	52, 51	53
methyl-tert-butyl ether	73	57	73
3-chloro-1-propene	41	76	41
trans-1,2-dichloroethene	96	61, 98	96
n-hexane	57	43	57
diisopropyl ether	45	43, 87	45
vinyl acetate	43	86	43
1,1-dichloroethane	63	65, 83	63
ethyl-t-butyl ether	59	87, 57	59
2-butanone	43	72	43
2-chloro-1,3-butadiene	53	88, 90, 51	53
propionitrile	54	52, 55, 40	54
2,2-dichloropropane	77	97	77
cis-1,2-dichloroethene	96	61, 98	96
chloroform	83	85	83
1-bromopropane	122	124	122
bromochloromethane	128	49, 130	128
methacrylonitrile	41	67, 39, 52, 66	67
isobutyl alcohol	43	41, 42, 74	73
Tetrahydofuran	42	72, 71	42
dibromofluoromethane **(surrogate)**	111	113	111
1,1,1-trichloroethane	97	99, 61	97
cyclohexane	56	84	56
1,1-dichloropropene	75	110, 77	75
t-amyl-methyl ether	73	55, 87	73
carbon tetrachloride	117	119	117
1,2-dichloroethane-d4 **(surrogate)**	65	67	65
1,2-dichloroethane	62	98	62
1-butanol	56	43	56

Appendix I (continued)

COMPOUND NAME	SUGGESTED PRIMARY ION	SUGGESTED SECONDARY ION	MICROBAC'S PRIMARY ION
benzene	78	77, 52	78
Trichloroethene	130	95, 97, 132	130
Methylcyclohexane	83	55, 198	83
1,2-dichloropropane	62	112	62
Methyl methacrylate	41	100, 39, 69	41
1,4-dioxane	88	58, 43, 47	88
bromodichloromethane	83	85, 127	83
2-nitropropane	43	41	43
ethyl acetate	43	61	43
methyl methacrylate	41	69, 100	41
dibromomethane	93	95, 174	93
2-chloroethylvinyl-ether	63	65, 106	63
4-methyl-2-pentanone	100	43, 58, 85	58
cis-1,3-dichloropropene	75	77, 39	75
dimethyl disulfide	79	94	79
chlorobenzene-d5 (internal std)	**117**	**82**	**117**
toluene-d8 **(surrogate)**	98	100	98
toluene	92	91	91
ethyl methacrylate	69	41, 99, 86, 114	69
Paraldehyde	89	87	89
trans-1,3-dichloropropene	75	77, 39	75
1,1,2-trichloroethane	83	97, 85	97
2-hexanone	43	58, 58, 57, 100	43
1,3-dichloropropane	76	78	76
tetrachloroethene	164	129, 131, 166	164
dibromochloromethane	129	127	129
1,2-dibromoethane	107	109, 188	107
1-chlorohexane	91	55	91
chlorobenzene	112	77, 114	112
1,1,1,2-tetrachloroethane	131	133, 119	131
Ethylbenzene	106	91	106
m+p-xylene	106	91	106
Cyclohexanone	55	42, 98	55
o-xylene	106	91	106
styrene	104	78	104
bromoform	173	175, 254	173
isopropylbenzene	105	120	105
1,4-dichlorobenzene-d4 (internal std)	**152**	**115, 150**	**152**
1,1,2,2-tetrachloroethane	83	131, 85	83
p-bromofluorobenzene **(surrogate)**	95	174, 176	95
1,2,3-trichloropropane	75	77, 110	110
trans-1,4-dichloro-2-butene	53	88, 75	53
n-propyl-benzene	91	120	91
bromobenzene	156	77, 158	156
1,3,5-trimethylbenzene	105	120	105
2-chlorotoluene	91	126	91
4-chlorotoluene	91	126	91
alpha-methyl-styrene	118	103	118
tert-butyl-benzene	119	91, 134	119
1,2,4-trimethylbenzene	105	120	105
sec-butyl-benzene	105	134	105
p-isopropyl-toluene	119	134, 91	119

Appendix I (continued)

COMPOUND NAME	SUGGESTED PRIMARY ION	SUGGESTED SECONDARY ION	MICROBAC'S PRIMARY ION
1,3-dichlorobenzene	146	111, 148	146
1,4-dichlorobenzene	146	111, 148	146
n-butyl-benzene	91	92, 134	91
1,2-dichlorobenzene	146	111, 148	146
1,2-dibromo-3-chloropropane	75	115, 157	157
1,2,4-trichlorobenzene	180	182, 145	180
hexachlorobutadiene	225	223, 227	225
naphthalene	128	127	128
1,2,3-trichlorobenzene	180	182, 145	180

Appendix II
South Carolina Requirements

1.0 The following lists LCS control limits for analyses associated with South Carolina samples:

PARAMETER	WATER LCS	SOIL LCS
acetone*	60-140	60-140
benzene	70-130	70-130
bromobenzene	70-130	70-130
bromochloromethane	70-130	70-130
bromodichloromethane	70-130	70-130
bromoform*	60-140	60-140
bromomethane*	60-140	60-140
2-butanone*	60-140	60-140
n-butylbenzene	70-130	70-130
s-butylbenzene	70-130	70-130
t-butylbenzene	70-130	70-130
carbon disulfide	70-130	70-130
carbon tetrachloride	70-130	70-130
chlorobenzene	70-130	70-130
chlorodibromomethane	70-130	70-130
chloroethane*	60-140	60-140
2-chloroethyl vinyl ether*	60-140	60-140
chloroform	70-130	70-130
chloromethane*	60-140	60-140
2-chlorotoluene	70-130	70-130
4-chlorotoluene	70-130	70-130
1,2-dibromo-3-chloropropane*	60-140	60-140
1,2-dibromoethane	70-130	70-130
dibromomethane	70-130	70-130
1,2-dichlorobenzene	70-130	70-130
1,3-dichlorobenzene	70-130	70-130
1,4-dichlorobenzene	70-130	70-130
dichlorodifluoromethane*	60-140	60-140
1,1-dichloroethane	70-130	70-130
1,2-dichloroethane	70-130	70-130
1,1-dichloroethene	70-130	70-130
c-1.2-dichloroethene	70-130	70-130
t-1,2-dichloroethene	70-130	70-130

PARAMETER	WATER LCS	SOIL LCS
1,2-dichloropropane	70-130	70-130
1,3-dichloropropane	70-130	70-130
2,2-dichloropropane	70-130	70-130
1,1-dichloropropene	70-130	70-130
c-1,3-dichloropropene	70-130	70-130
t-1,3-dichloropropene	70-130	70-130
ethyl benzene	70-130	70-130
hexachlorobutadiene	70-130	70-130
2-hexanone*	60-140	60-140
isopropylbenzene	70-130	70-130
p-isoporpyltoluene	70-130	70-130
4-methyl-2-pentanone*	60-140	60-140
methylene chloride	70-130	70-130
naphthalene	70-130	70-130
n-propylbenzene	70-130	70-130
styrene	70-130	70-130
1,1,1,2- tetrachloroethene	70-130	70-130
1,1,2,2-tetrachloroethane	70-130	70-130
tetrachloroethene	70-130	70-130
toluene	70-130	70-130
1,2,3-trichlorobenzene	70-130	70-130
1,2,4-trichlorobenzene	70-130	70-130
1,1,1-trichloroethane	70-130	70-130
1,1,2-trichloroethane	70-130	70-130
trichloroethene	70-130	70-130
trichlorofluoromethane*	60-140	60-140
1,2,3-trichloropropane	70-130	70-130
1,2,4-trimethylbenzene	70-130	70-130
1,3,5-trimethylbenzene	70-130	70-130
vinyl acetate*	60-140	60-140
vinyl chloride*	60-140	60-140
o-xylene	70-130	70-130
m,p-xylene	70-130	70-130

* Denotes poor purging / poor performing compounds.

2.0 South Carolina requires all LCS compounds to meet acceptance criteria in Section 1.0 of Appendix V.

3.0 Analytes cannot be reported to South Carolina if the initial calibration fails to meet acceptance criteria. Quadratic calibration must never be used to compensate for a poorly maintained GC/MS system, and must not be used for analytes with a previous history of linear performance.

4.0 All surrogate compounds must be within control limits for samples from South Carolina.

5.0 Based on project requirements, water samples collected for the analysis of vinyl chloride, styrene, and 2-chloroethylvinylether must be collected without acid preservation and analyzed within seven days of collection. The laboratory will send two sets of sample containers (one set (2 vials) preserved with acid, the other set (2 vials) un-preserved for the three compounds) or collect all samples without acid preservation.

Table 1
Method Analytes for MSV01

ANALYTE	CAS NUMBER
1,1,1,2-tetrachloroethane	630-20-6
1,1,1-trichloroethane	71-55-6
1,1,2,2-tetrachloroethane	79-34-5
1,1,2-trichloro-1,2,2-trifluoroethane	76-13-1
1,1,2-trichloroethane	79-00-5
1,1-dichloroethane	75-34-3
1,1-dichloroethene	75-35-4
1,1-dichloropropene	563-58-6
1,2,3-trichlorobenzene	87-61-6
1,2,3-trichloropropane	98-18-4
1,2,4-trimethylbenzene	95-63-6
1,2,4-trimethylbenzene	95-63-6
1,2-dibromo-3-chloropropane	96-12-8
1,2-dibromoethane	106-93-4
1,2-dichlorobenzene	95-50-1
1,2-dichloroethane	107-06-2
1,2-dichloropropane	78-87-5
1,3,5-trimethylbenzene	108-67-8
1,3-butadiene	106-99-0
1,3-dichlorobenzene	541-73-1
1,3-dichloropropane	142-28-9
1,4-dichlorobenzene	106-46-7
1,4-dioxane	123-91-1
1-bromopropane	106-94-5
1-butanol	71-36-3
1-chlorohexane	544-10-5
2,2-dichloropropane	594-20-7
2-butanone	78-93-3
2-chloroethylvinylether	110-75-8
2-chlorotoluene	95-49-8
2-hexanone	591-78-6
2-nitropropane	79-46-9
4-chlorotoluene	106-43-4
4-methyl-2-pentanone	108-10-1
acetone	67-64-1
acetonitrile	75-05-8
acrolein	107-02-8
acrylonitrile	107-13-1
allylchloride (3-chloroprene)	107-05-1
a-methyl styrene	98-83-9
benzene	71-43-2
bromobenzene	108-86-1
bromochloromethane	74-97-5
bromodichloromethane	75-27-4
bromoform	75-25-2
bromomethane	74-83-9
carbon disulfide	75-15-0
carbon tetrachloride	56-23-5
chlorobenzene	108-90-7
chloroethane	75-00-3
chloroform	67-66-3
chloromethane	74-87-3

ANALYTE	CAS NUMBER
chloroprene (2-chloro-1,3-butadiene)	126-99-8
cis-1,2-dichloroethene	156-59-2
cis-1,3-dichloropropene	10061-01-5
cyclohexane	110-82-7
cyclohexanone	108-94-1
dibromochloromethane	124-48-1
dibromomethane	74-95-3
dichlorodifluoromethane	75-71-8
diethyl ether	60-29-7
diisopropyl ether	108-20-3
dimethyl disulfide	624-92-0
dimethylsulfide	75-18-3
ethyl acetate	141-78-6
ethyl ether	60-29-7
ethyl methacrylate	97-63-2
ethyl t-butyl ether	637-92-3
ethylbenzene	100-41-4
hexachlorobutadiene	87-68-3
iodomethane	74-88-4
isobutanol	78-83-1
isoprene	78-79-5
isopropyl benzene	98-82-8
m+p-xylene	179601-23-1
methacrylonitrile	126-98-7
methyl acetate	79-20-9
methyl cyclohexane	108-87-2
methylene chloride	75-09-2
methylmethacrylate	80-62-6
methyl-tert-butyl-ether	1634-04-4
naphthalene	91-20-3
n-butyl-benzene	104-51-8
n-hexane	110-54-3
n-propyl benzene	103-65-1
o-xylene	95-47-6
paraldehyde	123-63-7
p-isopropyl-toluene	99-87-6
propionitrile (ethyl cyanide)	107-12-0
sec-butyl-benzene	135-98-8
styrene	100-42-5
t-amylmethyl ether	994-05-8
t-butanol	75-65-0
tert-butyl-benzene	98-06-6
tetrachloroethene	127-18-4
tetrahydrofuran	109-99-9
toluene	108-88-3
trans-1,2-dichloroethene	156-60-5
trans-1,3-dichloropropene	10061-02-6
trans-1,4-dichloro-2-butene	110-57-6
trichloroethene	79-01-6
trichlorofluoromethane	75-69-4
vinyl acetate	108-05-4
vinyl chloride	75-01-4

Table 2
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR VOLATILE ORGANIC ANALYSES OF GROUNDWATER

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/L)	REPORTING LIMITS (ug/L)	LCS, MS/MSD, TRUE VALUE (ug/L)	SUGGESTED CALIBRATION RANGE (ug/L)
1,1,1,2-tetrachloroethane	630-20-6	80-130	20	0.25	5	20	200-300
1,1,1-trichloroethane	71-55-6	80-134	20	0.25	5	20	200-300
1,1,2,2-tetrachloroethane	79-34-5	79-125	20	0.2	5	20	200-300
1,1,2-trichloro-1,2,2-trif	76-13-1	40-160	20	1	10	20	200-300
1,1,2-trichloroethane	79-00-5	80-125	20	0.25	5	20	200-300
1,1-dichloroethane	75-34-3	80-125	20	0.125	5	20	200-300
1,1-dichloroethene	75-35-4	80-132	20	0.5	5	20	200-300
1,1-dichloropropene	563-58-6	75-130	20	0.25	5	20	200-300
1,2,3-trichlorobenzene	87-61-6	80-120	20	0.125	5	20	200-300
1,2,3-trichloropropane	96-18-4	75-125	20	0.75	5	20	200-300
1,2,4-trichlorobenzene	120-82-1	80-120	20	0.2	5	20	200-300
1,2,4-trimethylbenzene	95-63-6	80-125	20	0.25	5	20	200-300
1,2-dibromo-3-chloropropane	96-12-8	65-135	20	1.0	5	20	200-300
1,2-dibromoethane	106-93-4	80-129	20	0.25	5	20	200-300
1,2-dichlorobenzene	95-50-1	80-125	20	0.125	5	20	200-300
1,2-dichloroethane	107-06-2	80-129	20	0.25	5	20	200-300
1,2-dichloroethene (total)	540-59-0	80-124	20	0.25	5	40	200-300
1,2-dichloropropane	78-87-5	80-120	20	0.2	5	20	200-300
1,3,5-trimethylbenzene	108-67-8	80-127	20	0.25	5	20	200-300
1,3-butadiene	106-99-0	10-200	20	1	10	20	200-300
1,3-dichlorobenzene	541-73-1	80-120	20	0.25	5	20	200-300
1,3-dichloropropane	142-28-9	80-120	20	0.2	5	20	200-300
1,4-dichlorobenzene	106-46-7	80-120	20	0.125	5	20	200-300
1,4-dioxane	123-91-1	20-160	20	50	100	200	200-300
1-bromopropane	106-94-5	50-150	20	0.5	1	20	200-300
1-butanol	71-36-3	50-150	20	50	100	200	50-800
1-chlorohexane	544-10-5	80-127	20	0.125	1	20	200-300
2,2-dichloropropane	594-20-7	80-133	20	0.25	5	20	200-300
2-butanone	78-93-3	40-160	20	2.5	10	20	200-300
2-chloroethyl vinyl ether	110-75-8	45-160	20	2.0	10	20	200-300
2-chlorotoluene	95-49-8	80-127	20	0.125	5	20	200-300
2-hexanone	591-78-6	55-130	20	2.5	10	20	200-300
2-nitropropane	79-46-9	10-150	20	5	50	100	200-300
3-chloro-1-propene	107-05-1	70-130	20	2.5	10	20	200-300
4-chlorotoluene	106-43-4	80-126	20	0.25	5	20	200-300
4-methyl-2-pentanone	108-10-1	64-140	20	2.5	10	20	200-300
acetone	67-64-1	40-180	20	2.5	10	20	200-300
acetonitrile	75-05-8	70-130	20	5	100	100	200-300
Acrolein	107-02-8	10-200	20	20	100	20	200-300
acrylonitrile	107-13-1	50-150	20	2.5	100	20	200-300
alpha-methyl-styrene	98-83-9	50-150	20	0.5	10	20	200-300
benzene	71-43-2	80-121	20	0.125	5	20	200-300
bromobenzene	108-86-1	80-120	20	0.125	5	20	200-300
bromochloromethane	74-97-5	65-130	20	0.2	5	20	200-300
bromodichloromethane	75-27-4	80-131	20	0.25	5	20	200-300
bromoform	75-25-2	70-130	20	0.5	5	20	200-300

Table 2 (continued)

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/L)	REPORTING LIMITS (ug/L)	LCS, MS/MSD, TRUE VALUE (ug/L)	SUGGESTED CALIBRATION RANGE (ug/L)
bromomethane	74-83-9	50-150	20	0.50	10	20	200-300
carbon disulfide	75-15-0	58-128	20	0.50	5	20	200-300
carbon tetrachloride	56-23-5	65-140	20	0.25	5	20	200-300
chlorobenzene	108-90-7	80-120	20	0.125	5	20	200-300
chloroethane	75-00-3	60-135	20	0.5	10	20	200-300
chloroform	67-66-3	80-125	20	0.125	5	20	200-300
Chloromethane	74-87-3	60-140	20	0.5	10	20	200-300
chloroprene	126-99-8	70-130	20	2.5	100	100	200-300
cis-1,2-dichloroethene	156-59-2	75-125	20	0.25	5	20	200-300
cis-1,3-dichloropropene	10061-01-5	75-125	20	0.25	5	20	200-300
cyclohexane	110-82-7	70-130	20	0.58	10	20	200-300
cyclohexanone	108-94-1	10-140	20	5	100	100	200-300
dibromochloromethane	124-48-1	70-130	20	0.25	5	20	200-300
dibromomethane	74-95-3	75-125	20	0.25	5	20	200-300
dichlorodifluoromethane	75-71-8	40-160	20	0.25	5	20	200-300
diethyl ether	60-29-7	70-130	20	5	10	100	200-300
diisopropyl ether	108-20-3	70-130	20	5	10	100	200-300
dimethyl disulfide	624-92-0	70-130	20	1.0	10	20	200-300
dimethyl sulfide	75-18-3	70-130	20	0.5	10	20	200-300
ethyl acetate	141-78-6	70-130	20	5	50	100	200-300
ethyl benzene	100-41-4	80-122	20	0.25	5	20	200-300
ethyl methacrylate	97-63-2	70-130	20	1.0	10	20	200-300
ethyl-tert-butyl ether	637-92-3	70-130	20	5	10	100	200-300
hexachlorobutadiene	87-68-3	72-132	20	0.25	5	20	200-300
iodomethane	74-88-4	10-160	20	0.5	10	20	200-300
Isobutanol	78-83-1	10-180	20	50	100	200	50-800
isoprene	78-79-5	70-130	20	0.53	10	20	200-300
isopropylbenzene	98-82-8	80-122	20	0.25	5	20	200-300
m+p-xylene **	179601-23-1	80-122	20	0.5	5	40	5-800
methacrylonitrile	126-98-7	70-130	20	2.5	5	100	200-300
Methyl acetate	79-20-9	50-190	20	1	10	20	200-300
Methyl cyclohexane	108-87-2	80-130	20	1	10	20	200-300
methyl methacrylate	80-62-6	70-130	20	2.5	5	100	200-300
methylene chloride	75-09-2	80-123	20	0.25	5	20	200-300
methyl-tert-butyl ether	1634-04-4	75-130	20	0.5	5	20	200-300
naphthalene	91-20-3	59-130	20	0.2	5	20	200-300
n-butylbenzene	104-51-8	80-131	20	0.25	5	20	200-300
n-hexane	110-54-3	74-137	20	0.56	5	20	200-300
o-xylene	95-47-6	80-122	20	0.25	5	20	200-300
p-isopropyl-toluene	99-87-6	80-122	20	0.25	5	20	200-300
propionitrile	107-12-0	50-150	20	2.5	5	100	200-300
propylbenzene	103-65-1	80-129	20	0.125	5	20	200-300
sec-butylbenzene	135-98-8	80-127	20	0.25	5	20	200-300
styrene	100-42-5	80-123	20	0.125	5	20	200-300
tert-amyl-methyl ether	994-05-8	70-130	20	5	10	100	200-300
tert-butyl alcohol	75-65-0	10-180	20	50	100	200	50-800

Table 2 (continued)

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/L)	REPORTING LIMITS (ug/L)	LCS, MS/MSD, TRUE VALUE (ug/L)	SUGGESTED CALIBRATION RANGE (ug/L)
tert-butylbenzene	98-06-6	80.126	20	0.25	5	20	200-300
tetrachloroethene	127-18-4	80-124	20	0.25	5	20	200-300
tetrahydrofuran	109-99-9	60-140	20	5	50	100	200-300
Toluene	108-88-3	80-124	20	0.25	5	20	200-300
trans-1,2-dichloroethene	156-60-5	80-127	20	0.25	5	20	200-300
trans-1,3-dichloropropene	10061-02-6	80-130	20	0.5	5	20	200-300
trans-1,4-dichloro-2-butene	110-57-6	50-150	20	2.0	10	20	200-300
trichloroethene	79-01-6	80-122	20	0.25	5	20	200-300
trichlorofluoromethane	75-69-4	62-151	20	0.25	5	20	200-300
vinyl acetate	108-05-4	10-190	20	2.5	10	20	200-300
vinyl chloride	75-01-4	50-150	20	0.25	10	20	200-300
xylenes (total)	1330-27-7	80-121	20	0.5	15	60	5-1200

** Unresolvable compound

Table 3
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR VOLATILE ORGANIC ANALYSES OF SOLID WASTE

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/Kg)	REPORTING LIMITS (ug/Kg)	LCS, MS/MSD, TRUE VALUE (ug/Kg)	SUGGESTED CALIBRATION RANGE (ug/Kg)
1,1,1,2-tetrachloroethane	630-20-6	71-137	30	0.5	5	20	5-200
1,1,1-trichloroethane	71-55-6	70-135	30	0.5	5	20	5-200
1,1,2,2-tetrachloroethane	79-34-5	55-130	30	0.5	5	20	5-200
1,1,2-trichloro-1,2,2-trif	76-13-1	70-130	30	1.0	10	20	5-200
1,1,2-trichloroethane	79-00-5	60-125	30	0.5	5	20	5-200
1,1-dichloroethane	75-34-3	75-125	30	1.0	5	20	5-200
1,1-dichloroethene	75-35-4	65-135	30	0.5	5	20	5-200
1,1-dichloropropene	563-58-6	57-138	30	0.5	5	20	5-200
1,2,3-trichlorobenzene	87-61-6	60-135	30	0.5	5	20	5-200
1,2,3-trichloropropane	96-18-4	65-130	30	1.0	5	20	5-200
1,2,4-trichlorobenzene	120-82-1	65-130	30	0.5	5	20	5-200
1,2,4-trimethylbenzene	95-63-6	75-132	30	0.5	5	20	5-200
1,2-dibromo-3-chloropropane	96-12-8	40-135	30	2.0	5	20	5-200
1,2-dibromoethane	106-93-4	69-128	30	0.5	5	20	5-200
1,2-dichlorobenzene	95-50-1	70-130	30	0.5	5	20	5-200
1,2-dichloroethane	107-06-2	63-133	30	0.5	5	20	5-200
1,2-dichloroethene (total)	540-59-0	74-127	30	0.5	5	40	5-200
1,2-dichloropropane	78-87-5	72-130	30	0.5	5	20	5-200
1,3,5-trimethylbenzene	108-67-8	74-133	30	0.5	5	20	5-200
1,3-butadiene	106-99-0	40-160	30	1	10	20	10-200
1,3-dichlorobenzene	541-73-1	70-130	30	0.5	5	20	5-200
1,3-dichloropropane	142-28-9	65-128	30	0.5	5	20	5-200
1,4-dichlorobenzene	106-46-7	70-130	30	0.5	5	20	5-200
1,4-dioxane	123-91-1	50-150	30	50	100	200	5-200
1-butanol	71-36-3	50-150	30	50	100	200	50-400
1-chlorohexane	544-10-5	40-160	30	0.5	3	20	5-200
2,2-dichloropropane	594-20-7	66-135	30	0.5	5	20	5-200
2-butanone	78-93-3	37-180	30	2.5	10	20	5-200
2-chloroethyl vinyl ether	110-75-8	35-154	30	2.0	10	20	5-200
2-chlorotoluene	95-49-8	63-147	30	0.5	5	20	5-200
2-hexanone	591-78-6	45-145	30	2.5	10	20	5-200
2-nitropropane	79-46-9	60-140	30	5	50	100	5-200
3-choro-1-propene	107-05-1	50-150	30	2.5	10	20	5-200
4-chlorotoluene	106-43-4	70-138	30	0.5	5	20	5-200
4-methyl-2-pentanone	108-10-1	47-146	30	2.5	10	20	5-200
Acetone	67-64-1	20-160	30	5.0	10	20	5-200
acetonitrile	75-05-8	50-150	30	50	100	100	5-200
acrolein	107-02-8	50-150	30	20	100	20	5-200
acrylonitrile	107-13-1	60-140	30	2.5	100	20	5-200
alpha-methyl-styrene	98-83-9	70-130	30	0.5	10	20	5-200
benzene	71-43-2	70-130	30	0.5	5	20	5-200
bromobenzene	108-86-1	72-131	30	0.5	5	20	5-200
bromochloromethane	74-97-5	70-130	30	0.5	5	20	5-200
bromodichloromethane	75-27-4	72-137	30	0.5	5	20	5-200
bromoform	75-25-2	49-136	30	0.5	5	20	5-200
bromomethane	74-83-9	37-143	30	1.0	10	20	5-200

Table 3 (continued)

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/Kg)	REPORTING LIMITS (ug/Kg)	LCS, MS/MSD, TRUE VALUE (ug/Kg)	SUGGESTED CALIBRATION RANGE (ug/Kg)
carbon disulfide	75-15-0	39-139	30	0.5	5	20	5-200
carbon tetrachloride	56-23-5	59-136	30	0.5	5	20	5-200
chlorobenzene	108-90-7	70-130	30	0.5	5	20	5-200
chloroethane	75-00-3	52-135	30	1.0	10	20	5-200
chloroform	67-66-3	74-129	30	0.5	5	20	5-200
chloromethane	74-87-3	30-131	30	2.0	10	20	5-200
chloroprene	126-99-8	50-150	30	2.5	5	100	5-200
cis-1,2-dichloroethene	156-59-2	70-130	30	0.5	5	20	5-200
cis-1,3-dichloropropene	10061-01-5	70-142	30	0.5	5	20	5-200
Cyclohexane	110-82-7	70-130	30	1.0	10	20	5-200
cyclohexanone	108-94-1	60-140	30	5	50	100	5-200
dibromochloromethane	124-48-1	59-136	30	0.5	5	20	5-200
dibromomethane	74-95-3	69-130	30	0.5	5	20	5-200
dichlorodifluoromethane	75-71-8	25-130	30	1.0	5	20	5-200
diethyl ether	60-29-7	60-140	30	5	10	100	5-200
diisopropyl ether	108-20-3	60-140	30	5	10	100	5-200
dimethyl disulfide	624-92-0	60-140	30	0.5	10	20	5-200
dimethyl sulfide	75-18-3	60-140	30	0.5	10	20	5-200
ethyl acetate	141-78-6	60-140	30	5	50	100	5-200
ethyl benzene	100-41-4	70-130	30	0.5	5	20	5-200
ethyl methacrylate	97-63-2	60-140	30	1.0	10	20	5-200
ethyl-tert-butyl ether	637-92-3	60-140	30	5	10	100	5-200
hexachlorobutadiene	87-68-3	65-135	30	0.5	5	20	5-200
iodomethane	74-88-4	20-288	30	1.0	10	20	5-200
isobutanol	78-83-1	50-150	30	50	100	200	10-400
Isoprene	78-79-5	40-140	30	2.0	10	20	5-200
isopropylbenzene	98-82-8	68-129	30	0.5	5	20	5-200
m+p-xylene **	179601-23-1	70-130	30	0.5	5	40	5-200
methacrylonitrile	126-98-7	60-140	30	2.5	5	100	5-200
methyl acetate	79-20-9	70-130	30	1	10	20	5-200
methyl cyclohexane	108-87-2	70-130	30	1	10	20	5-200
methyl methacrylate	80-62-6	70-130	30	2.5	5	100	5-200
methylene chloride	75-09-2	74-128	30	1.0	5	20	5-200
methyl-tert-butyl ether	1634-04-4	54-151	30	0.5	5	20	5-200
naphthalene	91-20-3	50-146	30	0.5	5	20	5-200
n-butylbenzene	104-51-8	70-136	30	0.5	5	20	5-200
n-hexane	110-54-3	58-142	30	0.5	10	20	5-200
o-xylene	95-47-6	70-130	30	0.5	5	20	5-200
p-isopropyl-toluene	99-87-6	72-128	30	0.5	5	20	5-200
propionitrile	107-12-0	60-140	30	2.5	5	100	5-200
propylbenzene	103-65-1	72-136	30	0.5	5	20	5-200
sec-butylbenzene	135-98-8	71-132	30	0.5	5	20	5-200
styrene	100-42-5	74-130	30	0.5	5	20	5-200
tert-amyl-methyl ether	994-05-8	60-140	30	5	10	100	5-200
tert-butyl alcohol	75-65-0	50-150	30	50	100	200	50-400
tert-butylbenzene	98-06-6	72-130	30	0.5	5	20	5-200

Table 3 (continued)

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/Kg)	REPORTING LIMITS (ug/Kg)	LCS, MS/MSD, TRUE VALUE (ug/Kg)	SUGGESTED CALIBRATION RANGE (ug/Kg)
tetrachloroethene	127-18-4	72-130	30	0.5	5	20	5-200
tetrahydrofuran	109-99-9	50-150	30	25	50	100	5-200
toluene	108-88-3	77-126	30	0.5	5	20	5-200
trans-1,2-dichloroethene	156-60-5	72-127	30	0.5	5	20	5-200
trans-1,3-dichloropropene	10061-02-6	65-139	30	0.5	5	20	5-200
trans-1,4-dichloro-2-butene	110-57-6	50-150	30	1.0	10	20	5-200
trichloroethene	79-01-6	72-126	30	0.5	5	20	5-200
trichlorofluoromethane	75-69-4	48-154	30	1.0	5	20	5-200
vinyl acetate	108-05-4	10-150	30	2.5	10	20	5-200
vinyl chloride	75-01-4	45-140	30	1.0	10	20	5-200
xylenes (total)	1330-27-7	70-130	30	0.5	5	60	5-600

** Unresolvable compound

Table 4*

GC/MS PURGE AND TRAP PARAMETERS	
purge time	9 – 11 minutes
dry purge time	0 – 2 minutes
desorb preheat	245° C
desorb	0.5 – 1 minute at 250° C
bake	9 -12 minutes at 260° C
LSC temp	valve 150˚ C, lines: 150° C
Archon temp	valve 95˚ C, lines 110° C
Soil Vial Temp	40° C
GAS CHROMATOGRAPH PARAMETERS	
carrier gas	helium, 99.999 %
injector temperature	220° C
oven temperature program	35° C for 4 minutes 10° C/minute to 240° C (hold 2 minutes)
MASS SPECTROMETER PARAMETERS	
beginning mass	35
ending mass	265
scan rate / sampling	8 scans/sec

*** S**uggested parameters: Adjustments may be made to improve efficiency.

Table 5
BFB Key Ion Abundance Criteria

MASS	ION ABUNDANCE CRITERIA
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95%, but less than 101% of mass 174
177	5 to 9% of mass 176

Table 6
Surrogate Standards Recovery Limits

SURROGATE COMPOUND	8260 WATER *	AFCEE 1998 QAPP WATER	8260 SOIL *	AFCEE 1998 QAPP (3.0) SOIL	2001/2005 AFCEE WATER	2001/2005 AFCEE SOIL	8260 OIL
dibromofluoromethane	86-118	75-125	80-120	65-135	85-115	65-135	52-122
Toluene-d_8	88-110	75-125	81-117	65-135	81-120	84-116	35-127
4-bromofluorobenzene	86-115	75-125	74-121	65-135	76-119	84-118	26-158
1,2-dichloroethane-d_4	80-120	62-139	80-120	52-149	72-119	52-149	43-128

* Denotes one outlier permitted given % R > 10%.
Reanalysis required if two or more recovery results are outside acceptance limits.

Table 7
Quality Control Criteria
Volatile GC/MS Analysis
Method 8260B

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Mass spectral ion intensities (BFB criteria)	Every 12 hours prior to ICAL, ICV or CCV	See Table 5	Retune instrument and repeat BFB check
Initial Calibration (ICAL)	When Continuing Calibration is out of control or when system conditions have been altered.	≤ 30% RSD for CCC compounds, < 15% RSD for all target compounds, if >15% RSD, then linear regression, provided r ≥ 0.995 and % error ≤ 30%, then quadratic regression, provided r^2 ≥ 0.990 SPCC minimum $\overline{RRF}$	Evaluate cause; repeat calibration; or qualify data and discuss in narrative (1) See section 13.7 for additional corrective action.
Second source calibration verification (ICV)	After each initial calibration	≤ 20% drift for each analyte (1)	Re-analyze ICV; upon second failure, repeat initial calibration (1)
Continuing calibration verification (CCV)	Each 12 hours	SPCC minimum RRF, ≤ 20% difference/drift for each analyte	Re-analyze CCV; upon second failure, repeat initial calibration (1) See Section 13.8 for additional corrective action.
Internal standard (IS)	Every sample, standard, and quality control sample	Retention time within 30 seconds of IS retention time in ICAL midpoint STD and area within –50% t +100% of IS midpoint area	Check for MS malfunctions or interference; re-analyze sample
Method Blank (MB)	One per matrix/batch; maximum of 20 samples per batch	Target analytes < ½ RL/LLOQ except common laboratory solvents which must be < RL/LLOQ	Notify supervisor and initiate NCR; investigate; re-analyze samples
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	One per matrix/batch; maximum of 20 samples per batch	Target compounds within the designated ranges; use project QAPP or standard control criteria (1,2)	Notify supervisor and initiate NCR; investigate; re-analyze samples
Matrix Spikes/ Matrix Spike Duplicate (MS/MSD) Sample/Sample Duplicate	One per matrix/batch; maximum of 20 samples per batch	Target compounds within the designated ranges; use project QAPP or standard control criteria (1,2)	Qualify data and/or address in the report narrative
Surrogate spike	Every sample, standard, and quality control sample	Recoveries within designated ranges: use project QAPP or standard control criteria; (one surrogate outlier permitted provided % R > 10%) (1)	Notify supervisor and initiate NCR; investigate; re-analyze samples

(1) Evaluation criteria are often project specific. Check the project QAPP.
(2) Standard criteria are set at three standard deviations from the mean; 10% marginal failure allowed, otherwise re-extract and re-analyze batch; consult supervisor and project QAPP for any exceptions.

Figure 1

Checklist ID: 6687

Microbac Laboratories Inc.

Data Checklist

Date: _____

Analyst: _____

Analyst: NA

Method: _____

Instrument: _____

Curve Workgroup: NA

Runlog ID: _____

Analytical Workgroups: _____

Runlog ID number:	
System Performance Check	
BFB	
Initial Calibration	
Average RF	
Linear Reg or Higher Order Curve	
Second Source standard % Difference	
Continuing Calibration /Check Standards	
Project/Client Specific Requirements	
Special Standards	
Blanks	
TCL's	
Surrogates	
LCS (Laboratory Control Sample)	
Recoveries	
Surrogates	
MS/MSD/Duplicates	
Samples	
TCL Hits	
Spectra of TCL Hits	
Surrogates	
Internal Standards Criteria	
Library Searches	
Calculations & Correct Factors	
Dilutions Run	
Reruns	
Manual Integrations	
Case Narrative	
Results Reporting/Data Qualifiers	
KOBRA Workgroup Data	
Check for Completeness	
Primary Reviewer	
Secondary Reviewer	
Check for compliance with method and project specific requirements	X
Check the completeness of reported information	X
Check the information for the report narrative	X
Check the reasonableness of the results	X

Primary Reviewer: Secondary Reviewer:

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Figure 2
VOA Preparation/Preservation and Extraction Log

Microbac Laboratories Inc.
VOA Preparation/Preservation/Extraction Log

Workgroup (AAB#):WG270339
Method:8260
Reagent ID:RGT10001

Analyst:DGB
Run Date:06/05/2008 09:50

SAMPLE #	Fraction	Collected	Preserved	PCT-S	Tare Wt	Total Wt	Sample Wt	Water	MeOH	Vt	Comments
L08020355-08	A	04/20/08 11:12	06/05/08 09:50	79.86			30.12	5		5	5
L08020355-09	A	04/20/08 11:25	06/05/08 09:50	77.08			30.12	5		5	5
L08020355-09	B	04/20/08 11:25	06/05/08 09:51	77.08	5.12	30.1	24.98	5		5	5
L08020355-09	C	04/20/08 11:25	06/05/08 09:52	77.08			25.08		10	15.748816	5

Comments: 1 = improperly sealed cap; 2 = preserved out of hold; 3 = effervesced; 4 = preserved with NaHSO4; 5 = preserved by freezing; 6 = preserved in field

Analyst: Douglas Butcher

VOA_EXT - Modified 05/22/2008
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MICROBAC®

STANDARD OPERATING PROCEDURE
THERMO iCAP 6000/7000 SERIES
INDUCTIVELY COUPLED PLASMA
ATOMIC EMISSION SPECTROSCOPY
SW-846 METHOD 6010 / EPA 200.7

Issue / Implementation Date: 15 September 2014

Last Review Date: 15 September 2016

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved by:

Kim H. Rhodes, Metal Supervisor — 9-14-2016 Date

Maren M. Beery, Quality Assurance Officer — 9-14-16 Date

Leslie S. Bucina, Laboratory Manager — 9/14/16 Date

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SECTION **PAGE**

1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure covers the operation of a Thermo iCAP 6000/7000 Series Inductively Coupled Plasma spectrometer according to methods 6010B, 6010C, and 200.7 for the analysis of metals in digested soils, sludges, wastes, extracts, drinking and non-potable waters. Filtered waters preserved in acid may also be analyzed by this method.

1.2 For elements for which this method is applicable see Table 1.1.

Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices. The data shown in Section 4.0 provides concentration ranges for clean aqueous samples. Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

1.3 Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods (See Microbac SOPs ME401A, ME406 and ME407). The instrument is prepared for operation and stabilized through a warmup period. Once stabilized the instrument is calibrated and initial quality control elements are analyzed. When initial quality control requirements have been met sample analysis begins. Samples are analyzed for metals content by optical emission spectroscopy. The Thermo iCAP 6000/7000 optical design combines a high resolution echelle polychromometer with an upgraded charge injection device CID 86 chip detector. It is also equipped with a solid state dual-view RF generator. The instrument software provides real-time results for the analysis and allows for automated upload into the Laboratory LIMS system for editing and reporting purposes. iTEVA software is completely Windows based and easy to use. A basic understanding of windows operation enables the analyst to move throughout the software.

1.4 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

AVS	Acid Volatile Sulfides
BS	Blank spike
BSD	Blank spike duplicate
CCB	Continuing calibration blank
CCV	Continuing calibration verification
CID	Charge Injection Devise
COC	Coefficient of Correlation
DI water	Deionized water
HCl	Hydrochloric acid
HNO3	Nitric Acid

ICB	Initial calibration blank
ICV	Initial calibration verification
ICP	Inductively Coupled Plasma
IDL	Instrument Detection Limit
LCS	Laboratory control sample
LCSD	Laboratory control sample Duplicate
LIMS	Laboratory Information Management System
LLCCV	Low level continuing calibration verification
LLICV	Low level initial calibration verification
LOD	Limit of detection
LOQ/LLQD	Limit of Quantitation/Lower Limit of Quantitation
MDL	Method detection limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NCR	Nonconformance Report
OES	Optical Emission Spectrometry
QC	Quality Control
RGT	Reagent
RL	Reporting limit
SEM	Simultaneously Extracted Metals
SOP	Standard Operating Procedure
SDS	Safety Data Sheet
STD	Standard

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP

2.0 SAFETY PRECAUTIONS

2.1 The iCAP 6000/7000 is equipped with safety interlocks to protect the analyst and instrument from harm if something out of the ordinary happens. The system checks water flow, argon pressures, sample compartment door interlocks, and plasma stability. These interlocks are constantly monitored and displayed on the screen. If any interlock is interrupted, the plasma is automatically shut down. Never attempt to defeat any interlocks.

2.1.1 The following interlocks must be satisfied in order to ignite the plasma:

- The front door on the sample compartment must be closed
- Argon pressures for the torch must be correct
- Cooling water must be flowing to the RF coil

2.1.2 The following interlocks must not be interrupted while the system is operating:

- Argon or nitrogen purge for the detectors and the spectrometer optics must be functioning properly.
- The temperature of the optics housing must be 36-40° C
- The temperature of the generator must be less than 24° C
- The camera temperature must be -45°C to -50°C.

2.2 **WARNING:** Due to the high voltages and temperature, caution must be used in maintenance and troubleshooting.

2.3 **WARNING:** Use gloves, safety glasses, lab coats and/or other appropriate safety precautions when handling samples and reagents. Acids used in preparation and analysis of samples for metals are corrosive and may cause severe burns to body tissue. All samples are unknowns and must be treated as potentially hazardous.

2.4 SDSs for each analyte and reagent used within the laboratory are available to all employees. Consult SDSs prior to handling chemicals.

3.0 SAMPLE PRESERVATION AND STORAGE

Measurement	Digestion Vol./Wt. Req.*	Collection Vol./Wt.	Preservative	Holding Time**
Total recoverable	50 mL	250 - 1000 mL p	HNO3 to pH <2	6 months
Dissolved	50 mL	250 - 1000 mL p	HNO3 to pH <2 Filter on-site	6 months
Suspended	50 mL	250 - 1000 mL p	Filter on-site	6 months
Total	50 mL	250 - 1000 mL p	HNO3 to pH <2	6 months
Soil	1 g	200 g glass	≤6° C	6 months

p = plastic

* If insufficient sample volume is received a smaller volume of sample will be used and the reagents ratio will be reduced accordingly except for soils.

** Storage time allowed between sample collection and analysis when properly preserved and stored.

3.1 Sample preservation should be < 6° C. Samples exceeding the upper temperature limit are to be flagged CT1.

4.0 METHOD PERFORMANCE

Instrument Detection Limits

4.1 6010B/200.7 IDLs

6010B/200.7 IDLs are calculated by multiplying the standard deviation obtained from analysis of a reagent blank solution with seven consecutive measurements by the one-sided 98% confidence level t-statistic (3.14 is the t-statistic for seven samples). Each measurement is performed as though it were a separate sample (i.e, with rinsing in between).

IDLs must be determined quarterly. The IDL is required to be numerically less than the associated MDL.

6010C IDLs

6010C IDLs are estimated by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement is performed as though it were a separate sample (i.e., with rinsing in between). IDLs must be determined quarterly.

Method Detection Limits

4.2 The laboratory performed an initial assessment of the MDL using the procedures outlined in 40 CFR Part 136. Results are filed electronically at H:\DATA\COMMON\MDL.

4.3 The LOD, or verified MDL, are presented in Table 4.1 for 6010/200.7. These limits were established using verification procedures outlined in Microbac SOP 45.

4.4 The LOQ are the nominal laboratory RLs and were established per Microbac SOP 45. Actual project RLs may be higher.

4.5 Precision and accuracy data in Tables 4.2a and 4.2b were derived from an initial demonstration of capability using spiked control samples. Going forward, the laboratory will use results from LCS to assess precision/accuracy and to annually evaluate the associated control limits.

4.6 MDLs for EPA Method 200.7 must be determined annually using the procedures in 40CFR Part 136. Additionally, MDLs must be redetermined whenever there is any change to the sample preparation procedure, or any significant change to the instrument

Linear calibration ranges

4.7 Calibrate the instrument, as described in Section 10.0.

4.8 Run a series of increasing concentration standards close to the upper linear range of the instrument. It is suggested that multi-element standards be used for the procedure whenever possible.

4.9 The linear range is defined as the highest concentration where the measured value is within 10 % of the actual prepared value of the standard. The values reported in Tables 4.1a and 4.1b are 90% of the verified upper linear range.

4.10 Linear Dynamic Range verifications are analyzed quarterly and edited in LIMS and in the instrument software.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 IEC's (InterElement Corrections) are minimized by the use of the eschelle grating. However this grating does not eliminate all interferences due to spectral overlap. Table 5-1 lists the approximate IEC's necessary of analyte per unit (mg/L) of interferant for the Thermo iCAP 6000/7000. These IEC's are subject to change with modifications made to operating conditions, such as changes in coolant flow, power, nebulizer or even new torches.

5.2 The use of a peristaltic pump reduces physical interferences. However, samples with high dissolved solids, high acid concentration or high viscosity may need to be diluted.

6.0 EQUIPMENT AND SUPPLIES

6.1a Thermo iCAP 6000 with ESI SC-8 DXFAST autosampler.

6.1b Thermo iCAP 7000 with ESI SC-8 DXFAST autosampler.

6.2 Argon gas supply (liquid).

6.3 Dell intel Core 2 VPRO or DUO computer with Thermo Fisher Scientific iTEVA Analyst software.

6.4 IBM compatible printer

6.5 Peristaltic pump tubing

6.5.1 Black/Black – 0.32 mL/min (Sample and flush station).

6.5.2 Yellow/Orange – 0.51 mm I.D > (Internal Standard)

6.5.3 Red/Red – 1.14 mm I.D. (drain)

6.6 Mixing tee

6.7 Calibrated mechanical pipettes:

6.7.1 10 - 100 uL

6.7.2 100 - 1000 uL

6.7.3 1000 - 5000 uL

6.8 Metal-free plastic pipette tips (for the pipettes specified in 6.7)

6.9 Metal-free 15 mL plastic test tubes

6.10 Metal-free 50 mL plastic test tubes

6.11 Class A glass pipettes – for preparation of standard solutions

7.0 STANDARDS AND REAGENTS

7.1 Acids used in the preparation of standards and for sample processing must be reagent grade or better. Redistilled acids may be used.

All purchased stock solutions and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

Calibration Solutions

7.2 Mixed calibration stock standards are purchased from Inorganic Ventures as a KEM-CONC- 1A, 2, 3A standard set or equivalent.

7.3 1000 ug/mL zirconium - Inorganic Ventures or equivalent.

7.4 Working Calibration Solutions - For the high standard water matrix, dilute 5 mL each of 7.2 and 1 mL of 7.3 into 500 mL of 5% HCl 2% $HN0_3$ in DI water. For the 200.7 water matrix, dilute into 500 mL of 2% HCl and 2% HNO_3 in DI water. For the soil matrix, dilute into 500 ml of 5% HCl nad 5% HNO_3 in DI water. From the high standard, dilutions are made with the appropriate matrix matched water. Standard S0 is a matrix matched blank. Concentrations of each element in the calibration standard are given in Table 7.4.1 for Thermo iCAP 6000/7000.

7.5 Working CCV Solutions – The CCV is prepared for three different matrices. For the water matrix, dilute 5 mL each of 7.2 and 1 mL of 7.3 into 1000 mL of 5% HCL and 2% $HN0_3$ in DI water. For 200.7 water matrix, dilute into 1000 mL of 2% HCL and 2% HNO_3 in DI water. For soil matrix, dilute into 1000 mL 5% HCL and 5% HNO_3 in DI water. Concentrations for each element are listed in Table 7.5.1.

7.6 Working Calibration Blank Solution - A calibration blank of 5% HCL, 5% HNO_3 for soil, 5% HCL, 2% HNO_3 for water, and 2% HCl, 2% HNO_3 for 200.7 water is used to establish the analytical curve and is analyzed following each initial and continuing calibration standard analysis.

Initial Calibration Verification Solutions

7.7 The ICV concentration is at the same level as the midpoint standard used in the calibration and is a separately prepared, quality control analyzed and certified source from that of the calibration standards.

7.8 Mixed ICV stock solution is purchased from SPEX as XKES-S-250 and from SCP Science as custom mixes Plasmacal 1 and Plasmacal 3A or equivalent.

7.9 1000 ug/mL silicon – Inorganic Ventures or equivalent.

7.10 10,000 ug/mL phosphorus – CPI or equivalent

7.11 1000 ug/mL zirconium – CPI or equivalent.

7.12 Working ICV Solution – The ICV is prepared for three different matrices. For the water matrix, dilute 1 mL of 7.8, 1 mL of 7.9, a 0.2 mL of 7.10, and 1 mL of 7.11 into 200 mL of 5% HCl and 2% HNO_3 in DI water. For 200.7 water matrix, dilute into 200 mL 2% HCL and 2%HNO_3 in DI water. For soil matrix, dilute into 200 mL 5% HCL and 5% HNO_3 in DI water. Concentrations for each element are listed in Table 7.5.1.

Low Level Calibration Verification Solutions

7.13 The LLICV and LLCCV are analyzed for 6010C only. The LLICV and LLCCV are analyzed using the same solution with concentrations at or below the RL.

7.14 Mixed LLCV stock solution is purchased from Inorganic Ventures or equivalent. Concentrations of each metal are listed in Table 7.6.1

.

7.15 For the LLIC/LLCCV, dilute 7.14 to desired concentration so that the concentration of each element is at or below the RL. Dilute using the appropriate matrix matched acid water.

Interference Check Sample Solutions

7.16 Interference Check Sample Stock - An ICSA stock solution is purchased from Inorganic Ventures as CLPP-ICS-A or equivalent.

ICSAB standards are purchased from Inorganic Ventures as CLPP-ICS-A, CLPP-ICS-B and KEM-ICS-B-1A or equivalent.

7.17 Working Interference Check Sample Solutions -The ICSA is prepared for three different matrices. For the water matrix dilute 10 mL of the ICSA stock solution into 200 mL 5% HCl and 2% HNO_3 in DI water. For 200.7 water matrix, dilute 10 mL of the ICSA stock solution into 200 mL of 2% HCL and 2% HNO_3 in DI water. For the soil matrix dilute 10 mL of the ICSA stock solution into 200 mL 5% HCl and 5% HNO_3 in DI water. The ICSA solution will contain only aluminum, calcium, iron and magnesium in high concentrations. See Table 7.7.1.

The ICSAB is made for three different matrices. For the water mixture dilute 10 mL of CLPP-ICS-A, 1mL of CLPP-ICS-B and 1 mL of KEM-ICS-B-1A into 200 mL 5% HCL and 2% HNO_3 in DI water. For the 200.7 water matrix, dilute the same volumes of the ICSAB into 200 mL of 2% HCL and 2% HNO_3 in DI water. For the soil matrix dilute same volumes of the ICSAB into 200 mL 5% HCL and 5% HNO_3 in DI water. The ICSAB will have the same concentrations for aluminum, calcium, iron and magnesium as the ICSA with additional metals spiked at detectable levels. See Table 7.7.1.

Internal Standard Solutions

7.18 Internal Standard Stock - single element yttrium 10,000 ug/mL from Inorganic Ventures or equivalent.

7.19 Working Internal Standard Solution - For ICP-Thermo 1 and ICP Thermo 2, 5mg/L yttrium is prepared by diluting 1 mL of Y stock into 2000 mL of the appropriate matrix matched acid water.

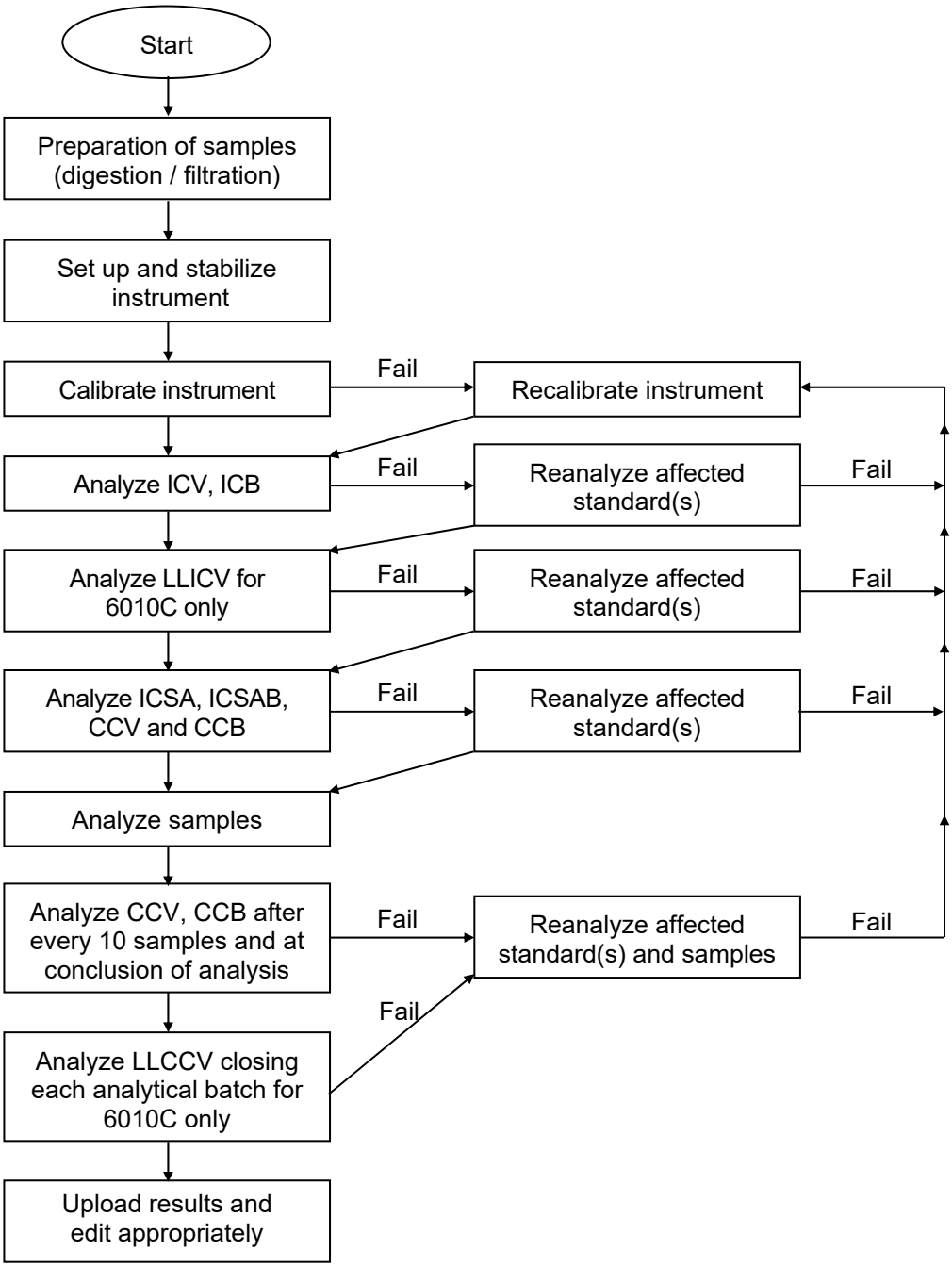
For ICP- Thermo 3 and ICP Thermo 4, 10 mg/L yttrium is prepared by diluting 2 mL of Y stock into 2000 mL of the appropriate matrix matched acid water.

7.20 Concentrated hydrochloric acid (HCl). Baker Instra Analyzed Grade or better

7.21 Concentrated nitric acid ($HNO_{3)}$. Baker Instra Analyzed Grade or better.

7.22 DI water – ASTM Type II or equivalent (ASTMD 1193)

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



9.0 SAMPLE PREPARATION

9.1 Sample preparation is dependent on matrix and digestion type. Refer to the following methods:

- ME401A - ME401A "Preparation Aqueous Samples for Determination of Metals By Inductively Coupled Plasma Atomic Emission Spectroscopy and Inductively Coupled Plasma Mass Spectroscopy (EPA 200.7) (EPA 200.8)"
- ME406 - Microwave Digestion of Sediments/Sludges/Soils/Oils, SW-846 Method 3051
- ME407 - Microwave Digestion – Aqueous, SW-846 Method 3015

10.0 CALIBRATION PROCEDURES

10.1 Initial Calibration – The instrument is calibrated before analysis of any samples with a blank and four calibration standards or alternatively with a blank and the high standard S4 from 7.4 (6010C only). Calibration Standards are prepared from SCP Science and SPEX stock. The dilutions of the calibration standards are listed in Section 7.4. The concentrations are listed in Table 7.4.1. For the multipoint calibration the instrument performs a weighted linear regression with a calculated intercept for all analyzed elements. For the single point calibration the instrument performs a linear regression for all analyzed elements (See Appendix A for calibration algorithms.). The correlation coefficients can be printed out when calibration is complete. The first standard run must be the calibration blank, followed by standards of increasing concentration in order to minimize cross-contamination and carryover. The prepared calibration standards are analyzed in three replicates with the reported results being the arithmetic mean (average) of the three replicate readings.

When performing the multipoint calibration the low calibration standard must contain the elements of interest at concentrations at or below the RL or a low level calibration check standard at or below the RL must be analyzed after calibration and before sample analysis. The LLICV must always be analyzed for 6010C. See Tables 13.1, 13.2 and 13.4 for acceptance criteria and corrective action for the curve and low level calibration check standard.

10.2 ICV Analysis - ICV analysis must be performed immediately after calibration standards to verify calibration. See Tables 13.1, 13.2, and 13.4 for acceptance criteria and corrective actions.

10.3 CCV Analysis - The CCV is required to be run after every 10 samples, at the end of the analysis and prior to sample analysis. See Tables 13.1, 1 3.2, and 13.4 for acceptance criteria and corrective action.

10.4 ICB and CCB Analysis - The solution used is the calibration blank (7.6). See Tables 13.1, 13.2, and 13.4 for acceptance criteria and corrective action.

10.5 ICSA and ICSAB Analysis – Required at the beginning of analytical run. See Tables 13.1, 13.2 and 13.4 for acceptance criteria and corrective action.

10.6 LLICV and LLCCV Analysis – For 6010C only, is required at the beginning of the run (LLICV) and at a minimum after every analytical batch (LLCCV). See Table 13.4 for acceptance criteria and corrective action.

10.7 Calibration training materials are available on the intranet home page in the "General" links section, "Calibration Training". Review of "Calibration Models" and "The Effect that Saturation of the Detector has Upon Calibration" are recommended training for all new analysts. There are additional calibration training materials available through the same link on the homepage.

11.0 ANALYTICAL PROCEDURES

11.1 Preliminary treatment of all matrices is always necessary because of the complexity and variability of sample matrices.

11.2 Startup Procedures

11.2.1 Pump tubing on instrument must be pliable and have only slight discoloration. If tubing is worn, replace. If tubing appears to be in good condition or once new tubing is installed, then connect the pump winding.

11.2.2 Turn on autosampler and chiller.

11.2.3 Turn on computer. Instrument and argon remain on at all times except when the instrument is to be shut down for an extended period of time.

11.2.4 Open ESI Software by double clicking on the icon. Click "Initialize Autosampler". Open iTEVA Control software by double clicking on the icon, enter analyst user name, click "OK". Wait for the "Initializing Instrument" procedure to complete.

11.2.5 Set up the instrument with the proper operating conditions. These conditions are found under Source Settings:

Plasma Settings:

- Flash Pump Rate 10 rpm
- Analysis Pump Rate 10 rpm
- Pump Stabilization time 0 sec

Source Settings:

- RF Power 1300W
- Auxiliary Gas Flow 0.70 L/min
- Nebulizer Gas Flow 0.65 L/min
- Coolant Gas Flow 12 L/min
- Water circulation on

11.2.6 After instrument is initialized, click on [Ignite] tab. This will open the "Plasma Status" box. Click on the "Plasma On", releasing the argon by lifting up the button from the Argon Humidifier to start the ignition sequence. After plasma has ignited, the setting will read approximately:

R.F. Power	1300 watts
Auxiliary Flow	0.7 L/min
Nebulizer Flow	0.65 L/min
Pump Rate	10-20 rpm
Coolant Gas Flow	12 L/min
Purge Gas Flow	Normal

11.2.7 The ICP must be allowed to become thermally stable before beginning analysis. (This usually requires approximately 30 minutes of operation prior to calibration.)

11.3 Setup of Analytical Sequence

11.3.1 After igniting plasma click the "Analyst" icon on iTEVA Control Center. This will open the "Select a Method" screen. Highlight the appropriate method and click [OK].

11.3.2 To set up the sample table Click on the "Sequence" tab. Right click on "Untitled(Manual)". Click on "Modify", in "Modify Automation Session" Box, choose "Autosampler", choose "CETAC by ESI SC8" (Emulation by ESI) . Click on [New], this will open "New Sequence" box. Highlight "Import from Delimited Text File" and choose file "FAST". Click [OK] "New Sequence" box, click [OK] on "Modify Automation Session" box. This will open the screen where sample information is entered. Make sure that work group numbers are filled in the "Comment" column

and dilution factors are filled in “Custom ID1” column. From “auto-session” tool bar, save the sample file.

11.3.3 To edit the Autosampler Table, double click “CETAC by ESI SC8” and double click “S_ICP-THERMO”. Positions for standards and samples will appear. Connect the autosampler to the PC by clicking on the “Initialize” icon in the sequence tab.

11.3.4 Click “Auto-Session” on the screen to open the appropriate sample file and make sure positions for samples are highlighted. Double click the second “Untitled CETAC by ESI SC8, then double click “S_ICP-THERMO”. The number of standards and samples will appear. Right click on “Standards, then click “Auto-Locate All” to make sure positions for standards are highlighted.

11.3.5 Torch Alignment: The torch alignment should only be performed after the torch has been replaced and before the auto peak. Manually place the carrier probe and the internal standard probe into a 2 mg/L zinc solution. Click the “Analysis” tab, select “Torch Alignment” from the instrument menu. Aspirate the zinc solution and click run. This procedure takes approximately ten minutes. After the torch alignment is complete, rinse both the carrier and internal standard probes with DI water before returning them back to their solutions.

11.3.6 Auto Peak: The peak location for the method lines associated with each high standard need to be justified. Manually place the carrier probe in the S4 cup. Click the “Analysis” tab, select “Perform Auto Peak Adjust” from the instrument menu, which presents the “Auto Peak Adjust” dialog box. Select “All Elements”, aspirate the high standard, and click run. Allow a delay after failures before re-initializing the auto peak routine. After the auto peak is complete, rinse the probe with DI water, then return the probe back to the carrier solution.

11.3.7 On the method tab, click “Automated Output”, change appropriate file name, click “Apply to All Sample Types” to set the new file name. Save the method.

11.4 6010/200.7

The prepared calibration standards, CCV and CCB are placed into 50 mL cups and are assigned positions by the analyst. The ICV, ICB, ICSA and ICSAB are placed into 15 mL test tubes and assigned positions by the analyst. This can be printed out by clicking on the “List View” icon in the “Sequence” screen to show sequence page and clicking on “Print Samples List”.

6010C only

The prepared calibration standards, CCV, and CCB are placed in 50 mL cups and are assigned positions by the analyst. The ICV, ICB, ICSA, ICSAB, LLICV, and LLCCV are placed into 15 mL rest tubes and assigned positions by the analyst. This can be printed out by clicking on the "List View" icon in the "Sequence" screen to show sequence page and clicking on "Print Samples List."

11.4.1 To start calibration, click on "Run-Auto-Session" in the "Sequence" screen.

11.4.2 To print correlation coefficients, click on "Element Calibration Report" from the "Method" screen. Right click on mouse in the [Calibration Report] screen, clicking [Print]. The correlation coefficients must be 0.995 or better for all elements needed for analysis for 6010B/200.7 and 0.998 or better for 6010C. Calibration is automatically stored until a new calibration is performed.

11.5 The prepared calibration standards, ICV, ICB/CCB, ICSA, ICSAB, CCV, LLICV, and LLCCV are analyzed in three replicates with the reported results being the average of the three replicate readings.

11.6 Upon completion of the calibration, begin following the analytical sequence as described below for:

6010B/200.7
ICV
ICB
ICSA
ICSAB
CCV
CCB
10 or less samples
CCV
CCB
Continue with the last three (3) steps until the end of run, always ending with a CCV/CCB.

6010C
ICV
ICB
LLICV
ICSA
ICSAB
CCV
CCB
10 samples or less
CCV
CCB
LLCCV
Continue with the last three (3) steps until the end of the run, always ending with a CCV, CCB, and LLCCV.

11.6.1 The samples are analyzed in three replicates with the reported results being the average of the three replicate readings. For any analyte with a result greater than the reporting detection limit, the %RSD between the replicate readings must be less than ten.

11.7 Flush the system with the rinse solution for thirty (30) seconds before the analysis of each sample. Analyze the CCV and CCB after each 10 samples at a minimum, and at the end of each analytical batch for 6010B/200.7. Analyze the CCV and CCB after every 10 samples at a minimum, and the CCV, CCB and LLCCV at the end of each analytical batch for 6010C.

11.8 Dilute and reanalyze samples that are more concentrated than the linear dynamic range.

11.9 After all sample analyses are complete, rinse the system by analyzing acid water and DI water, followed by air. Extinguish the plasma and release the pump tension. Turn off the autosampler.

11.10 Exit software and turn off computer.

12.0 DETAILS OF CALCULATIONS

12.1 All calculations for samples and standards are computed from the mean of three exposures. Each metal has a specified linear range. Refer to Section 4.0 for upper limits.

12.2 After the multipoint calibration is complete, the software performs a weighted linear regression with a calculated intercept for all metals analyzed. The instrument calculates the correlation coefficients for each metal and the analyst can view each curve for acceptance.

Following a single point calibration, the software performs a linear regression with a calculated intercept. Calculated correlation coefficients in this case are all equal to 1.0 (See Appendix A for the calibration algorithms.). All sample results are calculated from the calibration curve.

12.3 Dilution factors and preparation factors are calculated into the final result which is computed from the mean of three exposures.

12.3.1 For Liquid Samples:

$$mg/L\ metal\ in\ sample = mg/L\ in\ digestate * \frac{Final\ Prepared\ Volume\ (mL)}{Initial\ Volume\ (mL)} * \frac{Total\ Diluted\ Volume}{Sample\ Aliquot}$$

12.3.2 For Solid Samples:

$$mg/Kg\ metal\ in\ sample = mg/L\ in\ digestate * \frac{Final\ Prepared\ Volume\ (mL)}{Initial\ Weight\ (g)} * \frac{Total\ Diluted\ Volume}{Sample\ Aliquot}$$

12.3.3 LCS/LCS Duplicate % Recovery

$$\%\ Recovery = \frac{C_s}{C_t} * 100\%$$

where:

C_s = the LCS sample result
C_t = the LCS true value

12.3.4 MS/MSD % Recovery

$$\%\ Recovery = \frac{(C_s - C_a)}{C_t} * 100\%$$

where:

C_s = the MS/MSD sample result
C_a = the reference sample result
C_t = the MS/MSD true value

12.3.5 Post Digestion Spike Recovery

$$\%\ Recovery = \frac{(C_s - BC_a)}{C_t} * 100\%$$

where:

C_s = the spike sample result
C_a = the reference sample result
B = a factor to account for the dilution of the spiked sample relative to the reference sample (usually B = 0.9)
C_t = the spike true value

12.3.6 Duplicate RPD

$$RPD = \frac{|C_a - C_b|}{(C_a + C_b)/2} * 100\%$$

where:

C_a = the reference sample result
C_b = the duplicate sample result

12.3.7 Serial Dilution % Difference

$$\%D = \frac{|C_a - 5C_b|}{C_a} * 100\%$$

where:

C_a = the reference sample result
C_b = the diluted sample result

12.4 Calcium and magnesium results can be used to calculate water hardness results. The LIMS will use the uploaded calcium and magnesium results to calculate the hardness results. The automated hardness calculation is manually verified annually.

$$Hardness(mg/L) = 2.497[Ca(mg/L)] + 4.118[Mg(mg/L)]$$

12.5 Silicon results can be used to calculate silica results. The calculation assumes all silicon recovered from 6010 is in the form Si02. LIMS converts the results as follows:

$$Silica\,(mg/L) = silicon(mg/L) * 2.13923$$

12.6 See figure 12.1 for a sample calculation summary.

13.0 QUALITY CONTROL REQUIREMENTS

Overview

13.1 Refer to Section 10.0 for instrument calibration and instrument quality control samples. Each preparation batch (or workgroup) consists of a maximum of twenty (20) samples plus QC samples. The QC samples are prepared and digested identically to the analytical samples. The following QC are digested and or

analyzed with every preparation batch. The frequency, acceptance criteria and corrective action for this QC is listed in Tables 13.1, 13.2 and 13.4.

Batch Quality Control

13.2 Method blank (Prep Blank (PB)) an aliquot of DI water that is digested with the sample batch and contains all reagents identical with the samples.

13.3 LCS - for water matrix, a spiked DI water that is digested with the sample batch. For soil matrix; 1.0 gram of PTFE Boiling Stones (Teflon) Chemware is spiked prior to digestion and carried through the entire digestion procedure.

A LCSD may also be analyzed with the batch and is prepared identically to the LCS.

(**NOTE**: The LCS and LCSD must pass acceptance criteria or the samples will have to be re-digested/re-analyzed for the analyte in question.)

13.4 Sample duplicate - a sample prepared in duplicate, both carried through the digestion procedure. (EPA 200.7 or by client request only)

13.5 MS and MSD - a sample that is spiked in duplicate and then digested with the sample batch. It is prepared by taking 3 aliquots of sample, 2 of which are spiked with 5 mL of KEM-SPK-1F for each 50 mL of sample. The final concentration spiked into the two spiked samples will be the same as the aqueous and soil LCS level in Table 13.3.2.

Interference Tests-Post Digestion

13.6 With every analytical batch a post digestion spike and a serial dilution must be analyzed. If the matrix spike fails the 75 to 125% acceptance criteria, one of the following interference tests must pass for the analyte outlier:

13.7 Serial dilution: If the analyte concentration is sufficiently high (at least a factor of 50 above the MDL before dilution), analysis of a 1:5 dilution should agree within ± 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

13.8 Post Digestion Spike: an analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 to 125% of the known value for 6010B/200.7 and within 80 to 120% of the known value for 6010C. The spike addition should produce a minimum level of 10 times the instrument detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

13.9 Method of standard additions: This method must be employed for certain TCLP extracts and can also be used to compensate for sample constituents which enhance or depress the analyte signal producing a slope different from that of the calibration standards. It will not correct for additive interferences which cause a base line shift.

The simplest version of this techniques is the single-addition method, in which two identical aliquots of the sample, each of volume Vx are taken. To the first (A) a small volume (Vs) of a standard analyte concentration (Cs) is added. To the second (B) a volume (Vs) of the solvent is added. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration (Cx) is calculated:

$$Cx = (Sb)(Vs)(Cs)/(Sa - Sb)(Vx)$$

where:

Sa and *Sb* are the analytical signals of A and B respectively. *Vs* and *Cs* should be chosen so that *Sa* is roughly twice *Sb* on the average. It is best if *Vs* is made much less than *Vx*, and thus *Cs* is much greater than *Cx*, to avoid excess dilution of the sample matrix. Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected concentration from the endogenous analyte in the sample.

Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample concentration. The concentration of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero concentration, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

13.10 Linear Range Analysis/High-Level Standard

A linear range analysis is performed quarterly to determine the highest concentrations for each analyte at which the instrument yields a result within 10% of the true concentration. When use of the Department of Defense version 3 QAP is indicated, a high-level check standard with analyte concentrations at the linear range of the instrument may be analyzed

subsequent to sample analysis for any analyte which exceeds the calibration range. The recovery must be within 10% of the true concentration.

13.11 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.12 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Tables 13.1, 13.2 or 13.4, does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per 13.1, 13.2 or 13.4.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch, (Figure 14-1). A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.13 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RCA, corrections, corrective action(s) and evidence of effectiveness.

13.14 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

13.15 All data is scrutinized by the analysts for method and project specific compliance. Check lists are utilized and accompany each data batch (figure 14-1).

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data Review

Data is archived from the instrument computer to the LIMS where it is stored in a CSV format. When analysis is complete the analyst must upload the relevant CSV files including calibration, check standards, QA/QC samples and client samples into LIMS. This is done via Microbac's customized upload program.

When the upload is complete, the analyst must check the sample data for correct digestion factors, dilutions and RLs. Any elements that are not to be reported must be checked as excluded. This will be determined by the primary analyst through real time review of all quality control elements as summarized in Tables 13.1, 13.2, and 13.4. The analyst must certify that this primary review has been carried out by completing the Data Review Checklist (Figure 14.1), signing and dating**.** The analyst generates batch QA/QC summary forms automatically through an oracle program. The analyst must then assemble the hardcopy QA/QC summaries, batch upload reports, digestion logs and runlogs, case narratives if required and LIMS workgroup reports. The Data Review Checklist acts as a cover page. The completed package is then submitted for secondary review.

The secondary review consists of an additional 100% review of the hardcopy data for QA/QC compliance. This review consists of a double check of the batch QA/QC summary and associated post spikes and serial dilutions. Sample results are reviewed for completeness, reasonableness and compliance with any special project or client requirements. The case narrative, if any, is also checked for accuracy and completeness. The supervisor (or designate) also signs and dates the Data Review Checklist.

When all levels of review have been completed, the elements being reported on each sample are taken to a done status in LIMS.

15.0 PREVENTIVE MAINTENANCE

15.1 Check the torch and nebulizer every day and clean when needed depending on sample load.

15.2 Tubing needs to be changed when it loses pliability and is worn.

15.3 Clean the valve and all tubing when needed depending on sample load. Replace the rotor every six months or every 15,000 samples, whichever comes first.

15.4 The instruments are under service contracts so that every year a service representative will perform a systems check.

15.5 The water in the re-circulator/cooler must be changed yearly.

15.6 Troubleshooting and maintenance procedures can be found in Chapter 13 of the iCAP 6000 Series ICP-OES Spectrometer Hardware Manual and in the Help Menu of the iTEVA Analyst Software Control Center.

15.7 Performance problems (such as loss of signal or poor precision) are often related to the sample introduction system.

15.7.1 Perform the sodium bullet test to monitor sample flow.

15.7.2 Check pump tubing for flat spots, leaks or discoloration.

15.7.3 Check pump rollers for grooves or binding.

15.7.4 Check pump tubing for crimps, pinching and clogging.

15.7.5 Check nebulizer and spray chamber for leakage and proper drainage.

15.7.6 Check nebulizer spray pattern. The spray must not be uneven or sputtering.

15.7.7 Check the torch and injector for deposits, deformation and security of fit. O-rings must not be cracked or worn.

15.7.8 Check that the purge window is not cloudy or dirty.

15.8 The instrument configurations for the Thermo iCAP 6000/7000 series instruments can be found in the corresponding maintenance run logs. The template maintenance logs are found at numbers 13196 (ICP-Thermo1), 13199 (ICP-Thermo2), and 51236 (ICP-Thermo3) in LIMS. Replacement of instrument components will be recorded in the relevant maintenance log and updated in the log header as needed.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

16.2 Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2.1 The waste streams for the Metals Laboratory are as follows:

The metals laboratory waste is neutralized with sodium bicarbonate and flushed down the drain with tap water as per agreement with the Marietta Department of Water and Waste Water.

16.3 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33, Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in

accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous waste generator, we are subject to inspection from the Ohio EPA.

17.0 REFERENCES

17.1 Inductively Coupled Plasma – Atomic Emission Spectrometry, US EPA SW-846 Method 6010B, Revision 2, December 1996, EPA Publication SW-846

17.2 Us Environmental Protection Agency, EPA Method 200.7, Revision 4.4, 1994, 40CFR pt.136.

17.3 Inductively Coupled Plasma-Atomic Emission Spectrometry, US EPA SW-846 Method 6010C, Revision 3, February 2007, EPA publication SW-846.

17.4 Microbac SOP LQAP "Laboratory Quality Assurance Plan"

17.5 Microbac SOP 45 "Method Validation Procedures"

17.6 Microbac SOP ME401A "Preparation Aqueous Samples for Determination of Metals By Inductively Coupled Plasma Atomic Emission Spectroscopy and Inductively Coupled Plasma Mass Spectroscopy (EPA 200.7) (EPA 200.8)"

17.7 Microbac SOP ME406 "Microwave Digestion of Sediments, Sludges, Soils and Oils (3051)".

17.8 Microbac SOP ME407 "Microwave Digestion – Aqueous SW846-3015".

17.9 Microbac SOP 33 "Laboratory Waste Management"

17.10 Microbac SOP GP-CAPA "Corrective Action/Preventive Action: Initiating, Tracking and Monitoring"

17.11 Microbac SOP GP-RCA "Root Cause Analysis"

17.12 Microbac SOP KAVS "Analytical method for Determination of Acid Volatile Sulfide (AVS) in Sediment"

17.13 USEPA, 1991. Draft Analytical method for determination of Acid Volatile Sulfide in Sediment, EPA-821-R-91-100. Office of Water, U.S. Environmental Protection Agency, Washington, D.C.

Appendix A

Linear Calibration Algorithm

This calibration curve is established by assuming that the relationship between concentration (the X values) and intensity (the Y values) is linear and that the following equation describes this relationship:

$$Y=MX+B$$

where:

X = concentration
Y = intensity
M = slope of the calibration curve
B = y-axis intercept

Given 2 or more data points, the values for M and B are calculated using the following equations [1, 2].

N = number of standards (includes the blank)

In this equation, the blank is subtracted from all solutions and included in the calculation of the calibration curve.

[1] $$M = \frac{(n)\sum_{n}^{i=1}(X_iY_i) - \sum_{n}^{i=1}(X_i)\sum_{n}^{i=1}(Y_i)}{(n)\sum_{n}^{i=1}(X_i^2) - \left(\sum_{n}^{i-1}X_i\right)^2}$$

[2] $$B = \frac{\sum_{n}^{i=1}(X_i^2)\sum_{n}^{i=1}(Y_i) - \sum_{n}^{i=1}(X_iY_i)\sum_{n}^{i=1}(X_i)}{(n)\sum_{n}^{i=1}(X_i^2) - \left(\sum_{n}^{i-1}X_i\right)^2}$$

Linear Calibration Algorithm (continued)

$$COC = \frac{\sum xy - \frac{\sum x \sum y}{n}}{\sqrt{\left(\sum x^2 - \frac{\left(\sum x\right)^2}{n}\right)\left(\sum y^2 - \frac{\left(\sum y\right)^2}{n}\right)}}$$

where:

x = standard concentration
y = mean intensity
n = number of standards

Weighted Linear Calibration Algorithm

When working at low level concentrations it provides an alternative calibration scheme that weighs the low standards to a greater degree.

Determine the slope, intercept, and correlation coefficient for the equation:

$$y = b_0 + b_i x$$

Where y is the measured net corrected intensity (blank subtracted). The weights are applied by multiplying the intensity by the weighting factor for each standard. In this calibration the weighting factor is the reciprocal of the square of the user entered concentration value for each standard.

where:

x = concentration value of the standard
y = measured intensity of the standard
n = number of standards
i = index for the standards
b_0 = intercept
b_1 = slope
COC = correlation coefficient

$$w_1 = \sum \frac{1}{x_i^2} \qquad w_4 = \sum \frac{y_i}{x_i^2}$$

$$w_2 = \sum \frac{x_i}{x_i^2} \qquad w_5 = \sum \frac{y_i^2}{x_i^2}$$

$$w_3 = \sum \frac{x_i^2}{x_i^2} = n \qquad w_6 = \sum \frac{x_i y_i}{x_i^2}$$

Intercept:

$$b_0 = \frac{(w_4 w_3) - (w_6 w_2)}{(w_1 w_3) - w_2^2}$$

Slope:

$$b_1 = \frac{(w_1 w_6) - (w_4 w_2)}{(w_1 w_3) - w_2^2}$$

Weighted Linear Calibration Algorithm (continued)

Correlation Coefficient: $$COC = \frac{(w_1 w_6) - (w_2 w_4)}{\sqrt{\left[(w_1 w_3) - w_2^{\,2}\right]\left[(w_1 w_5) - w_4^{\,2}\right]}}$$

Appendix B

Simultaneously Extracted Metals (SEM)

Document Control # 265 **Issued to: Document Master File**

SEM are operationally defined as metals (arsenic, cadmium, copper, lead, mercury, nickel, silver and zinc for example) that form sulfides less soluble than those of iron or manganese and which are at least partially released from the sediment under the conditions for generating Acid Volatile Sulfides (AVS) (see Microbac SOP KAVS). Since sulfide is a precipitant of heavy metals under anoxic conditions, the SEM to AVS ratio obtained by the method is an indicator of heavy metal bioavailability in the sediment sample.

AVS Generation and Filtration

AVS is generated in a reaction cell per the procedure in Microbac SOP KAVS. Once sulfide generation is complete the sediment and water from the reaction cell (Microbac SOP KAVS Section 11.10) is vacuum filtered through Whatman TCLP Glass microfiber Filters Acid Treated Low Metal (or equivalent) into a filtration apparatus that has been washed, rinsed in 0.1 M HNO_3 and then DI water rinsed prior to use.

The filtrate is transferred to B-D 60 mL sterile luer-lok syringes equipped with Corning 0.2 um membrane syringe filters (or equivalent) and filtered into an acid washed, DI water rinsed graduated cylinder.

The filter flask is rinsed with DI water and the rinsates are similarly 0.2 um filtered and added to the cylinder.

DI water is then used to bring the filtrate to a final volume of 250 mL. The sample is transferred to a labeled, certified precleaned 250 mL Nalgene bottle C and G Containers (or equivalent) and analyzed within two weeks.

Metal concentrations are determined by ICP-OES.

Batch QA/QC Samples

Method Blank – An aliquot of 4% HCl in DI water is filtered in the same manner as the samples. This filtrate will be used as the method blank.

LCS – In a digestion tube containing about 20 mL 4% HCl spike 5 mL of spike solution (7.25) and bring to a 50 mL final volume with 4% HCl. Filter the LCS in the same manner as the samples.

MS/MSD – If there are no client designated spike samples, choose one sample from the batch. Into two digestion tubes pour approximately 20 mL of the filtered sample. Add 5 mL of spike solution (7.25) and bring to final 50 mL volume with additional filtered sample.

Interference Tests – A post digestion spike will be prepared as in Section 13.8. Additionally, if appropriate, a serial dilution will be analyzed as in 13.7.

Calibration Solutions

Calibration Solutions, ICV and Interference Check Solutions are prepared as in Sections 7.2 through 7.13 and 7.17 through 7.18 except that the diluent used is 4% HCl (7.24).

The working calibration blank solution used is 4% HCl (7.24).

Analysis and Reporting

Metal concentrations are determined by ICP-OES following the procedures in Section 11.0.

The instrument is calibrated as in 10.1 followed by ICV/ICB, ICSA/ICSAB and CCV/CCB analysis. Acceptance criteria are found in Table 13.1. Samples are analyzed in groups of ten or less followed by CCV/CCB analyses. Concentrations of the metals are reported as umole per gram dry sediment (umole/g).

SEM is then calculated as the sum of the metal concentrations:

$$SEM = \sum [metals]$$

Table 1.1

Name	Symbol	Wavelength	Cas Number
Aluminum	Al	308.2	7429-90-5
Antimony	Sb	206.8	7440-36-0
Arsenic	As	189.0	7440-38-2
Barium	Ba	455.4	7440-39-3
Beryllium	Be	313.1	7440-41-7
Boron	B	249.6	7440-42-8
Cadmium	Cd	228.8	7440-43-9
Calcium	Ca	422.6	7440-70-2
Chromium	Cr	267.7	7440-47-3
Cobalt	Co	228.6	7440-48-4
Copper	Cu	224.7	7440-50-8
Iron	Fe	261.1	7439-89-6
Lead	Pb	220.3	7439-92-1
Lithium	Li	670.7	7439-93-2
Magnesium	Mg	279.0	7439-95-4
Manganese	Mn	257.6	7439-96-5
Molybdenum	Mo	202.0	7439-98-7
Nickel	Ni	231.6	7440-02-0
Potassium	K	766.4	7440-09-7
Selenium	Se	196.0	7782-49-2
Silicon	Si	212.4	7440-21-3
Silver	Ag	328.0	7440-22-4
Sodium	Na	589.5	7440-23-5
Strontium	Sr	407.7	7440-24-6
Thallium	Tl	190.8	7440-28-0
Tin	Sn	189.9	7440-31-5
Titanium	Ti	337.2	7440-32-6
Vanadium	V	292.4	7440-62-2
Zinc	Zn	206.2	7440-66-6
Phosphorus	P	214.9	7723-14-0
Zirconium	ZR	339.1	7704-67-7
Calculated			
Hardness, Calculated (as $CaCO_3$)	CaCO3	NA	72608-12-9
Silica (as SiO_2)	SiO_2	NA	99439-28-8

Table 4.1
iCAP 6000/7000 MDLS and Linear Ranges for 6010/200.7

Metal	Linear Dynamic Range (mg/L)	Verified MDL Water (mg/L)	Reporting Limit Water (mg/L)	Verified MDL Soil (mg/Kg)	Reporting Limit Soil (mg/Kg)
Aluminum	810	0.1	0.2	10	20
Antimony	90	0.05	0.1	0.5	1.0
Arsenic	90	0.005	0.01	0.5	1.0
Barium	45	0.005	0.01	0.25	0.5
Beryllium	4.5	0.001	0.002	0.05	0.1
Boron	90	0.05	0.1	2.5	5
Cadmium	9.0	0.0005	0.001	0.05	0.1
Calcium	900	0.25	0.5	25	50
Chromium	45	0.0025	0.005	0.125	0.25
Cobalt	9.0	0.0025	0.005	0.125	0.25
Copper	180	0.0025	0.005	0.5	1.0
Iron	900	0.025	0.1	1.5	3.0
Lead	225	0.005	0.01	0.5	1.0
Lithium	90	0.05	0.1	2.5	5.0
Magnesium	900	0.25	0.5	12.5	25.0
Manganese	27	0.005	0.01	0.25	0.5
Molybdenum	9.0	0.005	0.01	1.5	3.0
Nickel	90	0.01	0.04	1.0	2.0
Potassium	360	0.5	1.0	25	50
Selenium	90	0.01	0.02	0.5	1.0
Silicon	81	0.5	1.0	NA	NA
Silver	4.5	0.005	0.01	0.25	0.5
Sodium	360	0.25	0.5	12.5	25
Strontium	4.5	0.025	0.05	0.25	0.5
Thallium	9.0	0.1	0.2	1.0	2.0
Tin	90	0.25	0.5	12.5	25
Titanium	90	0.015	0.03	1.0	2.0
Vanadium	90	0.005	0.01	0.25	0.5
Zinc	45	0.01	0.02	0.5	1.0
Phosphorus	450	0.5	1.0	25.0	50.0
Zirconium	18	0.025	0.05	1.25	2.5
Silica (as SiO2)	193	1.07	2.14	NA	NA

Table 4.2a
Microbac's Precision & Accuracy for Inorganic Metals Analyses of Groundwater & Solid Waste for 6010B/200.7

Metal	Accuracy, Water (% Recovery)	Precision, Water (% RPD)	Accuracy, Soil (% Recovery)	Precision, Soil (% RPD)
Aluminum	85 - 115	0 - 20	80 - 120	0 - 20
Antimony	85 - 115	0 - 20	80 - 120	0 - 20
Arsenic	85 - 115	0 - 20	80 - 120	0 - 20
Barium	85 - 115	0 - 20	80 - 120	0 - 20
Beryllium	85 - 115	0 - 20	80 - 120	0 - 20
Boron	85 - 115	0 - 20	80 - 120	0 - 20
Cadmium	85 - 115	0 - 20	80 - 120	0 - 20
Calcium	85 - 115	0 - 20	80 - 120	0 - 20
Chromium	85 - 115	0 - 20	80 - 120	0 - 20
Cobalt	85 - 115	0 - 20	80 - 120	0 - 20
Copper	85 - 115	0 - 20	80 - 120	0 - 20
Iron	85 - 115	0 - 20	80 - 120	0 - 20
Lead	85 - 115	0 - 20	80 - 120	0 - 20
Lithium	85 - 115	0 - 20	80 - 120	0 - 20
Magnesium	85 - 115	0 - 20	80 - 120	0 - 20
Manganese	85 - 115	0 - 20	80 - 120	0 - 20
Molybdenum	85 - 115	0 - 20	80 - 120	0 - 20
Nickel	85 - 115	0 - 20	80 - 120	0 - 20
Potassium	85 - 115	0 - 20	80 - 120	0 - 20
Selenium	85 - 115	0 - 20	80 - 120	0 - 20
Silicon	85 - 115	0 - 20	80 - 120	0 - 20
Silver	85 - 115	0 - 20	80 - 120	0 - 20
Sodium	85 - 115	0 - 20	80 - 120	0 - 20
Strontium	85 - 115	0 - 20	80 - 120	0 - 20
Thallium	85 - 115	0 - 20	80 - 120	0 - 20
Tin	85 - 115	0 - 20	80 - 120	0 - 20
Titanium	85 - 115	0 - 20	80 - 120	0 - 20
Vanadium	85 - 115	0 - 20	80 - 120	0 - 20
Zinc	85 - 115	0 - 20	80 - 120	0 - 20
Phosphorus	85 - 115	0 - 20	80 - 120	0 - 20
Zirconium	85 - 115	0 - 20	80 - 120	0 - 20

Table 4.2b
Microbac's Precision & Accuracy for Inorganic Metals Analyses of Groundwater & Solid Waste for 6010C

Metal	Accuracy, Water (% Recovery)	Precision, Water (% RPD)	Accuracy, Soil (% Recovery)	Precision, Soil (% RPD)
Aluminum	80 - 120	0 - 20	80 - 120	0 - 20
Antimony	80 - 120	0 - 20	80 - 120	0 - 20
Arsenic	80 - 120	0 - 20	80 - 120	0 - 20
Barium	80 - 120	0 - 20	80 - 120	0 - 20
Beryllium	80 - 120	0 - 20	80 - 120	0 - 20
Boron	80 - 120	0 - 20	80 - 120	0 - 20
Cadmium	80 - 120	0 - 20	80 - 120	0 - 20
Calcium	80 - 120	0 - 20	80 - 120	0 - 20
Chromium	80 - 120	0 - 20	80 - 120	0 - 20
Cobalt	80 - 120	0 - 20	80 - 120	0 - 20
Copper	80 - 120	0 - 20	80 - 120	0 - 20
Iron	80 - 120	0 - 20	80 - 120	0 - 20
Lead	80 - 120	0 - 20	80 - 120	0 - 20
Lithium	80 - 120	0 - 20	80 - 120	0 - 20
Magnesium	80 - 120	0 - 20	80 - 120	0 - 20
Manganese	80 - 120	0 - 20	80 - 120	0 - 20
Molybdenum	80 - 120	0 - 20	80 - 120	0 - 20
Nickel	80 - 120	0 - 20	80 - 120	0 - 20
Potassium	80 - 120	0 - 20	80 - 120	0 - 20
Selenium	80 - 120	0 - 20	80 - 120	0 - 20
Silicon	80 - 120	0 - 20	80 - 120	0 - 20
Silver	80 - 120	0 - 20	80 - 120	0 - 20
Sodium	80 - 120	0 - 20	80 - 120	0 - 20
Strontium	80 - 120	0 - 20	80 - 120	0 - 20
Thallium	80 - 120	0 - 20	80 - 120	0 - 20
Tin	80 - 120	0 - 20	80 - 120	0 - 20
Titanium	80 - 120	0 - 20	80 - 120	0 - 20
Vanadium	80 - 120	0 - 20	80 - 120	0 - 20
Zinc	80 - 120	0 - 20	80 - 120	0 - 20
Phosphorus	80 – 120	0 – 20	80 – 120	0 - 20
Zirconium	80 – 120	0 – 20	80 – 120	0 - 20

Table 5-1
Interelement Correction Factors for Thermo iCAP 6000

Factors per 100 mg/L Interferant							
Analyte	**Al**	**As**	**Ba**	**Be**	**Ca**	**Co**	**Cr**
Aluminum	--	--	--	--	--	-0.00082	--
Antimony	0.00021	--	--	--	--	--	0.0095
Arsenic	--	--	--	--		--	0.00049
Barium	--	--	--	--	--	--	--
Beryllium	--	--	--	--	--	--	--
Boron	--	--	--	--	--	0.0343	--
Cadmium	--	0.0171	--	--	--	--	--
Calcium	--	--	--	--	--	--	--
Chromium	--	--	--	--	--	--	0.00108
Cobalt	--	--	--	--	--	0.000077	--
Copper	--	--	--	--	--	-0.000093	-0.000172
Iron	--	--	--	--	--	--	--
Lead	-0.000081	--	--	--	--	--	--
Lithium	--	--	--	--	--	--	--
Magnesium	--	--	--	--	--	-0.000092	--
Manganese	--	--	--	--	--	--	--
Molybdenum	--	--	--	--	--	0.00016	--
Nickel	--	--	--	--	--	--	--
Potassium	--	--	--	--	--	--	--
Selenium	-0.00024	--	--	--	--	--	--
Silicon	--	--	--	--	--	--	--
Silver	--	--	--	--	--	--	--
Sodium	--	--	--	--	--	--	--
Strontium	--	--	--	--	--	--	--
Thallium	-0.000012	--	--	--	--	0.00397	0.000276
Tin	--	--	--	--	--	--	--
Titanium	--	--	--	--	--	--	--
Vanadium	--	--	--	--	--	--	--
Zinc	--	--	--	--	--	-0.00074	--
Phosphorus	0.00075	--	--	--	--	--	--
Zirconium	--	--	--	--	--	--	--

Table 5-1
Interelement Correction Factors for Thermo iCAP 6000 (continued)

Factors per 100 mg/L Interferant						
Analyte	**Cu**	**Fe**	**Mg**	**Mn**	**Mo**	**Ni**
Aluminum	--	--	--	--	0.0153	--
Antimony	--	--0.000056	--	--	0.00067	--
Arsenic	--	-0.00025	--	--	0.00109	--
Barium	--	--	--	--	--	--
Beryllium	--	--	--	--	--	--
Boron	--	-0.000398	--	--	-0.00169	--
Cadmium	--	0.000002	--	--	0.000022	-0.000128
Calcium	--	--	--	--	--	--
Chromium	--	-0.000006	--	0.00016	--	--
Cobalt	--	--	--	--	-0.000983	0.000175
Copper	--	0.00021	--	--	0.00274	-0.00455
Iron	--	--	---	--	--	--
Lead	0.000809	0.000021	--	--	-0.00183	0.00011
Lithium	--	--	--	--	--	--
Magnesium	--	--	--	--	--	--
Manganese	--	--	--	--	--	--
Molybdenum	--	--	--	--	--	--
Nickel	--	--	--	--	--	--
Potassium	--	--	--	--	--	--
Selenium	--	--	--	--	0.000156	--
Silicon	--	--	--	--	0.0187	--
Silver	--	--	--	--	-0.000044	--
Sodium	--	--	--	--	--	--
Strontium	--	--	--	--	--	--
Thallium	--	--	--	--	--	--
Tin	--	--	--	--	--	--
Titanium	--	--	--	--	-0.000153	--
Vanadium	--	0.00002	--	--	-0.00778	--
Zinc	--	--	--	--	--	--
Phosphorus	0.002	0.00165	--	--	0.008	--
Zirconium	--	-0.000031	--	--	--	--

Table 5-1
Interelement Correction Factors for Thermo iCAP 6000 (continued)

Factors per 100 mg/L Interferant							
Analyte	**Sb**	**Se**	**Sn**	**Ti**	**Tl**	**V**	**Zn**
Aluminum	--	--	--	--		0.003	--
Antimony	--	--	-0.0084	-0.00099	--	-0.00438	--
Arsenic	--	--	--	--	--	0.000107	--
Barium	--	--	--	--	--	--	--
Beryllium	--	--	--	-0.00033	--	0.00025	--
Boron	--	--	--	--	--	--	--
Cadmium	--	--	--	-.0249829	--	0.000102	--
Calcium	--	--	--	--	--	--	--
Chromium	--	--	--	0.000055	--	0.00002	--
Cobalt	--	--	--	0.00188	--	--	--
Copper	--	--	--	0.000469	--	--	--
Iron	--	--	--	--	--	--	--
Lead	--	--	--	--	--	--	--
Lithium	--	--	--	--	--	--	--
Magnesium	--	--	--	--	--	--	--
Manganese	--	--	--	--	--	--	--
Molybdenum	--	--	--	--	--	-0.00011	--
Nickel	--	--	--	-	--	--	--
Potassium	--	--	--	--	--	--	--
Selenium	--	--	--	-	--	--	--
Silicon	--	--	--	--	--	--	--
Silver	--	--	--	-0.0062	--	--	--
Sodium	--	--	--	--	--	--	--
Strontium	--	--	--	--	--	--	--
Thallium	--	--	--	-0.0017	--	0.0282	--
Tin	--	--	--	-0.0022	--	--	--
Titanium	--	--	--	--	--	--	--
Vanadium	--	--	--	0.000824	--	--	--
Zinc	--	--	--	--	--	--	--
Phosphorus	--	--	--	--	--	-0.005	--
Zirconium	--	--	--	--	--	--	--

Table 7.4.1
Water Matrix
Calibration Standards Concentrations in mg/L for Thermo iCAP 6000/7000

Element	S0	S1	S2	S3	S4	Stock Conc.
Aluminum	0.0	0.08	0.16	10.0	20.0	2000
Antimony	0.0	0.0096	0.0192	1.2	2.4	240
Arsenic	0.0	NA	0.0064	0.4	0.8	80
Barium	0.0	0.008	0.016	1.0	2.0	200
Beryllium	0.0	0.0004	0.0008	0.05	0.1	10
Boron	0.0	NA	0.008	0.5	1.0	100
Cadmium	0.0	0.0004	0.0008	0.05	0.1	10
Calcium	0.0	0.08	0.16	10.0	20.0	2000
Chromium	0.0	0.004	0.008	0.5	1.0	100
Cobalt	0.0	0.0016	0.0032	0.2	0.4	40
Copper	0.0	0.004	0.008	0.5	1.0	100
Iron	0.0	0.032	0.064	4.0	8.0	800
Lead	0.0	0.004	0.008	0.5	1.0	100
Lithium	0.0	0.008	0.016	1.0	2.0	200
Magnesium	0.0	0.08	0.16	10.0	20.0	2000
Manganese	0.0	0.004	0.008	0.5	1.0	100
Molybdenum	0.0	0.008	0.016	1.0	2.0	200
Nickel	0.0	0.004	0.008	0.5	1.0	100
Potassium	0.0	0.4	0.8	50.0	100.0	10000
Selenium	0.0	NA	0.0062	0.4	0.8	80
Silicon	0.0	0.04	0.08	5.0	10.0	1000
Silver	0.0	0.0032	0.0064	0.4	0.8	80
Sodium	0.0	0.4	0.8	50.0	100.0	10000
Strontium	0.0	0.008	0.016	1.0	2.0	200
Thallium	0.0	NA	0.008	0.5	1.0	100
Tin	0.0	0.008	0.016	1.0	2.0	200
Titanium	0.0	0.008	0.016	1.0	2.0	200
Vanadium	0.0	0.008	0.016	1.0	2.0	200
Zinc	0.0	0.008	0.016	1.0	2.0	200
Phosphorus	0.0	0.08	0.16	10.0	20.0	1000
Zirconium	0.0	0.008	0.016	1.0	2.0	1000

*S0 through S4 are used for 6010B and 200.7.
6010C may use S0 and S4 only.

NA = S1 is not used in construction of the calibration curve for this element. The concentration of S2 is at or below the reporting limit for this element.

Table 7.5.1
Initial and Continuing Calibration Standards Concentration in mg/L

Element	ICV/CCV	Stock Concentration
Aluminum	10.0	2000
Antimony	1.2	240
Arsenic	0.4	80
Barium	1.0	200
Beryllium	0.05	10
Boron	0.5	100
Cadmium	0.05	10
Calcium	10.0	2000
Chromium	0.5	100
Cobalt	0.2	40
Copper	0.5	100
Iron	4.0	800
Lead	0.5	100
Lithium	1.0	200
Magnesium	10.0	2000
Manganese	0.5	100
Molybdenum	1.0	200
Nickel	0.5	100
Potassium	50.0	10000
Selenium	0.4	80
Silicon	5.0	1000
Silver	0.4	80
Sodium	50.0	10000
Strontium	1.0	200
Thallium	0.5	100
Tin	1.0	200
Titanium	1.0	200
Vanadium	1.0	200
Zinc	1.0	200
Phosphorus	10.0	1000
Zirconium	1.0	1000

Table 7.6.1
Low Level Calibration Verification Solution Concentrations

Metal		Stock Concentration (mg/L)
Aluminum		20
Antimony		10
Arsenic		1
Barium		1
Beryllium		.2
Boron		10
Cadmium		.1
Calcium		50
Chromium		.5
Cobalt		.5
Copper		.5
Iron		10
Lead		1
Lithium		10
Magnesium		50
Manganese		1
Molybdenum		1
Nickel		2
Potassium		100
Selenium		2
Silicon		100
Silver		1
Sodium		50
Strontium		5
Thallium		20
Tin		50
Titanium		3
Vanadium		1
Zinc		2

Table 7.7.1
ICSA and ICSAB Concentration in mg/L

Element	ICSA	ICSAB	Stock Concentration
Aluminum	250	250	5000
Antimony	--	0.5	100
Arsenic	--	0.25	50
Barium	--	0.25	50
Beryllium	--	0.25	50
Cadmium	--	0.5	100
Calcium	250	250	5000
Chromium	--	0.25	50
Cobalt	--	0.25	50
Copper	--	0.25	50
Iron	100	100	2000
Lead	--	0.5	100
Magnesium	250	250	5000
Manganese	--	0.25	50
Nickel	--	0.5	100
Potassium	--	5	1000
Selenium	--	0.25	50
Silver	--	0.5	100
Sodium	--	5.0	1000
Thallium	--	0.5	100
Vanadium	--	0.25	50
Zinc	--	0.5	100

The final concentration of the aqueous and soil LCS is as follows:

Table 13.1
Quality Control Criteria
Total Metals - ICP
Method 6010B

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial calibration	Daily at beginning of analytical run	COC ≥ 0.995	Investigate, recalibrate
Initial Calibration Verification (ICV)	After calibration	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Verification (CCV)	Minimum every 10 samples	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Initial Calibration Blank (ICB)	After ICV	< RDL < 1/2 RDL < 3 x IDL < MDL (1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank (CCB)	minimum every 10 samples	< RDL < 1/2 RDL < 3 x IDL < MDL (1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB
Method Blank	One per batch (20 samples maximum per batch)	< RDL or < MDL x 2 (1) <LLOQ	Stop analysis, investigate, reanalyze. If still > limit re-digest batch or qualify data and address in narrative
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One per batch (20 samples maximum per batch)	Control Limits 80 - 120% Soil 85 – 115 % Water	Stop analysis, investigate, reanalyze. If still outside limits re-digest batch or qualify data and address in narrative
Matrix spike/ Matrix Spike Duplicate (MS/MSD)	One per batch (20 samples maximum per batch)	80 - 120% recovery RPD ≤ 20%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified
Duplicate (DP)	One per batch (20 samples maximum per batch)	RPD ≤ 20%	Qualify data and address in narrative if client specified
ICP interference check Samples (ICSA/ICSAB)	Run at beginning of each run	80 - 120% of true value for EPA check sample element. < RL or project specific criteria for nonspiked elements	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Post digestion spike (PS)	5%, or minimum of 1 per batch	75 - 125% recovery	Serial dilution if applicable or dilute and repeat post digestion spike.
Serial Dilution (DL)	If post digestion spike fails	± 10% of original determination	Dilute and repeat Post digestion spike

(1) Acceptance criteria are analyte specific and instrument specific

Table 13.2
Quality Control Criteria
Total Metals - ICP
Method 200.7

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA (1)	CORRECTIVE ACTION
Initial calibration	Daily at beginning of analytical run	COC ≥ 0.995	Investigate, recalibrate
Initial Calibration Verification (ICV)	After calibration	95 – 105% RSD < 3%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Verification (CCV)	Minimum every 10 samples	90 – 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Initial Calibration Blank (ICB)	After ICV	< RDL < 1/2 RDL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank (CCB)	minimum every 10 samples	< RDL < 1/2 RDL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB
Method Blank	One per batch (20 samples maximum per batch)	< RDL or <MDL	Stop analysis, investigate, reanalyze. If still > limit re-digest batch or qualify data and address in narrative
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One per batch (20 samples maximum per batch)	Water 85 – 115 % (STD) 80 – 120 % (DoD) Soil 80-120 %	Stop analysis, investigate, reanalyze. If still outside limits re-digest batch or qualify data and address in narrative
Matrix spike/Matrix Spike Duplicate (MS/MSD)	One per ten samples (20 samples maximum per batch)	80- 120% recovery RPD ≤ 20%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified
Duplicate (DP)	One per batch (20 samples maximum per batch)	RPD ≤ 20%	Qualify data and address in narrative if client specified
ICP Interference Check Samples (ICSA/ICSAB)	Run at beginning of each run	Spiked Elements 80 – 120 % Nonspiked Elements <RL (STD) < 2 x MDL (DoD)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Post digestion spike (PS)	5%, or minimum of 1 per batch	75 – 125% recovery	Serial dilution if applicable or dilute and repeat post digestion spike.
Serial Dilution (DL)	If post digestion spike fails	± 10% of original determination	Dilute and repeat Post digestion spike

(1) Acceptance criteria are project specific. Consult QAPP

Table 13.3.2
Final Concentration of the Aqueous and Soil LCS

ELEMENT	WATER mg/L	SOIL mg/Kg
Potassium Sodium	25	1250
Aluminum Calcium Magnesium	5	250
Silicon	2.5	NA
Iron	2	100
Antimony	0.6	30
Barium Lithium Molybdenum Strontium Titanium Vanadium Zinc Phosphorus Zirconium Tin	0.5	25
Boron Chromium Copper Manganese Nickel Lead Thallium	0.25	12.5
Arsenic Selenium Silver	0.2	10
Cobalt	0.1	5
Beryllium Cadmium	0.025	1.25

Table 13.4
Quality Control Criteria
Total Metals – ICP
Method 6010C

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration	Daily, at beginning of run	COC ≥ 0.998	Investigate, recalibrate.
Initial Calibration Verification (ICV)	After calibration	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Continuing Calibration Verification (CCV)	After ICSA/ICSAB and minimum every 10 samples	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, and reanalyze all samples analyzed after last compliant CCV.
Initial Calibration Blank (ICB)	After ICV	< RL ½RL 3X IDL 2X MDL(1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Continuing Calibration Blank (CCB)	After every CCV, minimum every 10 samples	< RL ½RL 3X IDL 2X MDL(1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, and reanalyze all samples analyzed after last compliant CCB.
Low Level Initial Calibration Verification (LLICV)	After ICB	70 - 130%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Low Level Continuing Calibration Verification (LLCCV)	Minimum following closing batch CCB	70 - 130%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, and reanalyze all samples analyzed after last compliant LLCCV.
Method Blank	Once per batch (20 samples maximum per batch)	< RL ½RL 2X MDL(1) <LLOQ	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch or qualify data and address in narrative
Laboratory Control Sample /Laboratory Control Sample Duplicate (LCS/LCSD)	Once per batch (20 samples maximum per batch)	80 - 120%	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch or qualify data and address in narrative
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	Once per batch (20 samples maximum per batch)	75 - 125%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified.
Duplicate (DP)	Once per batch (20 samples maximum per batch) upon client request	RPD ≤ 20%	Qualify data and address in narrative if client specified.
ICP Interference Check Samples (ICSA/ICSAB)	After ICB, prior to sample analysis	80 - 120% for spiked analytes; < RL or other project specific criteria for nonspiked analytes	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Post Digestion Spike (PS)	One per batch	80 - 120%	Serial dilution if applicable or dilute and repeat post digestion spike.
Serial Dilution (DL)	One per batch, if sample concentration > 50X MDL	± 10% of original determination	Dilute and repeat post digestion spike.

(1) Acceptance criteria are analyte specific and instrument specific.

Figure 12.1

Example 6010 Calculations
Thermo Scientific iCAP 6500

1.0 Initial Calibration (ICAL) Parameters
For a multi-point calibration, the system performs linear regression from data consisting of a blank and four standards.

2.0 Calculating the concentration (C) of an element in water using data from prep log, run log, and quantitation report (NOTE: the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Vi} \times D$$

Where:	**Example:**
Cs = Concentration computed by the data system in ug/mL (ppm)	0.08
Vf = Final volume (mL)	50
Vi = Initial volume (mL)	40
D = Dilution factor as a multiplier (10X = 10)	1
Cx = Concentration of element in ug/mL (mg/L)	0.1

3.0 Calculating the concentration (C) of an element in soil using data from prep log, run log, and quantitation report (NOTE: the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Vi} \times D$$

Where**:**	**Example:**
Cs = Concentration computed by the data system (mg/L) (ppm)	0.1
Vf = Final volume (mL)	50
Vi = Initial weight (g)	1
D = Dilution factor as a multiplier (10X = 10)	1
Cx = Concentration of element in ug/g (mg/kg)	5

4.0 Adjusting the concentration to dry weight:

$$Cdry = \frac{Cx \times 100}{Px}$$

Where**:**	**Example:**
Cx = Concentration calculated as received (wet basis)	5
Px = Percent solids of sample (% wt)	80
Cdry = Concentration calculated as dry weight (mg/kg)	6.25

Figure 14.1

Checklist ID: 79093

Microbac Laboratories Inc.

Data Checklist

Date: 16-AUG-2013
Analyst: QX
Analyst: NA
Method: 6010B/6010C/200.7
Instrument: ICP-THERMO2
Curve Workgroup: 441689
Runlog ID: 55099
Analytical Workgroups: 441377, 441535, 441656, 441660, 441661, 441677

Calibration/Linearity	X
ICV/CCV	X
ICV RSD <= 3% (EPA 200.7 only)	X
ICB/CCB	X
ICSA/ICSAB	X
CRI	
Blank/LCS	X
MS/MSD	X
Post Spike/Serial Dilution	X
Upload Results	X
Data Qualifiers	
Generate PDF Instrument Data	X
Sign/Annotate PDF Data	X
Upload Curve Data	X
Workgroup Forms	X
Case Narrative	
Client Forms	
Level X	
Level 3	
Level 4	
Check for compliance with method and project specific requirements	X
Check the completeness of reported information	X
Check the information for the report narrative	X
Primary Reviewer	KHR
Secondary Reviewer	SLP
Comments	

Primary Reviewer: 19-AUG-2013

Secondary Reviewer: 19-AUG-2013

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ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)
REFERENCED METHOD:	EPA TO-15
SOP ID:	VOA-TO15
REV. NUMBER:	23.0
EFFECTIVE DATE:	04/30/2016

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STANDARD OPERATING PROCEDURE

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

EPA TO-15

SOP ID:	VOA-TO15	Rev. Number:	23.0	Effective Date:	04/30/2016

Approved By: Wida Ang — Team Leader (VOA GC/MS) - Wida Ang — Date: 4/22/16

Approved By: Chris Parnell — Technical Manager (VOA GC/MS) - Chris Parnell — Date: 4/21/16

Approved By: Chaney Humphrey — QA Manager - Chaney Humphrey — Date: 4/21/16

Approved By: Kelly Horiuchi — Laboratory Director - Kelly Horiuchi — Date: 4/22/16

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TABLE OF CONTENTS

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

1) Scope and Applicability

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

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

2) Summary of Procedure

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

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

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

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

3) Definitions

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

3.21 SOP Standard Operating Procedure

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

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

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

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

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

4) Health and Safety Warnings

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

4.2 Pollution Prevention and Waste Management

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

4.3 This procedure may include CHEMICAL, OPERATIONAL and/or EQUIPMENT hazards. Employees must review and understand the following hazards and their preventive measures prior to proceeding with this activity.

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

Hazard information related to this activity which is not included or referenced in this document, should be immediately brought to the attention of the Department Supervisor.

5) Cautions

5.1 A maintenance log will be kept documenting maintenance performed on each analytical system. The serial numbers of each instrument shall be recorded, and each log entry

must include a description of the maintenance performed and be initialed by the analyst performing or observing/authorizing maintenance by an outside contractor.

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

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

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

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

5.2 Concentrating Trap

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

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

5.3 GC System

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

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

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

5.4 Mass Spectrometer

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

5.5 Instrument Tuning

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

5.6 Computer Troubleshooting

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

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

Basic Troubleshooting Outline:

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

6) Interferences

6.1 Summa Canisters

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

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

Current laboratory practice entails the segregation of 6L canisters into ambient (low) level and source levels. All the ambient canisters are used for low level (indoor air, ambient air) projects and not intentionally for soil gas, SVE monitoring, or other higher

level applications. It may be necessary to "retire" an ambient canister and re-assign for source level use if high concentrations are encountered. This decision will be made by management based on analytical concentrations and what compounds were encountered at these levels. If the level of any analyte is detected above 5,000ug/m*3* in the ambient can, then the supervisor/team leader must be contacted to determine if the canister(s) is to be retired. If retirement is decided upon, make a notation on the sample tag (or other color coded tag) of each canister in question. The notation must contain the analyte, threshold levels and retirement from ambient use (initial and date notation) so that the canister conditioning/management department may properly execute the retirement.

6.2 Analytical System

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

6.3 Carbon Dioxide

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

6.4 Gas Collection Bags

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

6.5 Glassware

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

7) Personnel Qualifications and Responsibilities

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

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

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

7.4 Demonstration of Capability

All analysts must be trained in accordance with the guidelines detailed in the *SOP for Training Policy*. Demonstrations shall also be performed in accordance with the 2009 TNI Standards (Volume 1 Module 4 Section 1.6) and DoD Quality Systems Manual 5.0. Attachment 1 shall be used to document the training plan for new analysts' initial demonstration. Additionally, these demonstrations are performed anytime there is a change in instrument type, personnel or method.

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

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

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

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

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

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

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

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

8) Sample Collection, Handling, and Preservation

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

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

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

9) Equipment and Supplies

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

9.2 Gas Chromatograph (GC)

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

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

9.3 Autosampler

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

9.4 Mass Spectrometer (MS)

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

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

9.4.1 Ionization Gauge Controller

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

9.5 Analytical Column

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

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

  OR

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

9.6 Data Systems

IBM-compatible PC with Windows 95/98/NT/XP/7 (Microsoft Office EXCEL version 2003 or newer) and Hewlett Packard Chemstation software including EnviroQuant with Extracted Ion Current Profile (EICP), National Institute of Standards and Technology (NIST) library (2002 version or newer) or equivalent.

9.7 Canister Pressurization Station

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

9.8 Canister Sampling Devices

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

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

9.9 Gas Collection Devices

- Lab Commerce, Aerosphere Model S6L, 6.0L Summa Passivated Canisters or equivalent
- Lab Commerce, Stabilizer Model 22.4L, 2.4L Canisters or equivalent
- Restek Corporation, #24203, 3.0L Silco Canisters or equivalent

- Tedlar bags – 0.5L, 1L, 3L, 5L, 10L, 25L, and 40L (other sizes are available; however, the volumes that are listed encompass the majority of the bags supplied and the samples submitted to the laboratory).

9.10 Dynamic Dilution System

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

10) Standards and Reagents

10.1 Reagents and Equipment

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

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

10.1.3 UHP/Zero Grade Air (canister pressurization)

10.1.4 ASTM Type II Water, DI water or equivalent

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

10.2 Standards

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

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

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

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

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

Where:

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

Example:

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

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

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

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

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

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

PV = PDF(V) (Equation 2)

Where:

PV Pressurized canister volume (L)

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

P_f Final Canister Pressure

P_i Initial Canister Pressure

V Volume of canister at 1atm

P_{atm} Atmospheric Pressure = 14.7psig

Example:

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

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

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

Where:

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

Example:

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

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

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

IS concentrations are factored out since they appear in the numerator and denominator of the final calculation.

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

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

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

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

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

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

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

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

Step1: Determine the required flow rate of the stock standards (positions #2 and #3). The range must be from 5 to 50sccm (standard cubic centimeters per minute, same as ml/min). The flows listed below are

guidelines to be used for the default standard flow (based on the desired standard concentration) and were chosen based on the ultimate final dilution required and limitations of the Dynamic Diluter (flows must be from 150 to 2000ml/min.).

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

Cocktail Preparation:

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

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

Where:

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

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

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

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

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

Where:

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

Example:

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

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

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

Where:

S Spike amount required in order to obtain the desired concentration (µL)

C_1 Desired concentration of SDB (µg/mL)

C_2 Concentration of cocktail (µg/µL)

V Volume of SDB (L)

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

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

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

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

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

Example:

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

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

10.2.2.2.4 *The Working standard* for extra compounds is prepared in a Summa canister by spiking an aliquot of the intermediate standard (glass SDB or Tedlar bag) using a heated gastight syringe. The preparation of these standards shall follow the

instructions detailed in Section 10.2.1.2. The concentrations for working standards are usually 20 and 200ng/L, however different concentrations can be chosen which work best for a particular project.

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

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

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

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

10.3 Storage and Expiration Dates

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

hour prior to use in preparing working standards. The guidelines for the storage and expiration date for the intermediate calibration standards are stated in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.

- Prepared Stock / Intermediate Calibration Standards prepared in Summa canisters (1000ng/L) may be stored at laboratory conditions for up to three months in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.
- Calibration or Working Calibration Standards prepared in canisters may be stored at laboratory conditions for one month in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

11) Method Calibration

11.1 Initial Calibration

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

Initial calibration requirements are as follows:

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

- Points may not be dropped from the interior of the curve unless an assignable cause (i.e., gross dilution error, missing internal standards, purge malfunction, standard preparation error, or instrument malfunction) is accounted for and documented. In these instances, all the analytes in that calibration standard must be dropped from the calibration curve as the corrective action (the reason must be documented and the results maintained with the documentation for the final ICAL).
- Dropping individual compound points from the upper or lower end of the calibration range to improve linearity is not considered an error correction. The reason for dropping these points does not need to be documented but the ICAL documentation must state the revised calibration range if the MRL must be adjusted or the calibration range is lowered for a particular compound. This must be documented on the ICAL Review Checklist.

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

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

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

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

SCAN

1. Determine if the lower end of the calibration range is to be 0.08ng or 0.4ng on column. If the low end is 0.08ng, then the 1ng/L standard must be utilized.

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

11.1.2 Recalibration Each GC/MS system must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument, if the continuing calibration verification acceptance criteria are not met and at least annually. The following procedure must be followed when updating an initial calibration method.

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

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

11.1.4 Procedure The system should be operated using temperature and flow rate parameters equivalent to those in Section 12.4. Use the standard prepared in accordance with Section 10.2.2 of this SOP. Attach the calibration standard and internal standard/surrogate canisters to the designated inlets on the

preconcentrator and open the canister valves. Analyzing different volume aliquots of the calibration standards produces differing concentrations.

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

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

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

11.1.4.2 LOQ Establishment, Verification and Acceptance Criteria

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

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

Perform a review of specific aspects of the calibration which might compromise data quality such as inappropriate extension of the calibration range with detector saturation and/or a lack of sensitivity for any analyte. Analyte concentrations which do not meet the signal to noise ratio or exhibit saturation are not to be reported and must be eliminated from the initial calibration. These instances should be followed by a short explanation regarding the reason for the omission.

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

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

11.2 Initial Calibration Verification Standard

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

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

12) Sample Preparation/Analysis

12.1 Sample Preparation

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

Depending on the size of the canister and location of sampling and as specified in the SOP below, samples may be pressurized to approximately 1.0psig to 3.5psig. Additional information may be found in the *SOP for Evaluation and Pressurization of*

Specially Prepared Stainless Steel Canisters. Initial and final pressures are recorded in LIMS and should be repeated on the back of the sample tag. The dilution factor created by filling the sample canister is calculated using equation number 12 in Section 15.7.

12.2 Screening

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

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

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

Where:

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

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

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

12.3 Analytical Sequence and Data System Setup

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

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

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

Analytical Sequence Guideline

With Calibration	Tune Check[1] Calibration Standards (5 Standards Minimum) ICV Standard[2] (Acts as the ICV and LCS) QC Canister Checks[6] MB[7] Sample(s) – 1-19 Laboratory Duplicate[4]
With Continuing	Tune Check[1] CCV Standard[5] QC Canister Checks[6] MB[7] LCS[3] MRL Check Standard[8] Sample(s) – 1-19 Laboratory Duplicate[4]

[1] The instrument performance check solution must be analyzed initially and once per 24 hour (or as specified by the project) time period (sequence / tune window) of operation. All analyses for a sequence must be initiated (injected) prior to the expiration of the tune window.

[2] In this scenario, the ICV may also be evaluated as the LCS (differing acceptance criteria).

[3] An LCS shall be analyzed at a rate of 1 in 20 or fewer samples. The LCS is the second source calibration check standard analyzed at the lower end of the calibration curve (below the midpoint).

[4] A laboratory duplicate must be analyzed at a rate of 1 per 20 or fewer samples. The duplicate must be rotated among clients, whenever possible. Also, a duplicate laboratory control sample may be analyzed to assess precision to meet project requirements or due to sample matrix effects.

[5] A CCV must be analyzed at the beginning of every analytical sequence.

[6] Any number of QC check canisters may be analyzed in the sequence to determine a canister cleaning batch or batches acceptability.

[7] Any of the QC Check Canisters may serve as the method blank as long as the minimum requirements detailed in this document are met. A method blank shall be analyzed at a rate of 1 in 20 or fewer samples.

[8] A MRL check standard may be analyzed with each batch of 20 or fewer samples (when an initial calibration is not analyzed within the same batch). Additional information is included in Section 12.15.

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

12.4 Conditions

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

Adsorbent Trap

Set Point: 35°

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

Refocusing Trap

Temperature:	-180˚C
Injection Temp.:	160˚C
Injection Time:	1.0 min

Adsorbent Trap Reconditioning Conditions

Temperature:	265°C
Initial Bakeout:	2 hours or until clean blank is obtained
After each run:	5-8 minutes

Sample Run Time

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

12.4.2 GC/MS System

Optimize GC conditions for compound separation and sensitivity.

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

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

12.5 Instrument Performance Check

Since the BFB tuning compound is included in the internal standard and surrogate standard canister and an autosampler is used, it is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to the

reduction and approval of any data collection. The 24-hour time period for GC/MS instrument performance check and standards calibration (initial calibration or continuing calibration verification criteria) begins at the injection of the BFB, which shall be documented in laboratory records. Upon completion of the successful BFB tune, the tune report must be printed and retained on file for future reference.

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

- Inject 50ng or less (on column)
- Three scans (peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.
- Background subtraction is conducted using a single scan prior to the elution of BFB.
- All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.
- The ion abundance criteria must not be changed from the requirement stated in this document (TO-15 or TO-14A, as requested).

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

12.6 Continuing Calibration Verification Standard

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

12.7 Canister Quality Control Check and Method Blank

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

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

12.7.1 Sampling Systems Section 7.1 and 8.4 of Method TO-15 describe the setup and certification procedure for a specific sampling apparatus that has been used by the EPA for several of its large air monitoring programs. These systems are rarely used for the types of projects that make up the bulk of the laboratory's work. The vast majority of samples analyzed by the laboratory are taken into

Summa canisters either as grab samples or using a simple time integrated sampling device (flow controller), as in Section 8.2.1 of the method, so these procedures are not part of the typical protocol for providing sampling materials to clients. The laboratory has developed an SOP for the cleaning and certification of the materials it provides its clients for obtaining air samples to be analyzed by method TO-15. Refer to the *SOP for Cleaning and Certification of Summa Canisters and Other Specially Prepared Canisters* for additional information.

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

12.8 Laboratory Control Sample

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

12.9 Sample Analysis

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

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

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

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

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

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

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

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

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

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

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

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

Tedlar bag dilution:

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

Direct injection:

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

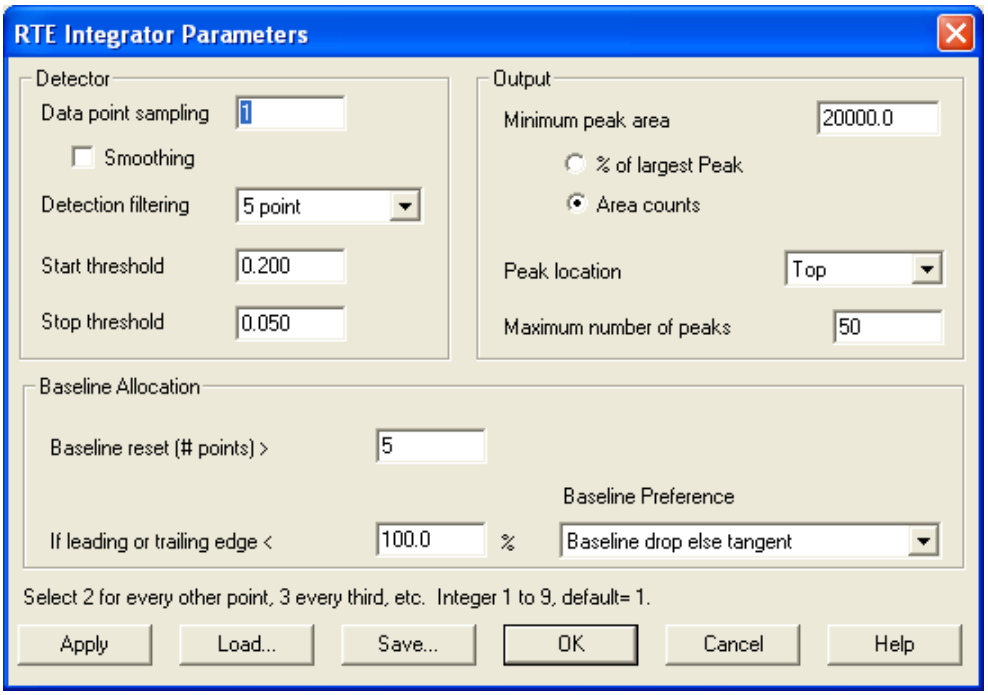
12.9.2 Tentatively Identified Compounds When requested, a mass spectral library search may be made for the purpose of tentatively identifying sample components not associated with the calibration standards. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system mass spectral library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

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

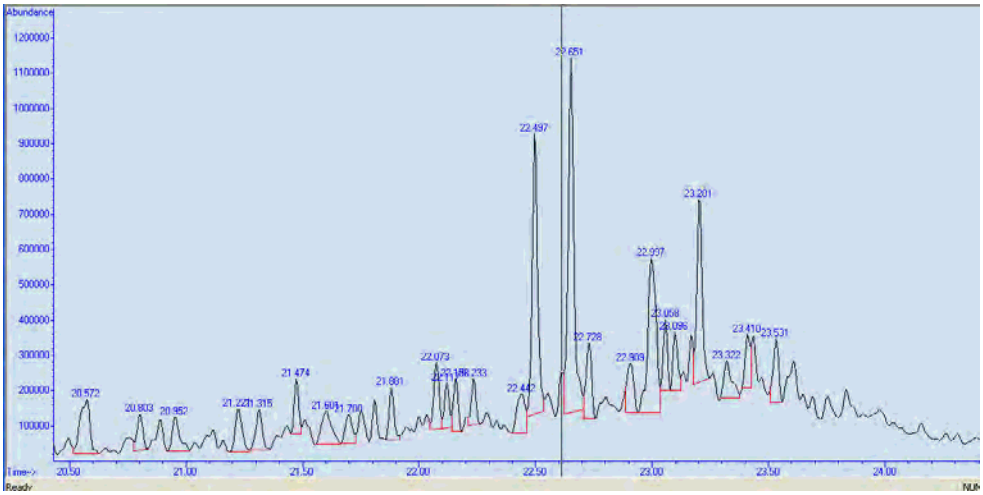
- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20%. For example, for an ion with an abundance of 50% in the standard spectrum, the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

Procedure for Reporting Tentatively Identified Compounds (TICs) for samples and associated Method Blanks

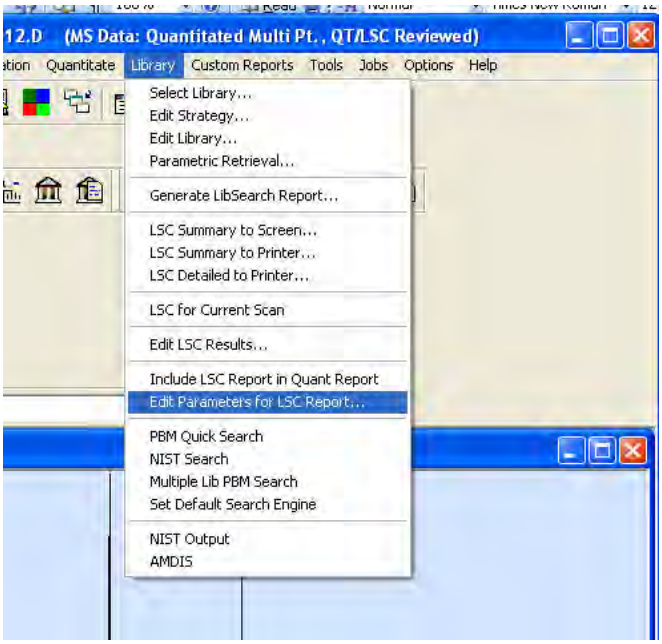
1. Load the datafile in the main Enviroquant window.
2. Load the TIC integration parameters (LSCINT.p). Typical setpoints are as shown below.



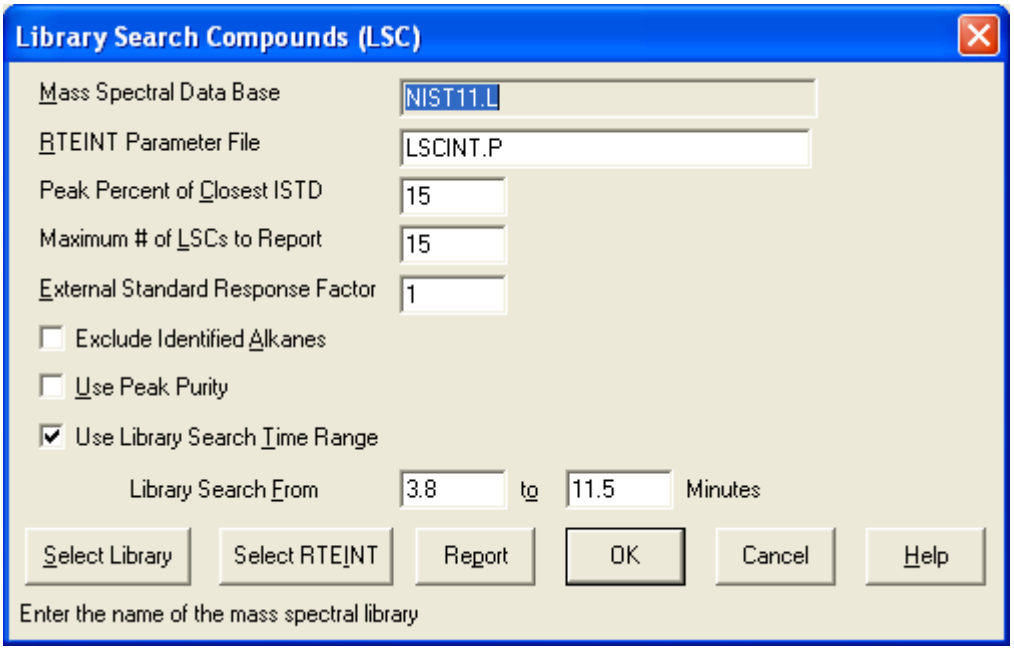
3. Integrate the chromatogram and inspect the peak integrations. Adjust the parameters as needed to achieve integration that will:
   - Resolve closely-eluting peaks that only have a small valley separating them.
   - Not include excess area below the peak in a complex matrix with an elevated baseline.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



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



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



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

12.10 Duplicate

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

12.11 Internal Standard (IS)

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

12.12 Surrogates

Internal standards/surrogates must be added at the same volume for every standard, sample and QC sample. Surrogate compound recoveries are requested by a number of

clients, but are more appropriately used as system monitoring compounds. This is due to the fact that the compounds are introduced directly into the analytical system and not into the canisters or bags. It is for this reason that they are not considered to be true surrogates and a fixed window is applied. Additionally, surrogates are not included in the ICAL because they are not required by the method and are only system monitoring compounds.

12.13 Manual Integration and Q Deletion

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

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

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

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

12.14 Detection Limits and Limits of Detection

The MDL study shall be performed annually for all target analytes on each instrument (with identical configurations) for which this method is performed. The MDL shall be performed in accordance with the procedure outlined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* The detection limit shall be used to determine the LOD for each analyte.

Once determined on each instrument, the highest LOD (for each analyte from all instrument determinations) shall be used as the uniform LOD. However, if a lower detection limit is reported, then the samples must have been run on that specific instrument on which the lower LOD was determined.

12.14.1 Performance and Acceptance Criteria

1. The MDL must be <0.5ppbV for each analyte (Method 11.11.1).
2. Perform Limit of Detection (LOD) verification on all instruments (performing this method) immediately following the MDL study. Spike the LOD at 2-4x the MDL; the spike level establishes the LOD.
3. LOD Acceptance
   - Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio).

- It is specific to each combination of analyte, matrix, method and instrument configuration.
- The LOD must be verified quarterly on each instrument (spiked at LOD) using the criteria listed above.

4. If the LOD verification fails (per #3), repeat the detection limit determination and LOD verification at a higher concentration or perform and pass two consecutive LOD verifications at a higher concentration and set the LOD at the higher concentration.
5. The laboratory shall maintain documentation for all detection limit determinations and LOD verifications (regardless of pass or fail).

12.15 Method Reporting Limit Check Standard

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

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

12.16 Method Modifications

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

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

13) Troubleshooting

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

14) Data Acquisition

14.1 Storing Electronic Data

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

14.2 Sufficient raw data records must be retained on file of all laboratory analyses described in this document including passing QC canister checks, tune checks, instrument calibrations, verifications, sample analyses and dilutions, QC checks, and method

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

Note: All data records must explicitly connect data to the initial instrument calibration. This includes all samples, continuing calibrations and QC samples.

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

15) Calculation and Data Reduction Requirements

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

15.2 Initial Calibration

Tabulate each of the following:

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

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

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

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

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

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

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

N is the number of calibration concentration levels.

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

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

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

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

N total number of calibration concentration levels

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

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

SD Standard Deviation calculated in equation number 3

$\overline{RRF}$ Average or Mean RRF

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

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

RT_C Retention time of the target compound, seconds.

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

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

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

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

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

n Number of calibration levels

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

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

Y_i Area response for the primary quantitation ion for the internal standard for each initial calibration standard.
n number of calibration concentration levels

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

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

$\overline{RT}$ Mean retention time, seconds
RT_i Retention time for the internal standard for each initial calibration standard, seconds.
n number of initial calibration levels

15.3 Continuing Calibration Verification

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

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

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

RRF_x is the RRF from the CCV being evaluated.
$\overline{RRF}$ is the mean RRF from the current calibration curve.

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

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

%R = X/TV x 100

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

15.5 Duplicate Analysis

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

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

x_1 First measurement value
x_2 Second measurement value

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

15.6 Internal Standards (IS)

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

15.7 Pressure Dilution Factor (PDF)

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

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

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

15.8 Results

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

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

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

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

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

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

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

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

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

MW is the molecular weight (Table 2) of the analyte, in g/mole.
24.46 is the molar volume of an ideal gas at 298 K (25 °C) and 760 mmHg (1 atm), in liters per mole (l/mol).

C_x the final analyte concentration in micrograms per cubic meter.

15.8.4 Equation Number 16 – Helium Pressurization (Injection Amount)

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

15.9 Data Review

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

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

15.10 Reporting

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

If the project requires that results be reported below the MRL (LOQ), but above the LOD all of the requirements specified for normal reporting apply (3:1 S/N ratio and ion abundance). This is regardless of the fact that the results will be qualified as estimated.

15.10.1 Analysis Observations / Case Narrative Summary Form

This form, which is included in the *SOP for Laboratory Storage, Analysis and Tracking*, must be generated when there are specific sample composition information or analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved. All data qualifiers and flags should follow those listed in the most recent Quality Assurance Manual or as defined in any client requirements.

This form is necessary as a means for documentation. This form, among other information, will be reviewed when compiling the final report and case narrative. All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through analysis, data reduction, and reporting.

15.10.2 NELAP\TNI Requirements

The following items do not comply with TNI standard requirements and must be reported accordingly. A statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAP certificate of approval.

- Reporting any compound which is not included in the second source standard (ICV or LCS) does not meet NELAP requirements.
- In addition, a report that contains a compound not included on the NELAP certificate of approval must also include the statement listed above.

15.10.2.1 Modifications

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

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

15.10.3 Surrogates

Only report surrogates at the request of the client. If any surrogate is out of control, all samples results (with surrogates requested) associated with the surrogate must be reported with the appropriate data qualifier.

15.10.4 DoD Requirements

Report results with the appropriate data qualifiers, if samples cannot be reanalyzed for any reason. In addition and at a minimum, the following situations are to be noted in the case narrative: manual integrations, CCV out of control, and results exceeding the calibration range.

16) Quality Control, Acceptance Criteria, and Corrective Action

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

16.2 Corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action*, where appropriate. Any maintenance which may alter instrument sensitivity or linearity must result in the re-analysis of the entire sequence including the tune compound, ICAL or CCV or any batch QC.

16.3 Instrument Performance Check

16.3.1 Acceptance Criteria

Refer to Tables 1 and 1A for the required ion abundance criteria.

16.3.2 Corrective Action Perform auto tune or manual tune and then re-analyze BFB. If the BFB acceptance criteria are still not met, the MS must be retuned according to the procedure outlined in the instrument user's manual. Perform necessary maintenance and make notations in the instrument maintenance logbook. It may be necessary to clean the ion source, or quadrupole, or take other necessary actions to achieve the acceptance criteria. An acceptable tune is required for sample results to be calculated and reported.

16.4 Initial Calibration

16.4.1 Acceptance Criteria Refer to the following acceptance criteria for the initial calibration.

- The RRT for each target compound at each calibration level must be within 0.06RRT units of the mean RRT for the compound.
- The calculated %RSD for the RRF for each compound in the calibration standard must be less than 30% with at most two exceptions up to a limit of 40% (this may not be true for all projects).
- For each Internal Standard the area response (Y) at each calibration level must be within 40% of the mean area response $\overline{Y}$ over the initial calibration range.
- The retention time shift for each of the internal standards at each calibration level must be within 20s of the mean retention time over the initial calibration range for each internal standard.
- All of the following information must be retained to permit reconstruction of the initial instrument calibration: calibration date, test method, instrument, analysis date, analyte identification, analyst's initials, concentration and responses, and response factors.
- All initial instrument calibrations must be verified with an acceptable ICV.

16.4.2 Corrective Action Follow the initial calibration requirements detailed in Section 11.1 for information on re-analyzing or dropping points and the restriction of maintenance performed during the analysis of the initial calibration standards.

If the initial calibration results are outside the established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed, if reanalysis of the samples is not possible, data associated with an unacceptable initial calibration shall be reported as estimated with the appropriate data qualifiers.

16.5 Initial Calibration Verification Standard (ICV)

16.5.1 Acceptance Criteria The percent recovery for each compound in the ICV must be between 70%-130% for all analytes except vinyl acetate, which must be within 50-150%. Exceptions to this allowance for the vinyl acetate recovery are project specific requirements and any DoD type project, which shall adhere to the 70-130% requirement for all target compounds.

16.5.2 Corrective Action If the initial calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column. Perform a new initial calibration if any performed maintenance has altered instrument linearity and/or sensitivity. Perform another

initial calibration or if reanalysis is not possible, data associated with an unacceptable ICAL/ICV shall be reported as estimated with the appropriate data qualifiers.

16.6 Continuing Calibration Verification (CCV)

16.6.1 Acceptance Criteria All compounds must be evaluated prior to rounding. The percent difference for each target analyte must be within plus or minus 30% of the initial calibration average RRFs.

16.6.2 Corrective Action If the continuing calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources of the problem and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column.

If any corrective action and/or reanalysis fails to produce continuing calibration verification within acceptance criteria (analyzed immediately following the initial failure), then either two consecutive successful verifications must be performed following corrective action or a new initial calibration must be performed; however, refer to 16.6.2.1 below.

DOD Requirement: If a CCV fails, the laboratory must immediately analyze two additional consecutive CCVs (The two consecutive CCVs must be analyzed within one hour).

- Both of these CCVs must meet acceptance criteria in order for samples to be reported without reanalysis.
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- Corrective action(s) and recalibration must occur if the above scenario fails.
- Flagging data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

16.6.2.1 Method Reporting Limit Check Standard

If a per batch MRL check standard is analyzed due to a failing CCV or client requirement and is unacceptable for any compound (sensitivity; ratio or %D), reanalyze at the same or higher level within the same batch and report data with the CCV flag and case narrative notes accordingly. Reporting data with these conditions must be acceptable per project and client requirements otherwise corrective action must be initiated and samples reanalyzed.

Refer to Section 11.1.4.2 for annual (NELAP and Navy) and quarterly (DoD) LOQ verification requirements.

16.7 Canister Quality Control Check

The actual cleaning procedure, number of cans to select for analysis (to release a cleaning batch) and corrective actions are covered in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* and are not covered in this section. However, the procedure for analyzing and certifying a cleaning batch is included. If a canister passes as a QC canister it meets all of the requirements

for a method blank (Method, TNI Standards, and Department of Defense Quality Systems Manual – DoD QSM, etc.).

16.7.1 Scan Analyses A canister is considered "clean" for normal SCAN analyses if the analysis shows <0.2ppbv of any target analyte (analyte exceptions listed in table below). If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, TNI Standards, and Department of Defense Quality Systems Manual - DoD QSM, etc.).

Low Level SCAN Analyses For those analytes with a MRL of 0.1ug/m3, the QC criteria of <MRL is acceptable; otherwise, <0.2ppbV is required (analyte exceptions listed in table below).

SIM Analyses Results <MRL will be acceptable as this complies with the <0.2ppbV method requirement.

ANALYTE EXCEPTION LIST					
Compounds	ppbV	On Column (ng)	Compounds	ppbV	On Column (ng)
Target Analytes	0.2	0.50	Acrylonitrile	0.2	0.43
Chloromethane	0.2	0.41	Acetone	1.5	3.5
1,3-Butadiene	0.2	0.44	Ethanol	1.9	3.5
Acetonitrile	0.2	0.33	Vinyl acetate	0.99	3.5
Acrolein	0.65	1.5	1-Butanol	0.23	0.70
Isopropanol	0.28	0.70	Carbon Disulfide	1.1	3.5
2-Butanone	1.2	3.5			

Document the status of the check in LIMS and return the canister to the canister conditioning room. Additionally, if the check was found to be acceptable, the quantitation report must be kept on file for future reference

16.7.2 Tentatively Identified Compounds (TIC) If the batch of canisters are to be used for tentatively identified compounds (TIC) analysis, any non-target peaks present in the QC check canister analysis must be evaluated and determined to be less than the TIC reporting limit (10% of the internal standard). The concentration is estimated by assuming a RRF of 1.0 and comparing the response of the TIC to the response of the nearest internal standard.

16.8 Method Blank

16.8.1 Acceptance Criteria

- The concentration of a targeted analyte in the blank cannot be at or above the MRL, AND be greater than 1/10 of the amount measured in any associated sample. For any project that requires reported results less than the MRL, all associated measurements found in the MB should result in a qualifier; however, project requirements may differ and must be followed. Refer to DoD requirements listed below.
- The method blank should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.
- For DoD samples, the method blank will be considered to be contaminated if:

1. The concentration of any target analyte in the blank exceeds 1/2 the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
2. The concentration of any common laboratory contaminant (acetone, ethanol, carbon disulfide, and methylene chloride) in the blank exceeds the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); or
3. The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives.

The laboratory shall evaluate whether reprocessing of the samples is necessary based on the above criteria.

16.8.2 Corrective Action If the analyte concentration results in the blank do not meet the acceptance criteria repeat analysis with remaining QC canisters until results are acceptable or prepare a canister per Section 12.7. If the analyte results in the blank still do not meet the acceptance criteria the source of the problem must be investigated and measures taken to eliminate the source. Each method blank must be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch. Determine whether the contamination is from the instrument or due to contamination in the blank container (if results from the new can are not acceptable then the system is probably contaminated). In all cases, the corrective action (reprocessing or data qualifying codes) must be documented. However, the specific corrective action depends on the type of project the blank is utilized for; therefore, refer (below) to the reporting/reprocessing requirements.

DEPARTMENT OF DEFENSE (DoD) QSM PROJECT: Any sample associated with a blank that fails the criteria shall be reprocessed in the same or subsequent analytical batch, except when the sample analysis resulted in a non-detect. If reanalysis is not performed, the results shall be reported with appropriate data qualifier.

OTHER PROJECT TYPE: Appropriate corrective measures must be taken and documented before sample analysis proceeds. However, if this is not a possibility and the results must be reported follow the reporting requirements stated in Section 18.4.

16.9 Laboratory Control Sample (LCS)

16.9.1 Acceptance Criteria Round all results to the nearest whole number prior to determining if the acceptance criteria have been met. The percent recoveries must be within the laboratory-generated limits and are referenced in the electronic TO-15 Method Manual. However, Arizona requires the percent recovery for each compound in the LCS to be 70%-130% (to match the ICV requirement). Therefore, the ICV exception for vinyl acetate stated in Section 16.5 requires the percent recovery for AZ samples to be 50-150%.

Note: Client project requirements, AFCEE and DoD requirements shall take precedence over the AZ requirement for AZ samples. Meaning if a sample is collected for a DoD project in AZ, DoD requirements specified in this document and the project specific QAPP (if supplied) are to be followed.

DoD Requirement: In the absence of client specified LCS reporting criteria, the LCS control limits outlined in the DoD QSM 5.0 Appendix C tables shall be used when reporting data for DoD projects.

16.9.2 Corrective Action If the LCS criteria are not met, determine whether the cause is instrumentation or the result of a poor injection. If the problem is instrumentation, perform maintenance and if the problem is with the injection re-analyze the LCS. DoD considers the same analyte exceeding the LCS control limits two out of three consecutive LCS to be indicative of non-random behavior; therefore, this trend should be monitored and the appropriate corrective action taken when it occurs.

16.10 Sample Results

16.10.1 Acceptance Criteria

- Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.
- The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, initial calibration verification technical acceptance criteria described in this document.
- All target analyte peaks must be within the initial calibration range, diluted or reported with the appropriate data qualifier.

16.10.2 Corrective Action

- If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis as needed.
- If the area for any internal standard changes by more than ±40 percent between the sample and the most recent calibration, check for possible matrix interferences and re-analyze at a greater dilution. If the requirement is still not met and matrix interference is not detected the GC/MS system must be inspected for malfunction and maintenance made where necessary.
- When corrective actions are made, samples analyzed while the instrument was not functioning properly must be re-analyzed or the appropriate data qualifiers must be attached to the results.

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

16.11 Laboratory Duplicate

16.11.1 Acceptance Criteria The relative percent difference must fall within ±25%. This RPD criterion also applies to duplicate laboratory control samples (DLCS).

16.11.2 Corrective Action If the duplicate results do not meet the technical acceptance criteria, perform another duplicate analysis. If the results are still unacceptable and the associated samples are not reanalyzed then all of the sample results in the associated batch must be flagged accordingly.

16.12 Internal Standards

16.12.1 Acceptance Criteria The following acceptance criteria must be applied to each run (except the ICAL – see Section 16.4).

- The area response for each internal standard in the blank must be within ±40 percent of the area response for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).
- The retention time for each internal standard must be within ±0.33 minutes of the retention time for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).

16.12.2 Corrective Action

- Internal Standard Responses If the problem is with the instrument, perform maintenance. If the problem is with a sample, check for interferences. If the response is high, it is likely that interference is present. In this case, lower the volume or aliquot of the sample and re-analyze. If the problem persists, report the results with the best quality and qualify the results. If the problem is corrected with the lower volume analysis, report those results.
- Internal Standard Retention Times If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis where required.

16.13 Surrogates

16.13.1 Acceptance Criteria Since the matrix precludes the use of true surrogates and there is no established method criterion, acceptable surrogate recoveries are based on a fixed window of 70 - 130%. This is the typical requirement from clients. Additionally, these limits are referenced in SW-846 for use as guidance in evaluating recoveries. These limits are sufficient for evaluating the effect indicated for the individual sample results.

16.13.2 Corrective Action Poor surrogate recovery should be followed by re-analyzing a smaller aliquot to mitigate any matrix interferences. Evaluate the out of control surrogate for the effect on individual sample results.

16.14 Method Reporting Limit Check Standard

16.14.1 Acceptance Criteria Per client requirements or if the CCV is biased low for any compound, then evaluate the MRL check standard. Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio). A percent difference +/-50% is recommended but program and client specific requirements must be followed if applicable.

16.15 Sample Holding Time Expired

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

17) Data Records Management

17.1 All data resubmittal forms and job documentation including Service Requests, Chain of Custody forms, Sample Acceptance Check forms and hardcopy electronic mail

messages must be filed in the project file. Final reports, revised reports, and final invoices are stored electronically.

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

18) Contingencies for Handling Out of Control Data

18.1 The following is specific information on how to report unacceptable data. If the data requires a data qualifier flag, as specified in this SOP, refer to Appendix D of the most recent version of the Quality Assurance Manual for the appropriate data qualifier.

18.2 Initial Calibration and/or Initial Calibration Verification

All results reported with an unacceptable ICAL must be reported as estimated and all data shall be reported using defined qualifiers or flags or explained in the case narrative accordingly.

18.3 Continuing Calibration Verification

All results associated with an unacceptable CCV (other than #1 below) must be reported with the appropriate data qualifier, flag and/or explained in the case narrative.

1. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported without a qualifier.
2. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples with detects, then those detects must be reported with a qualifier, flag and/or explained in the case narrative.
3. If however, the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, and there are associated samples that are non-detects, then those non-detects must be reported with qualifiers, flags and/or explained in the case narrative as having less certainty. However, along with the data qualifiers, the case narrative may include information stating the fact that the results were not significantly affected if:
   a. *An MRL check standard was analyzed and found to be acceptable. The MRL must be the same as that analyzed in the MRL check standard for those analytes that were biased low in the CCV. Adjust MRLs (if required), flag data and state the certainty in the case narrative where the sensitivity of the instrument was demonstrated at the MRL; therefore, results were not significantly affected.*
   b. *With the reporting limit adjusted to the next level in the calibration curve (typically 5 times higher) to prove the nonexistence of a false negative and note procedure in case narrative.*
4. If the acceptance criteria was exceeded (biased high) for the CCV and there were detectable results in a sample, the results may be "qualified" if the results exceeded the regulatory/decision limit (this is to be stated in the case narrative along with the data qualifiers or flags).
5. Data associated with a biased low CCV may be fully useable if the results reported exceed a maximum regulatory limit/decision level.

18.4 Method Blank

- If an analyte in the blank is found to be out of control and the analyte is also found in associated samples, those sample results shall be "flagged" in the report and the method blank results reported.
- If the analyte is found in the blank but not in the sample then the results for the sample may be reported without a qualifier.

18.5 Laboratory Control Sample

All results associated with an out of control laboratory control sample must be reported with the appropriate data qualifier. An indication of whether the LCS was out high or low should also be included.

18.6 Surrogate

Report sample results with the appropriate data qualifier.

18.7 Laboratory Duplicate

All batch sample results associated with an out of control laboratory duplicate must be flagged with the appropriate data qualifier.

18.8 Internal Standard

All target analytes associated with an out of control internal standard must be flagged with the appropriate data qualifier.

18.9 Estimated Sample Results

18.9.1 Sample Hold Time All occurrences of missed holding times must be included on the final report including those samples received and/or analyzed outside of the specified hold times detailed in this SOP.

18.9.2 Matrix Interference Sample data associated with matrix interference must be flagged with the appropriate data qualifier.

18.9.3 Results Outside Initial Calibration Range All sample results not bracketed by initial calibration standards (within calibration range) must be reported as having less certainty by reporting with the appropriate data qualifier.

19) Method Performance

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

19.2 Method Detection Limit (MDL)

The procedure used to determine the method detection limits are as stated in the *Code of Federal Regulations* (40 CFR 136 Appendix B) as defined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations are listed in Tables 2 and 2A for both SCAN and SIM modes and were obtained using spiked canisters prepared with humidified zero air, making at least seven replicate measurements of the compounds of interest, computing the standard deviation, and multiplying this value by the appropriate Student's t value for 99 percent confidence. The MDL actually achieved in a given analysis will vary depending on instrument

sensitivity and matrix effects. All MDLs, regardless of the mode of operation, meet the method performance criteria of <0.5ppbV.

19.3 Accuracy and Precision

Refer to Section 11.4 in the referenced method for information on replicate precision criteria for method performance. Single laboratory accuracy is presented as the second source initial calibration verification standard, which meets the method performance criteria of 30%. Additionally, laboratory generated control limit data for LCSs are presented for the analytes of interest and may be referenced in the electronic TO-15 Method Manual. Refer to Section 11.1.4.2 for the accuracy and precision requirements for concentrations at the LOQ/MRL.

19.4 Selectivity

Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multidetector arrangement of these (see discussion in Compendium Method TO-14A). The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification.

It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. Upon sample injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least ten scans per eluting chromatographic peak must be acquired. Scanning also allows identification of unknown compounds in the sample by searching through library spectra.

The sample analysis using the GC/MS is based in part on a combination of retention times and relative abundances of selected ions. The retention time of each chromatographic peak should be ±0.10 minutes of the library/reference retention time of the compound. The acceptance level for relative abundance should be set at ±20% of the expected abundance. The data should be manually examined by the analyst to determine the reason for the # flag [(#) = qualifier out of range], if present and whether the compound should be reported as found or if there is matrix interference. A background subtraction may aid in this determination. Manual inspection of the qualitative results should also be performed to verify concentrations outside the expected range.

Specific selectivity information is provided in this section and document (such as relative retention time) as well as in the referenced method. Refer to the method for additional information on selectivity.

- Use NIST Library 98 or newer version
- The *reference spectra updates* must be performed with every new ICAL utilizing the mid-level standard (minimum). If needed, the reference spectra may be updated sooner with the continuing calibration standard.
- *Retention time updates* must be performed using EasyID and not by updating to the method (InitCal \ Update Calibration). Refer to the Help selection of the software.

19.5 Demonstration of Capability

This laboratory has continuously performed this method since before July 1999. Therefore, ongoing demonstration of capable shall be performed and documented; however, the initial demonstration of method capability is not required.

19.6 Proficiency Testing (PT) Program

The laboratory shall participate in an air and emissions PT study for TO-15. The testing shall be performed in accordance with this document and meet the frequency and proficiency requirements detailed in the DoD QSM Version 5.0.

20) Summary of Changes

Table 20.1

Revision Number	Effective Date	Document Editor	Description of Changes
23.0	04/30/16	C. Humphrey	10.2.1.3 – Added 2nd and 3rd paragraph
			10.3 – 4th bullet – added last two sentences
			11.1 – Number 13 (5th bullet) – added 2nd paragraph
			11.1.4.2 – Revised; aligned with corporate SOP CE-QA011
			12.7 – First paragraph – added last sentence
			12.9.1 – Revised 2nd and 3rd sentences to match laboratory procedures
			12.9.2 – Added procedure for reporting Tentatively Identified Compounds (TICs); included requirement to follow procedure for samples and associated Method Blanks
			12.16 – Revised to update references to NELAC to TNI Standards and NELAP
			15.1 – Revised to remove reference to NELAC
			15.10 – Revised to remove reference to NELAC
			15.10.2 – Revised to remove references to NELAC
			15.10.2.1 – Revised to remove references to NELAC
			16.6.2 – Moved DoD Requirement Section (previously Section 16.6.3) into section
			16.6.2.1 – Revised to add clarification
			16.7 – Revised to remove reference to NELAC
			16.7.1 – Revised to remove reference to NELAC
			16.14.1 – Revised last sentence
			18.3 – Added #5
			21.5 – Revised to remove reference to NELAC
			Tables 3, 3A, 4, 4A – updated
			Attachments 2 and 3 – updated instruments on checklists
			Attachment 3 – Sample Review Checklist – Revised #7 to include MB reporting requirement for TICS
			Attachment 4 – Revised to remove reference to NELAC

21) References and Related Documents

21.1 EPA Method TO-14A, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.2 EPA Method TO-15, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.3 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, January 1999.

21.4 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Addendum, January 17, 2002.

21.5 2009 TNI Standards

21.6 *Preparation of Gas Phase Standards for Ambient Air Analysis,* Tekmar-DOHRMANN Application Note, Spring 96, Vol. 6.5.

21.7 *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 5.0, July 2013.

21.8 Arizona Administrative Code, Title 9. Health Services, Chapter 14. Department of Health Services Laboratories, December 31, 2006.

21.9 Florida Department of Environmental Protection, Chapter 62-160.

21.10 Minnesota Department of Health, 4740.2065, *Standard Operating Procedures*, Statutory Authority: MS s 144.97; 144.98; History: 31 SR 446, Posted: October 09, 2006, Revised April 16, 2010.

22) Appendix

22.1 Tables

Table 1: Instrument Tune Check Ion Abundance Criteria (TO-15)

Table 1A: Instrument Tune Check Ion Abundance Criteria (TO-14A)

Table 2: Volatile Organic Compounds, EPA Compendium Method TO-15 (SCAN)

Table 2A: Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)

Table 3: Standard Concentrations (SCAN) (Primary Sources)

Table 3A: Standard Concentrations (SIM) (Primary Sources)

Table 4: Standard Concentrations (SCAN) (Secondary Sources)

Table 4A: Standard Concentrations (SIM) (Secondary Sources)

22.2 Attachments

Attachment 1 – Training Plan

Attachment 2 – Initial Calibration Checklist

Attachment 3 – Daily QC and Sample Review Checklists

Attachment 4 – State and Project Specific Requirements

TABLE 1

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-15

Mass	Ion Abundance Criteria[1]
50	8.0 to 40.0 percent of m/e 95
75	30.0 to 66.0 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5.0 to 9.0 Percent of m/e 95
173	Less than 2.0 Percent of m/e 174
174	50.0 to 120.0 Percent of m/e 95
175	4.0 to 9.0 Percent of m/e 174
176	93.0 to 101.0 Percent of m/e 174
177	5.0 to 9.0 Percent of m/e 176

[1] All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

TABLE 1A

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-14A

Mass	Ion Abundance Criteria
50	15 to 40 percent of m/e 95
75	30 to 60 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5 to 9 Percent of m/e 95
173	Less than 2 Percent of m/e 174
174	>50 Percent of m/e 95
175	5 to 9 Percent of m/e 174
176	>95 and <101 Percent of m/e 174
177	5 to 9 Percent of m/e 176

Note: The criteria listed in Tables 1 and 1A shall be met or exceeded in order for EPA Compendium Methods TO-15 or TO-14A to be referenced.

TABLE 2 - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Bromochloromethane (IS1)	74-97-5	-	-	130	128, 132	-	-	-
Propene	115-07-1	42.08	NA	42	39,41	0.50	0.14	IS1
Dichlorodifluoromethane (CFC 12)	75-71-8	120.9	1.329	85	87, 101, 103	0.50	0.17	IS1
Chloromethane	74-87-3	50.49	0.911	50	52	0.50	0.15	IS1
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	76-14-2	170.9	1.455	135	137	0.50	0.19	IS1
Vinyl Chloride	75-01-4	62.50	0.9106	62	64	0.50	0.17	IS1
1,3-Butadiene	106-99-0	54.09	0.6149	54	39, 53	0.50	0.22	IS1
Bromomethane	74-83-9	94.94	1.6755	94	96	0.50	0.19	IS1
Chloroethane	75-00-3	64.52	0.8902	64	66	0.50	0.17	IS1
Ethanol	64-17-5	46.07	0.7893	45	46	5.0	0.80	IS1
Acetonitrile	75-05-8	41.05	0.7857	41	40	0.50	0.18	IS1
Acrolein	107-02-8	56.06	0.840	56	55	2.0	0.17	IS1
Acetone	67-64-1	58.08	0.7845	58	43	5.0	0.77	IS1
Trichlorofluoromethane	75-69-4	137.4	NA	101	103	0.50	0.17	IS1
Isopropyl Alcohol	67-63-0	60.10	0.7809	45	43	5.0	0.42	IS1
Acrylonitrile	107-13-1	53.06	0.8060	53	52	0.50	0.17	IS1
1,1-Dichloroethene	75-35-4	96.94	1.213	96	61	0.50	0.17	IS1
tert-Butanol	75-65-0	74.12	0.7887	59	57,41,43	1.0	0.33	IS1
Methylene Chloride	75-09-2	84.94	1.3266	84	49	0.50	0.17	IS1
Allyl Chloride	107-05-1	76.53	0.9376	41	76	0.50	0.16	IS1
Trichlorotrifluoroethane	76-13-1	187.38	1.5635	151	101	0.50	0.17	IS1

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Carbon Disulfide	75-15-0	76.14	1.2632	76	78	5.0	0.15	IS1
trans-1,2-Dichloroethene	156-60-5	96.94	1.2565	61	96	0.50	0.19	IS1
1,1-Dichloroethane	75-34-3	98.96	1.1757	63	65	0.50	0.16	IS1
Methyl tert-Butyl Ether	1634-04-4	88.15	0.7402	73	57	0.50	0.17	IS1
Vinyl Acetate	108-05-4	86.09	0.9317	86	43	5.0	0.65	IS1
2-Butanone (MEK)	78-93-3	72.11	0.7999	72	43	5.0	0.21	IS1
cis-1,2-Dichloroethene	156-59-2	96.94	1.2837	61	96	0.50	0.16	IS1
Diisopropyl Ether	108-20-3	102.18	0.7241	87	45,59,43	0.50	0.19	IS1
Ethyl Acetate	141-78-6	88.106	0.9003	61	70	1.0	0.35	IS1
n-Hexane	110-54-3	86.18	0.6548	57	86	0.50	0.15	IS1
Chloroform	67-66-3	119.4	1.4832	83	85	0.50	0.17	IS1
1,2-Dichloroethane-d4(S)	17060-07-0	-	-	65	67	-	-	IS1
Tetrahydrofuran	109-99-9	72.11	0.8892	72	71,42	0.50	0.20	IS1
Ethyl tert-Butyl Ether	637-92-3	102.176	0.7519	87	59,57	0.50	0.18	IS1
1,2-Dichloroethane	107-06-2	98.96	1.2351	62	64	0.50	0.16	IS1
1,4-Difluorobenzene(IS2)	540-36-3	-	-	114	88	-	-	-
1,1,1-Trichloroethane	71-55-6	133.4	1.3390	97	99, 61	0.50	0.17	IS2
Isopropyl acetate	108-21-4	102.13	0.8718	61	87,43	1.0	0.32	IS2
1-Butanol	71-36-3	74.1224	0.8098	56	41	1.0	0.48	IS2
Benzene	71-43-2	78.11	0.8765	78	77	0.50	0.16	IS2
Carbon Tetrachloride	56-23-5	153.8	1.5940	117	119	0.50	0.15	IS2

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Cyclohexane	110-82-7	84.16	0.7739	84	69,56	1.0	0.29	IS2
tert-Amyl Methyl Ether	994-05-8	102.176	0.7703	73	87,55,43	0.50	0.15	IS2
1,2-Dichloropropane	78-87-5	113	1.1560	63	62	0.50	0.16	IS2
Bromodichloromethane	75-27-4	163.8	1.980	83	85	0.50	0.15	IS2
Trichloroethene	79-01-6	131.4	1.4642	130	132	0.50	0.14	IS2
1,4-Dioxane	123-91-1	88.11	1.0337	88	58	0.50	0.16	IS2
Isooctane	540-84-1	114.23	0.6877	57	41	0.50	0.15	IS2
Methyl Methacrylate	80-62-6	100.12	0.944	100	69	1.0	0.31	IS2
n-Heptane	142-82-5	100.2	0.6837	71	57,100	0.50	0.17	IS2
cis-1,3-Dichloropropene	10061-01-5	111	1.224	75	77	0.50	0.14	IS2
4-Methyl-2-Pentanone	108-10-1	100.2	0.7965	58	85	0.50	0.16	IS2
trans-1,3-Dichloropropene	10061-02-6	111	1.217	75	77	0.50	0.16	IS2
1,1,2-Trichloroethane	79-00-5	133.4	1.4397	97	83	0.50	0.16	IS2
Chlorobenzene-d5(IS3)	3114-55-4	-	-	82	117	-	-	-
Toluene-d8(S)	2037-26-5	-	-	98	100	-	-	IS3
Toluene	108-88-3	92.14	0.8669	91	92	0.50	0.17	IS3
2-Hexanone	591-78-6	100.16	0.8113	43	58	0.50	0.16	IS3
Dibromochloromethane	124-48-1	208.3	2.451	129	127	0.50	0.16	IS3
1,2-Dibromoethane	106-93-4	187.9	2.1791	107	109	0.50	0.16	IS3
n-Butyl Acetate	123-86-4	116.16	0.8825	43	56, 73	0.50	0.16	IS3
n-Octane	111-65-9	114.23	0.6986	57	114	0.50	0.18	IS3

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Tetrachloroethene	127-18-4	165.8	1.6227	166	164	0.50	0.14	IS3
Chlorobenzene	108-90-7	112.6	1.1058	112	114	0.50	0.16	IS3
Ethylbenzene	100-41-4	106.2	0.8670	91	106	0.50	0.16	IS3
m-, p-Xylenes	179601-23-1	106.2	0.8642, 0.8611	91	106	1.0	0.30	IS3
Bromoform	75-25-2	252.8	2.899	173	175	0.50	0.15	IS3
Styrene	100-42-5	104.1	0.9060	104	78, 103	0.50	0.15	IS3
o-Xylene	95-47-6	106.2	0.8802	91	106	0.50	0.15	IS3
n-Nonane	111-84-2	128.26	0.7176	43	57, 85	0.50	0.15	IS3
1,1,2,2-Tetrachloroethane	79-34-5	167.9	1.5953	83	85	0.50	0.15	IS3
4-Bromofluorobenzene(S)	460-00-4	-	-	174	176	-	-	IS3
Cumene	98-82-8	120.2	0.8618	105	120	0.50	0.15	IS3
alpha-Pinene	80-56-8	136.24	0.8582	93	77	0.50	0.14	IS3
n-Propylbenzene	103-65-1	120.1938	0.8670	91	120,65	0.50	0.16	IS3
3-Ethyltoluene	620-14-4	120.2	0.8645	105	120	0.50	0.15	IS3
4-Ethyltoluene	622-96-8	120.2	0.8614	105	120	0.50	0.16	IS3
1,3,5-Trimethylbenzene	108-67-8	120.2	0.8652	105	120	0.50	0.16	IS3
alpha-Methylstyrene	98-83-9	118.19	0.9106	118	103,117	0.50	0.15	IS3
2-Ethyltoluene	611-14-3	120.2	0.8807	105	120	0.50	0.15	IS3
1,2,4-Trimethylbenzene	95-63-6	120.2	0.8758	105	120	0.50	0.15	IS3
n-Decane	124-18-5	142.28	0.7300	57	71,85	0.50	0.16	IS3
Benzyl Chloride	100-44-7	126.59	1.1004	91	126	0.50	0.11	IS3

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	CAS Number	Molecular Weight	Density	Primary Ion[2]	Secondary Ion(s)[2]	MRL[3] (µg/m³)	MDL[3] (µg/m³)	IS[4]
1,3-Dichlorobenzene	541-73-1	147	1.2884	146	148	0.50	0.15	IS3
1,4-Dichlorobenzene	106-46-7	147	1.2475	146	148	0.50	0.14	IS3
sec-Butylbenzene	135-98-8	134.2206	0.8601	105	134,91	0.50	0.16	IS3
p-Isopropyltoluene	99-87-6	134.2206	0.8573	119	134,91	0.50	0.15	IS3
1,2,3-Trimethylbenzene	526-73-8	120.1938	0.8944	105	120	0.50	0.15	IS3
1,2-Dichlorobenzene	95-50-1	147	1.3059	146	148	0.50	0.15	IS3
d-Limonene	5989-27-5	136.24	0.8402	68	93	0.50	0.14	IS3
1,2,Dibromo-3-Chloropropane	96-12-8	236.33	2.093	157	75, 39	0.50	0.099	IS3
n-Undecane	1120-21-4	156.31	0.7402	57	71, 85	0.50	0.15	IS3
1,2,4-Trichlorobenzene	120-82-1	181.5	1.459	180	182, 184	0.50	0.16	IS3
Naphthalene	91-20-3	128.17	1.0253	128	129	0.50	0.18	IS3
n-Dodecane	112-40-3	170.34	0.7487	57	71,85	0.50	0.13	IS3
Hexachlorobutadiene	87-68-3	260.8	1.556	225	227	0.50	0.14	IS3
Cyclohexanone	108-94-1	98.14	0.9478	55	42, 98	0.50	0.12	IS3
tert-Butylbenzene	98-06-6	134.22	0.867	119	134	0.50	0.15	IS3
n-Butylbenzene	104-51-8	134.22	0.867	91	134	0.50	0.17	IS3

(S) = Surrogate (IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3
NA = Not Available

Note 1: Additional compounds may be reported as long as the minimum requirements of this document are met. The compounds listed in this table are reported using TO-15 SCAN. The Selected Ion Monitoring (SIM) compounds are a subset of this list and are included in Table 2A.

Note 2: These are suggested primary and secondary ions. However, any ions in the analyte spectra that are sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 3: The laboratory performs three concentration level analyses (SIM, SCAN and Low Level SCAN). The method reporting limit listed is the standard SCAN limit (at or above lowest concentration in the initial calibration curve), but may change with each new initial calibration performed. Therefore, current reporting limits for the three analysis levels, MRLs in ppbv, and those from the Low Level SCAN should be reviewed in the electronic TO-15 Method Manual.

Note 4: The listing of the internal standard by which the compounds are quantitated is for TO-15 SCAN only. SIM compounds (SCAN subset) and their corresponding ions and internal standards are listed in Table 2A.

Note 5: m/e 101 is ~10% or less of m/e 85 (the base peak) and may not be present for low level results. Retention times must be carefully verified.

Table 2A – Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)					
Compound	**Primary Ion[1]**	**Secondary Ion[1]**	**MRL[2] (ug/m3)**	**MDL[2] (ug/m3)**	**IS**
Dichlorodifluoromethane	85	87	0.025	0.017	IS1
Chloromethane	52	50	0.025	0.019	IS1
Vinyl Chloride	62	64	0.025	0.0076	IS1
1,3-Butadiene	54	39	0.025	0.014	IS1
Bromomethane	94	96	0.025	0.0093	IS1
Chloroethane	64	66	0.025	0.0085	IS1
Acrolein	56	55	0.20	0.039	IS1
Acetone	58	43	2.5	0.056	IS1
Freon 11	101	103	0.025	0.015	IS1
1,1-Dichloroethene	96	98,61	0.025	0.0086	IS1
Methylene Chloride	84	49	0.10	0.013	IS1
Trichlorotrifluoroethane	151	153	0.025	0.0089	IS1
trans-1,2-Dichloroethene	96	98,61	0.025	0.0073	IS1
1,1-Dichloroethane	63	65	0.025	0.0061	IS1
Methyl tert-Butyl Ether	73	57	0.025	0.0093	IS1
cis-1,2-Dichloroethene	96	98,61	0.025	0.0092	IS1
Chloroform	83	85	0.10	0.018	IS1
1,2-Dichloroethane	62	64	0.025	0.0084	IS1
1,1,1-Trichloroethane	97	99	0.025	0.0059	IS1
Benzene	78	77	0.075	0.020	IS1
Carbon Tetrachloride	117	119	0.025	0.012	IS1
1,2-Dichloropropane	63	62,76	0.025	0.0073	IS2
Bromodichloromethane	83	85	0.025	0.0069	IS2
Trichloroethene	130	132	0.025	0.0085	IS2
1,4-Dioxane	88	58	0.10	0.0085	IS2
cis-1,3-Dichloropropene	75	77,39	0.025	0.0062	IS2
trans-1,3-Dichloropropene	75	77,39	0.025	0.0055	IS2
1,1,2-Trichloroethane	83	97,61	0.10	0.0079	IS2
Toluene	91	92	0.10	0.011	IS2
Dibromochloromethane	129	127	0.025	0.0088	IS3
1,2-Dibromoethane	107	109	0.025	0.0079	IS2
Tetrachloroethene	166	164	0.025	0.0082	IS2
Chlorobenzene	112	114	0.10	0.0092	IS3
Ethylbenzene	91	106	0.10	0.0097	IS3
m-&-p-Xylene	91	106	0.10	0.019	IS3
Styrene	104	103	0.10	0.0074	IS3
o-Xylene	91	106	0.10	0.0089	IS3
1,1,2,2-Tetrachloroethane	83	85	0.025	0.0072	IS3
1,3,5-Trimethylbenzene	105	120	0.10	0.0073	IS3
1,2,4-Trimethylbenzene	105	120	0.10	0.0083	IS3
1,3-Dichlorobenzene	146	148	0.025	0.0085	IS3
1,4-Dichlorobenzene	146	148	0.025	0.0081	IS3
1,2-Dichlorobenzene	146	148	0.025	0.0083	IS3
1,2-Dibromo-3-chloropropane	157	75	0.10	0.0095	IS3
1,2,4-Trichlorobenzene	182	184	0.025	0.013	IS3
Naphthalene	128	129	0.10	0.016	IS3
Hexachlorobutadiene	225	227	0.025	0.0092	IS3

NA = Not Available (IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3

Note 1: These are suggested primary and secondary ions. However, any ions in the analyte spectra that is sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 2: The method reporting limit listed is the standard SIM limit (lowest concentration in the initial calibration curve; must be higher than MDL), but may change with each new initial calibration performed. Therefore, current reporting limits should be reviewed. MDLs in ppbV may be reviewed in the electronic TO-15 Method Manual.

Table 3
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
Bromochloromethane (IS1)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Propene	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
Dichlorodifluoromethane (CFC 12)	0.0800	0.200	0.400	1.00	5.00	25.00	50.0	100
Chloromethane	0.0784	0.196	0.392	0.98	4.90	24.50	49.0	98
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
Vinyl Chloride	0.0800	0.200	0.400	1.00	5.00	25.00	50.0	100
1,3-Butadiene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
Bromomethane	0.0800	0.200	0.400	1.00	5.00	25.00	50.0	100
Chloroethane	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
Ethanol	0.4048	1.012	2.024	5.06	25.30	126.50	253.0	506
Acetonitrile	0.0816	0.204	0.408	1.02	5.10	25.50	51.0	102
Acrolein	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Acetone	0.4296	1.074	2.148	5.37	26.85	134.25	268.5	537
Trichlorofluoromethane	0.0792	0.198	0.396	0.99	4.95	24.75	49.5	99
Isopropyl Alcohol	0.1672	0.418	0.836	2.09	10.45	52.25	104.5	209
Acrylonitrile	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
1,1-Dichloroethene	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
tert-Butanol	0.1672	0.418	0.836	2.09	10.45	52.25	104.5	209
Methylene Chloride	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
Allyl Chloride	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
Trichlorotrifluoroethane	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
Carbon Disulfide	0.0784	0.196	0.392	0.98	4.90	24.50	49.0	98
trans-1,2-Dichloroethene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
1,1-Dichloroethane	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
Methyl tert-Butyl Ether	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
Vinyl Acetate	0.4056	1.014	2.028	5.07	25.35	126.75	253.5	507
2-Butanone (MEK)	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
cis-1,2-Dichloroethene	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Diisopropyl Ether	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
Ethyl Acetate	0.1696	0.424	0.848	2.12	10.60	53.00	106.0	212
n-Hexane	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
Chloroform	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
1,2-Dichloroethane-d4 (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Tetrahydrofuran	0.0816	0.204	0.408	1.02	5.10	25.50	51.0	102
Ethyl tert-Butyl Ether	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
1,2-Dichloroethane	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
1,4-Difluorobenzene(IS2)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
1,1,1-Trichloroethane	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
Isopropyl acetate	0.1768	0.442	0.884	2.21	11.05	55.25	110.5	221
1-Butanol	0.1808	0.452	0.904	2.26	11.30	56.50	113.0	226

Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
Benzene	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
Carbon Tetrachloride	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
Cyclohexane	0.1672	0.418	0.836	2.09	10.45	52.25	104.5	209
tert-Amyl Methyl Ether	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
1,2-Dichloropropane	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
Bromodichloromethane	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Trichloroethene	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
1,4-Dioxane	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
Isooctane	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
Methyl Methacrylate	0.1664	0.416	0.832	2.08	10.40	52.00	104.0	208
n-Heptane	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
cis-1,3-Dichloropropene	0.0896	0.224	0.448	1.12	5.60	28.00	56.0	112
4-Methyl-2-Pentanone	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
trans-1,3-Dichloropropene	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
1,1,2-Trichloroethane	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
Chlorobenzene-d5 (IS3)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Toluene-d8 (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Toluene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
2-Hexanone	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
Dibromochloromethane	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
1,2-Dibromoethane	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
n-Butyl Acetate	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
n-Octane	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
Tetrachloroethene	0.0792	0.198	0.396	0.99	4.95	24.75	49.5	99
Chlorobenzene	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Ethylbenzene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
m- & p-Xylene	0.1664	0.416	0.832	2.08	10.40	52.00	104.0	208
Bromoform	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Styrene	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
o-Xylene	0.0816	0.204	0.408	1.02	5.10	25.50	51.0	102
n-Nonane	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
1,1,2,2-Tetrachloroethane	0.0800	0.200	0.400	1.00	5.00	25.00	50.0	100
4-Bromofluorobenzene (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Cumene	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
alpha-Pinene	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
n-Propylbenzene	0.0800	0.200	0.400	1.00	5.00	25.00	50.0	100
3-Ethyltoluene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
4-Ethyltoluene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
1,3,5-Trimethylbenzene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
alpha-Methylstyrene	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
2-Ethyltoluene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
1,2,4-Trimethylbenzene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104

Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
n-Decane	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
Benzyl Chloride	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
1,3-Dichlorobenzene	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
1,4-Dichlorobenzene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
sec-Butylbenzene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
p-Isopropyltoluene	0.0800	0.200	0.400	1.00	5.00	25.00	50.0	100
1,2,3-Trimethylbenzene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
1,2-Dichlorobenzene	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
d-Limonene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
1,2-Dibromo-3-Chloropropane	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
n-Undecane	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
1,2,4-Trichlorobenzene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
Naphthalene	0.0800	0.200	0.400	1.00	5.00	25.00	50.0	100
n-Dodecane	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
Hexachlorobutadiene	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Methacrylonitrile	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
Cyclohexanone	0.0896	0.224	0.448	1.12	5.60	28.00	56.0	112
tert-Butylbenzene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
n-Butylbenzene	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 3A - Standard Concentrations (SIM) (Primary Sources)[1]

Compound Name	10pg	20pg	50pg	100pg	500pg	1000pg	2500pg	10,000pg	20,000pg	50,000pg
Freon-12	10.00	20.00	50.00	100.0	500	1000	2500	10000	20000	50000
Chloromethane	9.80	19.60	49.00	98.0	490	980	2450	9800	19600	49000
Vinyl Chloride	10.00	20.00	50.00	100.0	500	1000	2500	10000	20000	50000
1,3-Butadiene	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
Bromomethane	10.00	20.00	50.00	100.0	500	1000	2500	10000	20000	50000
Chloroethane	10.10	20.20	50.50	101.0	505	1010	2525	10100	20200	50500
Acrolein	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
Acetone	53.70	107.40	268.50	537.0	2685	5370	13425	53700	107400	268500
Freon-11	9.90	19.80	49.50	99.0	495	990	2475	9900	19800	49500
1,1-Dichloroethene	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
Methylene Chloride	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
Freon-113	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
trans-1,2-Dichloroethene	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
1,1-Dichloroethane	10.40	20.80	52.00	104.0	520	1040	2600	10400	20800	52000
Methyl tert-Butyl Ether	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
cis-1,2-Dichloroethene	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
Chloroform	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
1,2-Dichloroethane	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
1,1,1-Trichloroethane	10.30	20.60	51.50	103.0	515	1030	2575	10300	20600	51500
Benzene	11.10	22.20	55.50	111.0	555	1110	2775	11100	22200	55500
Carbon Tetrachloride	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
1,2-Dichloropropane	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
Bromodichloromethane	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
Trichloroethene	10.30	20.60	51.50	103.0	515	1030	2575	10300	20600	51500
1,4-Dioxane	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
cis-1,3-Dichloropropene	11.20	22.40	56.00	112.0	560	1120	2800	11200	22400	56000
trans-1,3-Dichloropropene	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
1,1,2-Trichloroethane	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
Toluene	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
Dibromochloromethane	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
1,2-Dibromoethane	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
Tetrachloroethene	9.90	19.80	49.50	99.0	495	990	2475	9900	19800	49500
Chlorobenzene	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
Ethylbenzene	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
m,p-Xylenes	20.80	41.60	104.00	208.0	1040	2080	5200	20800	41600	104000
Styrene	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
o-Xylene	10.20	20.40	51.00	102.0	510	1020	2550	10200	20400	51000
1,1,2,2-Tetrachloroethane	10.00	20.00	50.00	100.0	500	1000	2500	10000	20000	50000
1,3,5-Trimethylbenzene	10.40	20.80	52.00	104.0	520	1040	2600	10400	20800	52000
1,2,4-Trimethylbenzene	10.40	20.80	52.00	104.0	520	1040	2600	10400	20800	52000
1,3-Dichlorobenzene	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
1,4-Dichlorobenzene	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
1,2-Dichlorobenzene	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
1,2-Dibromo-3-chloropropane	10.40	20.80	52.00	104.0	520	1040	2600	10400	20800	52000
1,2,4-Trichlorobenzene	10.40	20.80	52.00	104.0	520	1040	2600	10400	20800	52000
Naphthalene	10.00	20.00	50.00	100.0	500	1000	2500	10000	20000	50000
Hexachloro-1,3-butadiene	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500

Note 1: The concentrations detailed in Table 3A may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 4 - Standard Concentrations (SCAN) (Secondary Sources)[1]

Compound Name	25ng	Compound Name	25ng	Compound Name	25ng
Bromochloromethane (IS1)	**25.0**	1,1,1-Trichloroethane	26.25	alpha-Pinene	26.50
Propene	24.50	Isopropyl acetate	57.25	n-Propylbenzene	25.50
Dichlorodifluoromethane (CFC 12)	23.50	1-Butanol	51.25	3-Ethyltoluene	26.75
Chloromethane	25.00	Benzene	28.25	4-Ethyltoluene	26.75
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	25.50	Carbon Tetrachloride	28.75	1,3,5-Trimethylbenzene	26.75
Vinyl Chloride	25.00	Cyclohexane	53.00	alpha-Methylstyrene	26.25
1,3-Butadiene	25.75	tert-Amyl Methyl Ether	26.75	2-Ethyltoluene	26.75
Bromomethane	25.25	1,2-Dichloropropane	27.00	1,2,4-Trimethylbenzene	27.25
Chloroethane	25.00	Bromodichloromethane	27.25	n-Decane	26.25
Ethanol	124.75	Trichloroethene	27.00	Benzyl Chloride	27.50
Acetonitrile	26.50	1,4-Dioxane	26.25	1,3-Dichlorobenzene	28.50
Acrolein	26.75	Isooctane	26.75	1,4-Dichlorobenzene	26.00
Acetone	134.75	Methyl Methacrylate	52.75	sec-Butylbenzene	27.25
Trichlorofluoromethane	27.00	n-Heptane	27.00	p-Isopropyltoluene	26.00
Isopropyl Alcohol	52.25	cis-1,3-Dichloropropene	26.00	1,2,3-Trimethylbenzene	26.50
Acrylonitrile	26.50	4-Methyl-2-Pentanone	27.50	1,2-Dichlorobenzene	27.50
1,1-Dichloroethene	27.00	trans-1,3-Dichloropropene	26.25	d-Limonene	26.25
tert-Butanol	50.00	1,1,2-Trichloroethane	27.00	1,2-Dibromo-3-Chloropropane	27.25
Methylene Chloride	27.75	**Chlorobenzene-d5 (IS3)**	**25.0**	n-Undecane	25.25
Allyl Chloride	27.25	**Toluene-d8 (S)**	**25.0**	1,2,4-Trichlorobenzene	28.75
Trichlorotrifluoroethane	27.50	Toluene	27.25	Naphthalene	27.25
Carbon Disulfide	26.25	2-Hexanone	27.50	n-Dodecane	27.25
trans-1,2-Dichloroethene	26.25	Dibromochloromethane	27.50	Hexachlorobutadiene	28.75
1,1-Dichloroethane	26.50	1,2-Dibromoethane	27.25	Methacrylonitrile	26.25
Methyl tert-Butyl Ether	27.00	Butyl Acetate	28.25	Cyclohexanone	27.50
Vinyl Acetate	129.75	n-Octane	26.25	tert-Butylbenzene	26.75
2-Butanone (MEK)	27.50	Tetrachloroethene	25.25	n-Butylbenzene	28.00
cis-1,2-Dichloroethene	27.25	Chlorobenzene	27.50		
Diisopropyl Ether	27.00	Ethylbenzene	27.25		
Ethyl Acetate	53.50	m- & p-Xylene	53.50		
n-Hexane	26.50	Bromoform	28.50		
Chloroform	28.00	Styrene	27.75		
1,2-Dichloroethane-d4 (S)	**25.0**	o-Xylene	26.25		
Tetrahydrofuran	27.50	n-Nonane	25.50		
Ethyl tert-Butyl Ether	26.75	1,1,2,2-Tetrachloroethane	26.25		
1,2-Dichloroethane	26.75	**4-Bromofluorobenzene (S)**	**25.0**		
1,4-Difluorobenzene(IS2)	**25.0**	Cumene	26.00		

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 4A – ICV/LCS Standard Concentrations (SIM) (Secondary Sources)[1]

Compound Name	500pg
Freon-12	470
Chloromethane	500
Vinyl Chloride	500
1,3-Butadiene	515
Bromomethane	505
Chloroethane	500
Acrolein	535
Acetone	2695
Freon-11	540
1,1-Dichloroethene	540
Methylene Chloride	555
Freon-113	550
trans-1,2-Dichloroethene	525
1,1-Dichloroethane	530
Methyl tert-Butyl Ether	540
cis-1,2-Dichloroethene	545
Chloroform	560
1,2-Dichloroethane	535
1,1,1-Trichloroethane	525
Benzene	565
Carbon Tetrachloride	575
1,2-Dichloropropane	540
Bromodichloromethane	545
Trichloroethene	540
1,4-Dioxane*	525
cis-1,3-Dichloropropene	520
trans-1,3-Dichloropropene	525
1,1,2-Trichloroethane	540
Toluene	545
Dibromochloromethane	550
1,2-Dibromoethane	545
Tetrachloroethene	505
Chlorobenzene	550
Ethylbenzene	545
m,p-Xylenes	1070
Styrene	555
o-Xylene	525
1,1,2,2-Tetrachloroethane	525
1,3,5-Trimethylbenzene	535
1,2,4-Trimethylbenzene	545
1,3-Dichlorobenzene	570
1,4-Dichlorobenzene	520
1,2-Dichlorobenzene	550
1,2-Dibromo-3-chloropropane	545
1,2,4-Trichlorobenzene	575
Naphthalene	545
Hexachloro-1,3-butadiene	575

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Attachment 1

Training Plan

Training Plan for Analysis of VOCs by GC/MS

Trainee _____ Trainer _____ Instrument _____ Training Completion Date _____

1. Read SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____

2. Read Methods TO-14A & TO-15A *Training Duration* _____ Trainer ____ Trainee ____ Date _____

3. Demonstrated understanding of the scientific basis of the analysis Trainer ____ Trainee ____ Date _____
   Whole air sample preconcentration techniques
   Gas chromatography *Training Duration* _____
   Mass spectrometry

4. Demonstrated familiarity with related SOPs Trainer ____ Trainee ____ Date _____
   SOP for Batches and Sequences; Rev. ___
   SOP for Making Entries onto Analytical Records; Rev. ___ *Training Duration* _____
   SOP for Manual Integration Policy; Rev. ____
   SOP for Significant Figures; Rev. ____
   SOP for Nonconformance and Corrective Action; Rev. ____
   SOP for Performing MDL Studies and Establishing Limits of Detection and Quantitation; Rev. ____
   SOP for Cleaning and Certification of Summa Canisters; Rev. ____

5. Observe performance of SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant introduction (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

6. Perform SOP with supervision *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant use (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

7. Independent performance of the SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant proficiency (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)
   ___initial demonstration of competency (4 Laboratory Control Samples)

8. Instrument operation and maintenance Trainer ____ Trainee ____ Date _____
   ___autosampler *Training Duration* _____
   ___GC and capillary column installation *Training Duration* _____
   ___mass spectrometer *Training Duration* _____
   ___data system *Training Duration* _____

Attachment 2

Initial Calibration Checklist

Initial Calibration Review Checklist - EPA Compendium Method TO-15

ICAL Date:_____ ICAL ID:_____ LIMS ICAL ID:_____

Instrument: ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21 ☐ MS22

Mode: ☐ SIM ☐ Scan Scan Low Level (0.1ng): ☐ Yes ☐ No

Analyst		Reviewer
☐	1. Is the required documentation in the ICAL file?	☐
	☐ BFB Tune analysis Report	☐
	☐ Calibration Status Report (aka Calibration History)	☐
	☐ Response Factor Report/Percent RSD	☐
	☐ Quant Report for each calibration std (including manual integration documentation)	☐
	☐ ICV Quantitation Report	☐
	☐ TO-15 Standard Calculation Spreadsheet	☐
☐	2. Was the ICAL performed continuously (not interrupted for maintenance or sample analysis)?	☐
☐	3. Have all the calibration standards been analyzed within 24 hours of each other?	☐
☐	4. Does the BFB tune check standard analysis at the start meet the tune criteria?	☐
☐	5. Are all the analytes in the blank analysis <MRL?	☐
☐	6. Does each analyte's ICAL include a minimum of 5 concentrations at 5 consecutive levels?	☐
☐	7. Were the standards analyzed from low concentration to high concentration?	☐
☐	8. For each analyte, are there no levels skipped?	☐
☐	9. For each analyte, is there only one value used for each calibration level?	☐
☐	10. For each analyte, is the lowest standard's concentration at or below the analyte's MRL?	☐
☐	11. For each analyte, is the corresponding signal to noise ratio at least 3:1 at the lowest point on the curve?	☐
☐	12. For each analyte, are the corresponding upper levels free from saturation?	☐
☐	13. If a calibration level is dropped, are all the responses for each target analyte dropped and is the information noted in the ICAL explaining the reason?	☐
☐	14. Is the average RSD ≤30% for all analytes, with no more than two exceptions ≤40%?	☐
☐	15. Is the response Y at each calibration level within 40% of the mean area response over the initial calibration range for each internal standard?	☐
☐	16. Percent recovery for each analyte in the ICV 70%-130% (50-150% for VA, unless AFCEE or DoD)?	☐
☐	17. Was the RRT for each target compound at each calibration level within 0.06RRT units of the mean RRT for the compound?	☐
☐	18. Is the retention time shift for each of the internal standards at each calibration level within 20s of the mean retention time over the initial calibration range for each standard?	☐
☐	19. If there are any manual integrations, are they performed correctly according to the corresponding SOP? If so, initial and date the appropriate pages.	☐
☐	20. Is the ICAL good at 0.5ng (or 0.1ng)–100ng (Scan) or 10-20000pg (SIM) for all compounds? ☐ Yes ☐ No Note exceptions and corresponding MRLs below – *Specify applicable range*...	☐
☐	21. Are ALL of the peak selections for each analyte correct according to retention time (all RTs must be checked by both the initial and peer reviewer)?	☐

COMMENTS:

Analyst: _____ Secondary Reviewer: _____

Attachment 3

Daily QC and Sample Review Checklists

EPA Compendium Method TO-15 - Daily QC Review Checklist
(Note exceptions in Comments and include Analysis Observations/Case Narrative Summary Form as appropriate)

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____

Instrument: ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21 ☐ MS22

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst **Reviewer**

☐ 1. Is the required documentation present? ☐
- ☐ CORRECT BFB Tune analysis Report
- ☐ CCV analysis Quantitation Report & %D Report
- ☐ LCS analysis Quantitation Report
- ☐ MB analysis Quantitation Report

☐ 2. BFB **tune** check standard analysis meet the tune criteria for the method indicated above? ☐

☐ 3. Analyses within the tune's **24-hr window or** ☐ **Client's 12hr window requirement**? ☐

☐ 4. Does the **CCV** have a difference ≤30% for all analytes? ☐
[Note all outliers biased high and/or low]

☐ 5. All **IS** retention times within 20 seconds of the CCV RT or the RT from the midpoint (ICAL)? ☐

☐ 6. All **IS** responses within ±40% of CCV or the midpoint in the ICAL? ☐

☐ 7. All **surrogate** recoveries (in CCVs, MB, LCSs, etc.) within acceptance limits (70%-130%) ☐

☐ 8. All analytes in the **MB** <MRL? (DoD <1/2MRL, except Acetone, MeCl2, EtOH, Carbon Disulfide)? ☐

☐ 9. **LCS** %R within lab control limits for all analytes except AZ samples (70%-130%, VA 50%-150%)? ☐

☐ 10. All analytes in the **Lab Duplicate / DLCS** within ±25% or the client specified limits? ☐

Air-Phase Petroleum Hydrocarbons

☐ 1. Does the **CCV** meet the following criteria? ☐
- Percent difference ≤30%.
- One compound or range can be >30%, but less than 50%.
- No single analyte or range may be >50%.

[Note outliers biased high and/or low in comments below]

☐ 2. Does lab **duplicate** meet an RPD of ≤30% for results >5x MRL? Repeat analysis if: ☐

RPD >30 (where both analyses are >5x RL	1st analysis detect @ >5x MRL, Dup=ND
1st analysis ≤5x RL; Dup=ND (RPD not calculable)	

☐ 3. Are the analytes in the **LCS** within 70%-130% recovery? ☐

COMMENTS:

Analyst/LIMS Run Approval: _____ Secondary/LIMS Supervisor Approval: _____

EPA Compendium Method TO-15 - Sample Review Checklist

(Note exceptions in Comments and include Analysis Observations/Case Narrative Summary Form as appropriate)

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____ **Project #:** _____

Instrument: ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21 ☐ MS22

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst		Reviewer
☐	1. All analyte hits in the samples within the **calibration range** and/or noted?	☐
☐	2. All **peak integrations** acceptable?	☐
☐	3. All **manual integrations** flagged and documented?	☐
☐	4. Have **Q values** been verified for each peak?	☐
☐	5. All **calculations** correct?	☐
☐	6. Has the analyst initialed and dated each **quantitation report**?	☐
☐	7. For **TICs** are the relative intensity and other requirements met (associated MB reported)?	☐
☐	8. **Auto report** correct?	☐
☐	9. **MRL** = _____ ☐ ng ☐ pg (ethanol, acetone, vinyl acetate = 5.0ng)	☐
☐	10. Pressurized with **Helium**? Is the worksheet completed for all samples?	☐
☐	11. Report to **MDL**? ☐ Yes ☐ No	☐
☐	12. **Global Minimum Detection Limit** = _____ ☐ ng ☐ pg	☐
☐	13. **DOD:** Are **manual integrations** notated in the **case narrative?**	☐

Air-Phase Petroleum Hydrocarbons

☐	1. Are all manual **integrations** flagged and documented (except for HC ranges)?	☐
☐	2. Are the associated ICAL responses correct?	☐
☐	3. Are the sample responses entered into the template correctly?	☐
☐	4. Are the TO-15 target compounds entered into the template correctly?	☐
☐	5. Does the lab **duplicate** meet RPD ≤30% for results >5x the MRL? Otherwise, repeat analyses if:	☐

RPD >30 (where both analyses are >5x RL	1st analysis detect @ >5x MRL, Dup=ND
1st analysis ≤5x RL; Dup=ND (RPD not calculable)	

COMMENTS:

☐	1. **CASE NARRATIVE COMPLETED?**	☐

Analyst/LIMS Run Approval: _____ Secondary/LIMS Supervisor Approval: _____

Attachment 4

State and Project Specific Requirements

Minnesota Requirements	
Item	**Criteria**
Holding Time (HT)	14 days
Tedlar bags	Not allowed for sampling or sample dilution
Canisters and flow controllers	Individually certified Individually leak checked before shipment
	Samples with concentrations outside of the calibration curve will have a zero canister analysis performed to check for carryover. If carryover is detected, system bake out shall be performed and documented. Additionally, in instances where the laboratory has evidence on file that a particular compound when present at a high concentration does not exhibit carry-over, the samples will not be reanalyzed. When samples are analyzed that have a higher concentration than the evidence on file, the above requirements must be followed. Also, samples that have hits below the MRL will not be reanalyzed when analyzed after a sample with concentrations over the calibration range.
Method Reporting Verification Check	Analyze a Method Reporting Verification at the beginning of the sequence prior to analyzing samples. Acceptance criteria ±40%.
Duplicates	10 percent laboratory duplicates
Record retention	MN/NELAP 5 years MPCA (Minnesota Pollution Control Agency) compliant samples 10 years
Tier level	TIII

Arizona Requirements	
Item	**Criteria**
LCS	70-130% (vinyl acetate 50-150%)

Department of Toxic Substances Control (DTSC) Requirements	
Item	**Criteria**
Holding Time (HT)	72 hour hold time for canisters

EPA Region 9 Requirements	
Item	**Criteria**
Holding Time (HT)	14 days

MICROBAC

STANDARD OPERATING PROCEDURE
MICROWAVE DIGESTION - AQUEOUS
SW846-3015A

Issue/Implementation Date: 15 January 2016

Last Review Date: 15 December 2016

Microbac Laboratories, Inc.
Ohio Valley Division
158Starlite Drive
Marietta, Ohio 45750

Approved By:

Robert E. Kyer, Digest Group Leader — Date 12/01/2016

Kim H. Rhodes, Metal Supervisor — Date 12-2-2016

Maren M. Beery, Quality Assurance Officer — Date 12/02/2016

Leslie S. Bucina, Laboratory Manager — Date 12/5/16

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TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This procedure utilizes SW-846 Method 3015A and is an acid digestion procedure used to prepare surface water, groundwater, TCLP and mobility procedure extracts, and waste samples that contain suspended solids for analysis by Inductively Coupled Argon Plasma Spectroscopy (ICP) or by ICP-MS. Samples prepared by this method may be analyzed by ICP or ICP-MS for the metals in Table 1.

1.2 For the analysis of dissolved metals, the sample is filtered at the time of collection, prior to acidification with nitric acid.

1.3 Forty milliliters or less of a well shaken sample is transferred to a digestion vessel. Nitric acid is added and in the case of TCLP (SW846 1311) and SW846 6010 batches hydrochloric acid is also added. The closed vessels are microwave digested. The cooled digestates are transferred to graduated digestion tubes and brought to final volume. Samples are filtered and/or centrifuged prior to transfer and voluming if necessary. All batch QA/QC samples are treated identically to client samples.

1.4 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

BS	Blank Spike
BSD	Blank Spike Duplicate
DI water	Deionized water
DIG-ICP	Digest Inductively Coupled Plasma
HCl	hydrochloric acid
HNO_3	nitric acid
ICP	Inductively Coupled Plasma
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NCR	Nonconformance Report
OES	Optical Emission Spectroscopy
QA/QC	Quality Assurance/Quality Control
RGT	Reagent

SDS Safety Data Sheet
SOP Standard Operating Procedure
STD Standard
TCLP Toxicity Characteristic Leachate Procedure
For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

2.0 SAFETY PRECAUTIONS

2.1 Safety glasses, gloves and lab coats must always be worn when doing this procedure.

2.2 Always use a fume hood when adding concentrated Nitric acid (HNO_3) or hydrochloric acid (HCl) to the vessels.

2.3 Venting of the vessels must only be done when contents are at room temperature inside a hood with shield lowered to avoid the potential for chemical burns.

2.4 SDSs for each analyte and reagent used within the laboratory are available to all employees. Consult SDSs prior to handling chemicals.

3.0 SAMPLE PRESERVATION AND STORAGE

Measurement	Digestion Volume Requirement (mL)	Collection Volume (mL)	Preservative/ Holding Time*
Total	40	600	HNO_3 to pH <2 / 6 months
Dissolved	40	600	Filter on site; HNO_3 to pH <2 / 6 months
Suspended	40	600	Filter on site / 6 months

* Holding time is the storage time allowed between sample collection and analysis when properly preserved and stored.

3.1 All samples must be collected by the use of techniques that prevent contamination and cross-contamination between samples.

3.2 All sample containers must be pre-cleaned. Glass or plastic are both acceptable.

3.3 Total recoverable metals: All samples must be acidified at the time of collection with concentrated HNO_3 (5 mL/L).

3.4 Dissolved metals: All samples must be filtered through a 0.45 micrometer filter and then acidified at the time of collection with concentrated HNO_3 (5 mL/L).

3.5 Aqueous samples must be preserved to pH less than 2 with HNO_3. The pH is checked by the sample receiving department prior to login. For determinations of dissolved and suspended metals, the sample must be filtered before preservation on site. Samples that are received unpreserved are preserved by the digestion laboratory personnel on site and must sit for 24 hours prior to digestion. The pH determined upon receipt, after adjustment and after 24 hours is recorded in the Laboratory Preservation Log book. If the pH after resting 24 hours is found to be noncompliant, additional acid is added and the sample must sit for another 24 hours prior to a further pH check.

4.0 METHOD PERFORMANCE

4.1 For estimated quantitation limit and working linear range, refer to Section 4.0 of Microbac SOPs ME600 and ME700. Method performance data are acquired as per Microbac SOP 45.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Very reactive or volatile materials that create high pressure when heated may cause venting of the vessels with potential loss of sample and analytes. Samples that contain carbonates or other carbon dioxide generating compounds may cause enough pressure to vent the vessel. If this situation is anticipated the analyst may wish to use a smaller amount of sample.

6.0 EQUIPMENT AND SUPPLIES

6.1 Major Instrumentation

- Mars Xpress unit. Microwave unit must provide programmable power with a minimum of 574W and can be programmed to within ± 10W of required power.
- 75 mL Vessels for the Mars Express

6.2 Apparatus or Equipment

- Beckman GS-6 centrifuge or equivalent
- Analytical balance (600g capacity) or equivalent

6.3 Other Supplies

- Graduated digest tubes (50 mL or 100 mL Capacity)
- Quantitative filter paper, Whatman 41 or equivalent
- Volumetric pipettes
- VWR 50 mL disposable centrifuge tubes or equivalent

6.4 Mars Synergy Software: Version 194A11

6.5 Calibrated Mechanical Pippettes

6.5.1 100 - 1000 uL

6.5.2 50 uL - 200 uL

6.5.3 25 uL

7.0 STANDARDS AND REAGENTS

Acids used in the preparation of samples must be reagent grade or better. Redistilled acids may be used.

All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.1 QC-MS-1 Spike – 10 mg/L, As, Al, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Sb,Se, Ag, Tl, U, V and Zn in 2% HNO3 and tr HF – CPI International or equivalent.

7.2 Custom multi-element solution MIC-SPK-1A-REV2 from Inorganic Ventures or equivalent containing:

250 mg/L: K, Na
50 mg/L: Al, Ca, Mg, P

25 mg/L:	Si
20 mg/L:	Fe
6 mg/L:	Sb
5 mg/L:	Ba, Li, Mo, Ti, V, Zn, Sr, SN
2.5 mg/L:	Cr, Cu, Mn, Ni, Pb, Tl,
2 mg/L:	Ag, As, Se
1 mg/L:	Co
0.25 mg/L:	Be, Cd
10 mg/L:	B

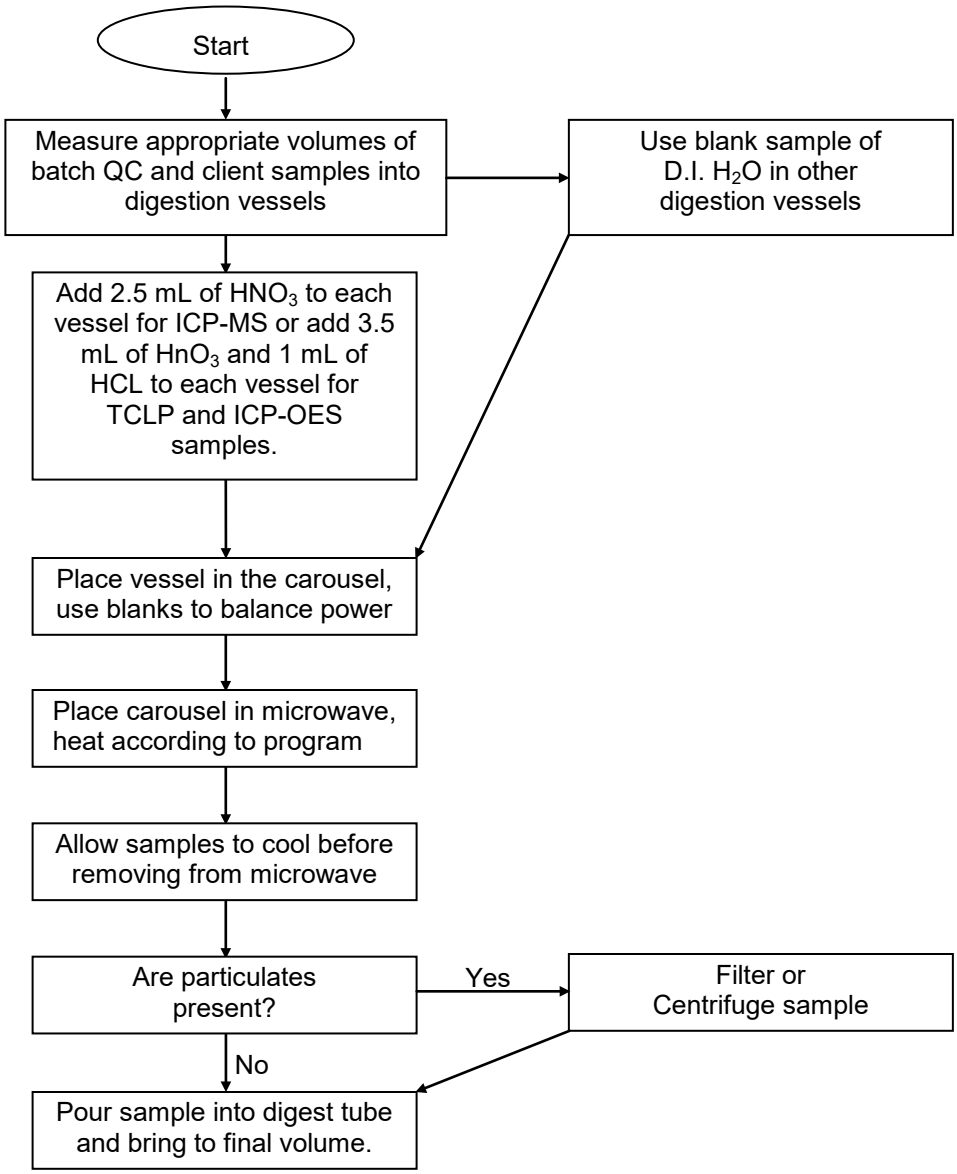
7.3 1000 ug/mL: Zr

7.4 Concentrated HNO_3 (Baker Instra analyzed or equivalent).

7.5 ASTM Type II Water (ASTM D1192): Water must be monitored for impurities.

7.6 Concentrated HCl (Baker Instra analyzed or equivalent)

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



9.0 SAMPLE PREPARATION

9.1 Samples are shaken to homogenize prior to digestion.

10.0 CALIBRATION PROCEDURES

10.1 The Mars Express is calibrated annually by the Manufacturer.

11.0 ANALYTICAL PROCEDURES

11.1 Choose 20 samples of similar matrix for the preparation batch.

11.2 For ICP-MS samples, measure 20 mL of a well shaken sample into a 50 mL digest tube and transfer the aliquot into a digestion vessel. 20 mL of DI water are used for the blank and LCS. Add 2.5 mL of HNO_3 to each vessel including blank and LCS.

For ICP samples, measure 40 mL of a well shaken sample into a 50 mL digest tube and transfer the aliquot into a digestion vessel. 40 mL of DI water are used for the blank and LCS.

NOTE: If a high organic content is suspected, such as TCLP extracts, 5 mL or less of sample may be used (the difference is made up with DI water).

For TCLP and DIG-ICP samples, add 3.5 mL of HNO_3 and 1.0 mL of HCl to all samples including blank and LCS.

11.2.1 Water batches for ICP-MS analysis are spiked with 0.25 mL of QC-MS-1 spike (7.1) CPI International or equivalent.

11.2.2 6010 Batches are spiked with 5 mL of custom multielement solution MIC-SPK-1A-REV2.

11.3 Mars Xpress – Follow these guidelines

11.4 Seal all samples with rubber stoppers and also the vessel cap. Hand tighten vessel cap only.
Weigh vessels and record weight in microwave electronic digestion log template.

Place vessels in carousel making sure they are pushed down completely. Then place the carousel in the microwave oven.

Click the “Run Date/Time:” box in the electronic digestion log template to insert the current date and time.

Select “Load Method” from main menu of microwave.

Select “User Directory”.

Select appropriate 3015 method to be used (EPA 3015-8V-Xpress, - 16 press, -24 Xpress).

Push start button.

11.4.1 After the program has finished, allow sample to cool down.

11.4.2 Remove carousel from unit and reweigh vessels and record weight in electronic digestion log template. If the weight has decreased more than 1% from the original weight, discard sample and start the sample over again.

11.4.3 Transfer the solution to a graduated digest tube and bring up to a 50 mL volume with DI H_2O.

Digestion tubes are calibrated volumetrically by lot number when received as per Microbac SOP K0002 “Calibration Procedures”.

11.4.4 Centrifugation: Transfer sample in 50 graduated centrifuge tube and place in centrifuge for at least 10 minutes at 2,000 – 3,000 RPM. Slowly decant sample into a clean digest tube and bring up to a 50 mL volume with DI Water. The sample is now ready for analysis. If filtration is needed after centrifugation, this is done by filtering sample through a .45 uL filter into a clean digest tube and bringing up to a 50 mL volume with DI water. The sample is now ready for analysis.

11.5 See Figure 11.1 for examples of the Metals Digest Logs.

12.0 DETAILS OF CALCULATIONS

12.1 Refer to individual methodology.

13.0 QUALITY CONTROL REQUIREMENTS

13.1 Each batch of up to 20 samples requires the following:

13.1.1 Method blank (MB) – an aliquot of DI water that is digested with the sample batch and treated identically as the client samples.

13.1.2 LCS – an aliquot of DI water that is spiked with spiking solution and digested with the sample batch and treated identically as the client samples.

13.1.3 Sample duplicate – A sample prepared in duplicate, both carried through the batch digestion and treated identically as the client samples- by client request only.

13.1.4 MS and MSD – two additional aliquots of a sample that are spiked with spiking solution and digested with the sample batch. Batches that include samples for method 200.8 will include a spiked sample for every ten (10) 200.8 samples. The MS and MSD are treated identically as the client samples.

13.2 Results of the analysis of the QA/QC samples are kept for easy reference.

13.3 All batch QC samples are subjected to exactly the same digestion and acid concentrations as those used on actual samples in the digestion batch.

13.4 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.4.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Tables 13.1 and 13.2 of Microbac SOPs ME600E and ME600G and Tables 13.1 and 13.4 of Microbac SOP ME700, does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Tables 13.1 and 13.2 of Microbac SOPs ME600E and ME600G and Tables 13.1 and 13.4 of Microbac SOP ME700.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch Figure 14.1 of Microbac SOPs ME600E, ME600G and ME700. A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.4.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RCA, corrections, corrective action(s) and evidence of effectiveness.

13.4.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate a NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank or calibration blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank or calibration blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 The appropriate log books must be checked and signed by the department supervisor for completeness.

15.0 PREVENTIVE MAINTENANCE

15.1 Monthly examine the door, door seals and door interlocks of the microwaves to verify they are clean and working properly. Ensure that the door closes securely.

15.2 Clean the inside of the microwave cavity, including the exhaust screen at the back of the cavity, with warm soapy water applied with a soft cloth. Rinse and thoroughly dry all cleaned areas.

15.3 Clean the exhaust outlet of the microwave by removing the exhaust hose and wiping the space inside the exhaust outlet with a disposable cloth. To clean the exhaust hose, disconnect if from the blower exhaust duct, flush it with water and allow it to dry before reconnecting it to the blower duct.

15.4 All microwaves are under service contracts, so every year a service representative will calibrate and do a systems check.

15.5 If temperature is not reaching desired temperature, check IR sensors using the IR calibrating device.

15.6 In case of system shut down, first check the (2) fuses in the back of the microwave. If they are good, call the service department to have them come check out the unit.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

16.2 The following are waste streams in the sample preparation area.

16.2.1 Non – Halogenated solvents: Acetone

16.2.2 Solid Waste: Filters, tongue depressors, gloves, any solid material that is a waste after being processed in the lab.

16.2.3 Acid: Dilute acid waste from soak tanks.

16.3 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33, Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee received training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

17.0 REFERENCES

17.1 Microwave Assisted Acid Digestion of Aqueous Samples and Extracts, US EPA SW-846, Method 3015A, Revision 1, February 2007, EPA Publication SW-846.

17.2 Microbac SOP LQAP “Laboratory Quality Assurance Plan”

17.3 Microbac SOP 33 “Laboratory Waste Management”

17.4 Microbac SOP 45 “Method Validation Procedures”

17.5 Microbac SOP GP-CAPA “Corrective Action and Preventive Action; Initiating, Tracking, and Monitoring”

17.6 Microbac SOP GP-RCA “Root Cause Analysis”

17.7 Microbac SOP ME600E “Perkin Elmer OPTIMA 4300 Inductively Coupled Plasma Atomic Emission Spectroscopy”

17.8 Microbac SOP ME600G “Thermo iCAP 6000 Series Inductively Coupled Plasma Atomic Emission Spectroscopy”

17.9 Microbac SOP ME700A “Perkin Elmer NexION 300X Inductively Coupled Plasma/Mass Spectrometer (SW-846 6020 / EPA METHOD 200.8)

17.10 Microbac SOP K0002 “Calibration Procedures”

17.11 40CFR Part136.3 Table II – Required Containers, Preservation Techniques, and Holding Times, footnote 19

An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (See footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances

in this footnote supersede the preservation and holding time requirements in the approved metals methods.

Footnote 2 defines immediately as within 15 minutes of collection.

17.12 Microbac SOP TCLP01 “Toxicity Characteristic Leachate Procedure”

Table 1
Method Analytes

Name	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
Boron	B	7440-42-8
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
Lithium	Li	7439-93-2
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Phosphorus	P	7723-14-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silicon	Si	7440-21-3
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Strontium	Sr	7440-23-5
Thallium	Tl	7440-28-0
Tin	Sn	7440-31-5
Titanium	Ti	7440-32-6
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6
Zirconium	Zr	7704-67-7
Calculated		
Hardness, Calculated (as $CaCO_3$)	$CaCO_3$	72608-12-9
Silica (as SiO_2)	SiO_2	99439-28-8

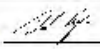
Figure 11.1

Microbac Laboratories Inc.
Microwave Digestion Log

Workgroup: WG419896
Analyst: REK
Spike Analyst: REK
Run Date: 02/05/2013 05:40
Method: 3015
Balance: BAL016
Instrument: MW-2
Instrument Start: 02/05/2013 05:48

SOP: ME407 Revison 13
Spike Solution: STD56032
Spike Witness: VC
Digestion Tubes Lot #: COA16400
HCL Lot #: COA16547
HNO3 Lot #: COA16631
6010 h2o mdl verfs LOQ LSTD55795

	SAMPLE #	Type	Matrix	Initial Amount	Final Volume	Initial Vessel Wt	Final Vessel Wt	Spike Amount	Due Date
1	WG419896-01	BLANK	1	40 mL	50 mL	202.949 g	202.943 g		
2	WG419896-02	LCS	1	40 mL	50 mL	207.665 g	207.643 g	5 mL	
3	L13010746-01	ML01	1	40 mL	50 mL	203.512 g	203.485 g	40 mL	02/08/13
4	L13010746-02	ML02	1	40 mL	50 mL	204.145 g	204.125 g	40 mL	02/08/13
5	L13010746-03	ML03	1	40 mL	50 mL	203.801 g	203.789 g	40 mL	02/08/13
6	L13010746-04	ML04	1	40 mL	50 mL	204.308 g	204.293 g	40 mL	02/08/13
7	L13010746-05	ML05	1	40 mL	50 mL	204.184 g	204.168 g	40 mL	02/08/13
8	L13010746-06	ML06	1	40 mL	50 mL	203.302 g	203.289 g	40 mL	02/08/13
9	L13010746-07	ML07	1	40 mL	50 mL	205.097 g	205.087 g	40 mL	02/08/13

Analyst: 

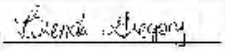
Reviewer: 

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Figure 11.2

Microbac Laboratories Inc.
Microwave Digestion Log

Workgroup: WG537928
Analyst: VC
Spike Analyst: VC
Run Date: 09/08/2015 08:48
Method: 3015
Balance: BAL016
Instrument: MW-3
Instrument Start: 09/08/2015 08:53

SOP: ME407 Revison 17
Spike Solution: STD71855
Spike Witness: ERP
HNO3 Lot #: COA18442
40 & 50 ML. DIGESTION TUCOA18222
MS Filters- fisher-Lot#rRGT32947

	SAMPLE #	Type	Matrix	Initial Amount	Final Volume	Initial Vessel Wt	Final Vessel Wt	Spike Amount	Due Date
1	WG537928-02	BLANK	1	20 mL	50 mL	180.18 g	180.167 g		
2	WG537928-03	LCS	1	20 mL	50 mL	183.302 g	183.29 g	.25 mL	
3	L15090184-01	SAMP	1	20 mL	50 mL	184.598 g	184.585 g		09/17/15
4	L15090185-01	SAMP	1	20 mL	50 mL	181.399 g	181.365 g		09/17/15
5	L15090201-01	SAMP	2	20 mL	50 mL	180.979 g	180.938 g		09/10/15
6	L15090257-01	SAMP	1	20 mL	50 mL	181.258 g	181.235 g		09/09/15
7	L15090257-02	SAMP	1	20 mL	50 mL	181.132 g	181.112 g		09/09/15
8	L15090257-03	SAMP	1	20 mL	50 mL	183.199 g	183.195 g		09/09/15
9	L15090257-04	SAMP	1	20 mL	50 mL	181.664 g	181.662 g		09/09/15
10	L15090296-01	SAMP	2	20 mL	50 mL	182.132 g	182.114 g		09/11/15
11	L15090296-06	SAMP	2	20 mL	50 mL	181.463 g	181.452 g		09/11/15
12	L15090296-08	SAMP	2	20 mL	50 mL	181.746 g	181.73 g		09/11/15
13	L15090299-01	SAMP	2	20 mL	50 mL	181.506 g	181.502 g		09/11/15
14	L15090299-02	SAMP	2	20 mL	50 mL	182.009 g	182.015 g		09/11/15
15	L15090299-03	SAMP	2	20 mL	50 mL	181.377 g	181.387 g		09/11/15
16	L15090302-01	SAMP	2	20 mL	50 mL	181.265 g	181.261 g		09/11/15
17	L15090308-01	SAMP	2	20 mL	50 mL	180.558 g	180.535 g		09/11/15
18	L15090315-01	SAMP	2	20 mL	50 mL	183.139 g	183.056 g		09/11/15
19	WG537928-01	REF	2	20 mL	50 mL	182.176 g	182.153 g		
20	L15090316-01	SAMP	2	20 mL	50 mL	182.176 g	182.153 g		09/11/15
21	L15090365-02	SAMP	1	20 mL	50 mL	184.761 g	184.723 g		09/10/15
22	WG537928-04	DUP	1	20 mL	50 mL	181.28 g	181.255 g		
23	WG537928-05	MS	1	20 mL	50 mL	182.81 g	182.779 g	.25 mL	
24	WG537928-06	MSD	1	20 mL	50 mL	181.874 g	181.84 g	.25 mL	

L15090184-01	FILTERED DIGESTATE
L15090299-02	FILTERED DIGESTATE
L15090299-03	FILTERED DIGESTATE
L15090315-01	FILTERED DIGESTATE
L15090365-02	FILTERED DIGESTATE

Analyst: Vicki Collins

Reviewer: Erin Potter

MW_DIG - Modified 09/30/2009
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Microbac

MICROBAC

STANDARD OPERATING PROCEDURE
SAMPLE RECEIVING AND LOGIN

Issue/Implementation Date: 18 February 2015

Last Review Date: 18 October 2016

Microbac Laboratories, Inc.
Ohio Valley Division
158 Starlite Drive
Marietta, Ohio 45750

Approved By:

Maren M Beery — 10/19/2016
Maren M. Beery, Quality Assurance Officer — Date

Wade T. Dr for LSB — 10-19-16
Leslie S. Bucina, Laboratory Manager — Date

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This document outlines procedures for sample handling, custody, container preparation, preservation, receipt, inspection/pH, login, Internal Chain of Custody, and storage. The document also addresses Temperature Monitoring and IR gun calibration.

1.2 Except as noted in Section 7.0, the support services supervisor shall have primary responsibility for implementation of these policies and procedures. Section 6.0 describes the duties of the sample custodians. Section 7.0 describes the specific duties of the customer service staff with respect to the procedures for logging samples into the LIMS.

1.3 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

COC	Chain of Custody
DI water	Deionized water
ICOC	Internal Chain of Custody
IR gun	Infrared Temperature Gun
LIMS	Laboratory Information Management System
LQAP	Laboratory Quality Assurance Plan
SDS	Safety data sheet
SOP	Standard Operating Procedure
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compounds

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

2.0 SAFETY PRECAUTIONS

2.1 Safety glasses with side shields, gloves, and lab coats are worn when samples are being handled. (Safety glasses are worn at all times in the laboratory.) Additional personal safety equipment (respirators and dust masks) are available in the login area.

2.2 Occasionally samples are received broken. When this occurs, the cooler is placed immediately under the hood and samples are removed. When possible, the

broken sample is placed in another container. The client is then notified by his/her Account Manager to confirm what appropriate action to take.

2.3 Broken glass is disposed of in the proper containers provided in the laboratory.

2.4 The cooler is cleaned by waste disposal personnel. All contaminated material is disposed of properly using proper waste disposal procedures. See Microbac SOP 33.

2.5 Unknown waste samples that are received broken are placed in a container and taken to sample archive for proper disposal.

2.6 Radiological screening of samples may be performed on coolers received for special projects if required by the project QAPP. The screening is performed by the login personnel and noted in receipt information.

2.7 When highly contaminated samples are received, special precautions are taken. These samples receive special handling and storage and are tagged with a "Special Instructions" sticker. Any comments available are entered into the LIMS for the laboratory.

2.8 SDSs for each analyte and reagent used within the laboratory are available to all employees. Consult SDSs prior to handling chemicals.

3.0 EQUIPMENT AND SUPPLIES

3.1 Thermometers

3.2 pH strips: Low range 0.0 - 6.0; High range 7.5 - 14.0

3.3 Hood

3.4 IR Temperature Guns

3.5 Pipets - disposable

3.6 Geiger Counter

3.7 Gloves - disposable

3.8 PDA, Laptop or notebook computer (equipped for bar coding)

4.0 REAGENTS

4.1 20% Nitric Acid (HNO_3) Baker Instra-analyzed, or equivalent; prepared when needed in the metals laboratory. A 1000 mL glass dispenser is used to store and dispense the preservative. When HNO_3 is used as a preservative, a red label with HNO_3 is placed on the lid of the container. Label container with expiration date of 6 months after date prepared.

4.2 1:1 Hydrochloric Acid (HCL) Baker Instra-analyzed, or equivalent; prepared in the metals laboratory. 500 mL of concentrated HCL is added to 400 mL of DI water and diluted to 1L. A 1000 mL glass dispenser is used to store and dispense the preservative. When HCL is used as a preservative, a blue label with HCL is placed on the lid of the container. Label container with expiration date of 6 months after date prepared.

4.3 1:1 Sulfuric Acid (H_2SO_4) Baker Instra-analyzed or equivalent; prepared when needed in the conventional lab. 500 mL of concentrated H_2SO_4 is added to 400 mL of DI water and diluted to 1 L. A 4 L glass container is used to refill to 1000 mL glass dispenser. When H_2SO_4 is used as a preservative, a yellow label with H_2SO_4 is placed on the lid of the container. Label the container with expiration date of 6 months after date prepared.

4.4 50% Sodium Hydroxide (NaOH) comes in a ready to use solution, or equivalent. No mixing is required. A 250 mL glass container is used to store and dispense this preservative. When NaOH is used as a preservative, a blue label with NaOH is placed on the lid of the container. A sterile pipet is used to add preservative to the container. Use expiration date from the manufacturer.

4.5 Zinc Acetate/Sodium Hydroxide (ZnAc/NaOH). Zinc Acetate: prepared when needed in the conventional lab. 88 g of Zinc Acetate dihydrate crystal is dissolved in 200 mL of DI water and diluted to 250 mL. A 250 mL amber glass container is used to store and dispense the preservative. To make ZnAc/NaOH, 2 mL of ZnAc is added to the container using a sterile pipet. Add 2 mL of NaOH with another sterile pipet. Then a gold dot with ZnAc/NaOH is placed on the lid of the container. This preservative is only used for sulfide. Label container with expiration date for 6 months after date prepared.

4.6 Ascorbic Acid (C6H806) comes ready to use Ascorbic Acid, Fine Powder. No mixing is required. A 125 mL pre-cleaned wide mouth glass container is used to store and dispense the preservative. A spatula is used to add 25 mg to a 40 mL, pre-cleaned vial with septa lid. Use expiration date from the manufacturer.

4.7 Hexane: 95% ULTRA resi-Analyzed; used in PCB – wipes.

4.8 Methylene Chloride (CH2CL2) ULTRA – Resi – Analyzed used in PAH – wipes.

4.9 Sodium Thiosulfate (Na2S2O3) tablets come ready to use in a sterile plastic container. No preparation is needed.

4.10 Methanol (MeOH): EDM OmniSolv or equivalent grade suitable for purge and trap.

5.0 CUSTOMIZED SAMPLE KITS

5.1 Microbac prepares customized sampling kits for many environmental sampling projects for wastewater, groundwater, soil and waste. The process for preparing a sample kit begins when an Account Manager generates a work order (B Number) and the associated kit request/packing list.

5.1.2 The support service staff member uses the packing list to assemble the specified containers for the work order.

5.1.3 Staff member then prints out labels from the LIMS database table that states client, test, preservative and the site where the samples will be taken.

5.1.4 Containers are then set up on the work station table to be labeled and preserved.

5.1.5 Sample containers are then packed in coolers with packing material, packing lists, custody seals and chain of custody forms.

5.1.6 Cooler is then sealed with the custody seal and shipped or delivered to client.

5.1.7 Container Preparation:

All sample containers are received pre-cleaned. Pre-cleaned containers are received in cases that are labeled. Lot numbers and analyte certification records are tracked and filed with start and end dates of the Certificate of Analysis. Sample containers are never reused.

5.2 Sample Containers, Volume and Preservative

Tables 1-6 list the container type, minimum volume, and type of preservative for each analysis parameter or method. Additional instructions are provided below for conventional and wet chemistry parameters.

5.2.1 Acidity (acid) – by itself in 250 mL plastic. No preservative.

5.2.2 Alkalinity (Alk) – by itself in 250 mL plastic. No preservative.

5.2.3 Biochemical Oxygen Demand (BOD) – by itself in 500 mL plastic. No preservative

5.2.4 Bromide (Br) – by itself in 250 mL plastic. No preservative.

5.2.5 Cyanide (CN) – by itself in 250 mL plastic with 3.5 mL of 50% NAOH = pH > 12.

5.2.6 Cyanide, Amenable (CN-A) – by itself in 250 mL plastic or with CN with 3.5 mL of 50% NAOH pH > 12.

5.2.7 Coliform Fecal (Col-FC) – by itself in sterile plastic container with pellet of $Na_2S_2O_3$. (These sterile containers come prepared.)

5.2.8 Coliform Total (Col-TC) – by itself plastic container with pellet of NA2S2O3. (These sterile containers come prepared.)

5.2.9 Dissolved Oxygen (DO) – by itself in 500 mL lab glass bottle with glass stopper. When sampling be sure there is no headspace. No preservative.

5.2.10 Fluoride, Total (distilled) (F-Dist.) – by itself in 250 mL plastic. No preservative.

5.2.10.1 Distilled (subbed out) 250 mL plastic by itself – no preservatives

5.2.10.2 Non –Distilled, 250 mL, no preservative

5.2.11 Hardness (Hard) – by itself in 250 mL plastic with 3 mL of 20% HNO_3 pH < 2.

5.2.12 Iodide (I) – by itself in 250 mL plastic. No preservative. Can be included with Br.

5.2.13 Coliform Fecal/MPN – by itself in sterile plastic container with pellet of $Na_2S_2O_3$.

5.2.14 Nitrogen Organic (N_ORG) –250 mL plastic with H_2SO_4 pH<2. Can be combined with other preservative parameters.

5.2.15 Oil and Grease (OG) – by itself in 1000 mL glass with 5 mL of 1:1 HCL pH<2.

5.2.16 Phenolics, Total (T-Phen) – by itself in 250 mL amber glass with 1 mL of 1:1 H_2SO_4= pH<2.

5.2.17 Sulfite (SO_3) – by itself in 250 mL plastic. No preservative. Must notify lab.

5.2.18 Settleable Solids (Set –S) – need two bottles in 1000 mL plastic. No preservative.

5.2.19 Silica Dissolved (Silica) – by itself in 250 mL plastic. No preservative.

5.2.20 Sulfide (S) – by itself in 500 mL plastic with 4 mL of ZnAce/NaOH – pH > 9.

5.2.21 Total Dissolved Solids (TDS) – by itself in a 250 mL plastic or together with TSS in a 500 mL plastic. No preservative.

5.2.22 Total Organic Carbon (TOC) – can be put with other parameters with the same preservative. If by itself it goes into 250 mL plastic with 1 mL 1:1 H_2SO_4. Other requirements for TOC are as follows: TOC-4 used 4x250 mL plastic with 1:1 H_2SO_4, TOC -14 uses 1x250 mL glass with 1:1 H_2SO_4, TOC-44 uses 4x250 mL glass with 1:1 H_2SO_4.

5.2.23 Total Organic Halides (TOX) – by itself in 250 mL amber glass, septa lid with 1 mL of 1:1 H_2SO_4. No headspace in sample. Other requirements for TOX are TOX-4 uses 4x250 mL amber glass, septa lid with 1:1 H_2SO_4. No headspace in sample.

5.2.24 Total Suspended Solids (TSS) – by itself in a 250 mL plastic or together with TDS in a 500 mL plastic. No preservative.

5.3 Volume of Preservative use for Container Sizes:

H_2SO_4:	40 mL	½ mL = pH < 2
	250 mL	1 mL = pH < 2
	500 mL	2 mL = pH < 2
	1000 mL	4 mL = pH < 2
HNO_3:	250 mL	3 mL = pH < 2
	500 mL	5 mL = pH < 2
	1000 mL	10 mL = pH < 2
HCL:	40 mL	3 drops = pH < 2
	1000 mL	5 mL = pH < 2
NAOH:	250 mL	3.5 mL = pH < 2
MeOH:	40 mL	10 mL used in 5035 Field Prep/tare weight

5.4 Special Procedures for Volatile Organics Analysis (VOA)

5.4.1 General

Samples for VOA analysis should always be collected in separate containers. If this is not possible, the VOA analyses should be performed first or split into a separate VOA container to avoid contamination of the sample with common lab solvents.

5.4.2 Water

All water VOA containers must be filled completely with no headspace/bubbles >6mm. Preservatives, if required, consist of 3 drops of 1:1 HCl per each 40 mL vial (pH should be < 2). Pre-preserved vials are also purchased from our container vendor. These include the HCl pre-preserved as well as the $Na_2S_2O_3$ pre-preserved.

5.4.3 Oils and Waste Samples

No preservative is required for waste samples. Depending on the client and project, waste and oil samples may be collected in various glass bottles or vials. Sample volume of 5-10 mL is normally sufficient for VOA analysis of wastes and oils.

5.5 Procedure for Method 5035 – Methanol Preserved Vials

Methanol preserved vials are obtained from a container vendor when possible. If out of stock, we will employ the following procedures to prepare these in-house. Contact the Quality Assurance Officer before proceeding.

5.5.1 Print labels with client's name, parameter and preservative (MeOH).

5.5.2 Go to volatiles lab and get 40 mL VOA vial for prep of 5035 method, place label on bottles.

5.5.3 Using a calibrated volumetric dispenser, add 10 mL of MeOH to each vial. Replace cap and septum.

5.5.4 Determine the tare weight to 0.01 g and record on the vial label (beside the parameter) using a permanent marker.

5.5.5 The kit request will specify the number of containers to be provided. Place the containers in bubble bags and pack them in shipping cooler. Label the outside of the cooler with a label that states "This package conforms to 49CFR – 173.4".

6.0 SAMPLE HANDLING AND CUSTODY PROCEDURES

6.1 This section describes the procedures for receipt, inspection/pH, labeling, storage internal custody and handling of laboratory samples. These are the primary duties and responsibilities of the sample custodian(s) under the direction of the support services supervisor. Many of the processes in this section require knowledge of the Microbac-OVD LIMS and specific training in the use of electronic devises including the IR temperature guns, barcode scanning devices, and PDAs, or laptop/notebook style computers. The following sections provide more details on these procedures and the tools required.

6.2 Sample Receipt Checklist/Discrepancies

The cooler inspection form contains the following details: the cooler received date, client identification, shipping agency and time received, opened by, login number, which IR gun was used, the assigned cooler number, the cooler temperature, the air bill number, the cooler seal information, was the chain of custody provided, were samples received intact, labels legible/complete, were correct containers used, were the correct preservatives used, was the pH range acceptable, were VOA's free of head space, is the chain of custody signed and dated, were samples received within EPA hold times, were the temperatures required for each type of sample in the cooler, any discrepancies are documented on Cooler Inspection Form and the Account Manager is notified. An example of Cooler Inspection Form is presented in Figure 1.

6.3 Sample Receipt and Inspection, General

6.3.1 Samples are received through shipping and receiving and moved directly to the login area. Coolers and/or boxes are checked to determine if they are sealed with tape or bandings when received.

6.3.2 When samples are returned through Microbac couriers, coolers are not sealed unless requested by client.

6.3.3 Coolers are opened in the login area. When coolers are identified as AFCEE or Radiological projects, the custodian will open them under the fume hood. Radiological screening will occur as outlined in the Microbac Radsafety SOP for known radiological projects before opening the coolers. This practice must be followed for all coolers containing potentially hazardous samples, unknowns, or any broken or leaking containers.

6.3.4 Samples that are delivered to the laboratory on the same day they are collected may not meet requirements of ≤6°C and not frozen. In these cases, the samples

shall be considered acceptable if the samples were received on ice. The allowance for samples exceeding temperature requirements when delivered shortly after sampling does not alleviate the requirement to record a temperature, even in the presence of ice. Documentation of receipt on ice is not sufficient to meet method requirements, since methods require the temperature upon receipt. Methods and regulations require that the temperature upon receipt be recorded, regardless of whether that information is in compliance or out of compliance. A temperature blank or sample is immediately removed from the cooler and the temperature is taken with the IR Temperature Gun. The temperature is taken by shooting the bottom of a sample. Upon receipt, samples that originate from the State of West Virginia are required to have the temperature of every bottle checked for preservation requirements. If a cooler temperature or bottle temperature requirement is not met, the affected samples must be tracked using the Discrepancy field on the inspection form. Each container must be listed from the cooler when out of temperature. See Section 9.0 for details of this procedure.

6.3.5 Samples are removed from coolers and lined up in order on carts with the Chain of Custody. When discrepancies occur between the Chain of Custody and the sample container labels, it is noted and the appropriate Account Manager is notified. The Account Manager then notifies the client. The client decides what information is correct. Corrections are noted in the discrepancy section of the Cooler Inspection Form. Corrected data is logged into the LIMS system.

6.3.6 Sample ID, date and time of collection is checked with the Chain of Custody versus sample container label. The login assistant will check the pH of all preserved water samples, with the exception of volatile organics and method 1664 (OG-HEM). These are checked at the bench by the analyst. In addition, the pH of unpreserved water samples submitted for the analyses listed in Table 7 will also be checked in order to assure that they were not inadvertently preserved. The pH for unpreserved samples must be 2<pH<10. The pH is checked by inserting a pipet into the sample and placing a drop of the sample onto pH paper of the appropriate range (see Section 3.2). A glass pipet is used for checking pH of organic parameters (8330) and plastic pipets are used to check inorganic parameters. The assistant will verify that the pH is acceptable. The LIMS will create and update the pH record for each sample container checked in the sample delivery group (L#). If there are pH exceptions to the default acceptance criteria, the assistant/analyst will edit the pH data in the LIMS Container Records, and forward the exceptions/discrepancies to the client representative. If additional preservative is added to the container, the amount, concentration, and adjusted pH shall be recorded in the comments field in Container Records table. If the pH is out of range, the sample ID and product of the sample are recorded in the ROR System sample discrepancy section of the Cooler Inspection Form as a discrepancy by login personnel. Prior to the adjusting

and storing of the samples, approval must be determined from the client through the Account Manager. The approval is noted, as well as how much, which type and lot number of the preservative was for the adjustment. Metals also requires date and time of the preservation to be recorded on the bottle and documented.

6.3.7 If a sample is received out of hold, or is improperly preserved, the client is notified and the sample is analyzed according to the client's request. The discrepancy is noted in the Cooler Inspection Form (Document Control #1957- see Figure 4) and in the LIMS system along with the client instructions. Minimum volume, container type, preservation and hold time are listed in Table 1-6. VOA Samples which are indicated on the COC as being unpreserved must have the "VOA-Unpreserved" product logged on the relevant samples with the analysis products.

6.3.8 If a single sample container of soil is received and multiple analyses including VOA are requested, a sub-sample for VOA analysis is removed by the VOA laboratory and placed in an appropriate sample container. This sub-sample for VOA analysis shall be taken prior to the sample container being opened for any other reason. Care is taken to eliminate as much headspace as possible in the new VOA sample container. This sub-sample is then handled, stored and otherwise treated the same as any other volatile sample.

6.3.9 The custodian will inspect all water samples collected in VOA vials for the acceptable levels of headspace. If any containers contain bubbles larger than 6mm, this information must be noted on the Cooler Inspections Form as a discrepancy.

6.3.10 When all information is correct, the samples are logged into the LIMS system. The LIMS automatically assigns a unique login number. See Sections 7.0 and 8.0 for logging procedures.

6.4 Electronic Checklist – Procedures for Using the PDA / Laptop / Notebook

This section describes the use of a PDA / Laptop or equivalent, (Section 3.8) to automate many of the cooler and sample checking procedures presented in Section 6.3. The system incorporates a barcode reader and a wireless connection to the LIMS, allowing the checklist to be completed in paperless mode. The screen displays an electronic facsimile of Figure 1, and the user is prompted to enter the information in a logical, step-wise manner. Staff must not attempt these procedures until they have been trained in the use of the PDA / Laptop or equivalent.

6.4.1 Turn the unit on and open a new receipt. The system will automatically assign a unique number for later tracing of the cooler and samples to a specific delivery group (B, P and final L number(s)).

6.4.2 Select the mode of delivery by pressing the appropriate radio button.

6.4.3 Go to “Add cooler information”

6.4.4 Scan the barcode that identifies the carrier’s airbill number.

6.4.5 Scan the barcode that identifies the cooler’s tracking number.

6.4.6 Remove the COC(s) and scan or record the COC number(s). (This can also be done later in the sequence if there are multiple COCs.). This scan of the COC will be included in the final report to our customer(s). It is important the scan be an accurate reflection of the COC as received. Therefore, maintenance of the scanning device is critical. No markings must be allowed to be introduced by the scanning process. For example, lines introduced by dust or debris on the scanner’s internal parts are not permitted. Careful and timely maintenance of the scanner is important. See Figure 2 for scanner maintenance and cleaning instructions.

6.4.7 Determine the cooler temperature with the IR Gun and enter temperature.

6.4.8 Complete the remaining checklist items using the keypad.

6.4.9 Note each exception or problem in the discrepancy files, and the preservation form. In addition, if no problems are found, note this on the form along with the pH paper lot number. (Document Control #1957, Figure 4)

6.4.10 Some samples will require priority due to short hold time or turnaround time. Use the “Priority” drop-down menu to select appropriate priority need. The short hold time may not be readily determined by information on the chain of custody. Tables 1 through 6 list hold times for each analysis. An abbreviated list is presented in Figure 3; this list is posted at the cooler inspection areas.

6.4.11 Proceed as in Section 6.3.5.

6.4.12 When you have finished this process, you may edit any checklist entry, add comments, or address discrepancy details at the login computer keyboard.

6.5 Sample Labeling

Labels for containers and mailing reports are then printed, label bottles, and put mailing report labels along with chain of custody form in folder.

6.6 Sample Storage

Samples are stored according to analyses. In the walk-in cooler (W-1) shelves are designated by departments. These identify the sample storage location for easy retrieval by the laboratory. Volatile samples (V-1) are stored in a separate refrigerator with the exception of waste samples. These are kept in the walk-in cooler or on a holding shelf in archive until analyses can be performed.

6.7 Internal Chain of Custody Procedures

If a client requests samples to be tracked throughout the lab, an ICOC form is generated through a computer program. "ICOC" prints out on the label. This label also contains a unique bar code. The analyst is responsible for scanning out the samples using his/her bar coded badges. The samples are then relinquished by the appropriate personnel in the login area. Other information as to the location of the sample, which department the sample is going to can be scanned from a bar coded template. When the analyst is ready to return the samples, it is scanned back into the computer by the relinquishing employee using his/her bar coded badges, one login personnel's badge and the location to which the sample is being returned (walk-in/archive/disposal). It is not always possible to have a custodian present when samples are removed from storage. The analyst will scan a special barcode designated for all "after hours" removal of samples from the login area storage unit.

6.8 Sample Disposal

After all analyses have been completed, the sample custodians will move the sample residuals to the archive storage units, where they will remain for the time specified in the client agreement. At regular intervals the sample custodian or other trained staff member will dispose of aqueous, solid, and organic-matrix samples in accordance with Microbac SOP 33 (Laboratory Waste Management). Alternatively, the laboratory may return selected sample residuals to the client.

6.9 Saturday Receipt of Short Hold Time Samples

It is the responsibility of the Account Managers and their assistants to notify the laboratory and the sample custodians if samples with short hold times (Figure 3) are to be received on Saturdays. The notification will be in the form of an email

addressed to "OVD-Headsup" which reaches all of the relevant personnel. Samples for which the laboratory has been notified of shipment do not require any specials actions on the part of the sample custodian receiving the cooler(s). In the case that unannounced short hold time samples are received on Saturday, it is the responsibility of the sample custodian to notify the affected department supervisor or designee by telephone so that the appropriate action can be taken to process the samples within the holding time.

7.0 OVERVIEW OF LOGGING PROCESS

This section describes the duties of the sales/service team, specifically the Account Managers and their assistants, as they pertain to logging of project and sample information into the LIMS. These duties are summarized below:

7.1 The sales/service department must enter account and project information into the LIMS as a prerequisite to preparing quotations and work-orders or to the actual logging of samples. The LIMS assigns unique serial numbers for each account and for the associate project.

7.2 Quotations (Q number)

Quotations are used to enter special pricing in the LIMS and are often included as part of the sales proposal. The LIMS assigns a unique serial number for each quotation. (Q-number)

7.3 Work-orders (B number)

The service team creates a work order upon new project award, or when sample containers are requested for an existing project. The work order includes sample, matrix and product information in sufficient detail to generate a packing list for sample kits and for the efficient logging of samples once received at the laboratory and to create templates for repetitive sampling events. The LIMS assigns a unique serial number (B number) for each work order.

7.4 Sample Pre-logging (P number)

Pre-logging is the preliminary process of logging samples into the LIMS either upon receipt of the samples, or when chain of custody forms are provided to the lab in advance of sample receipt. The LIMS assigns a unique serial number (P number) for each sample in the pre-log status, and information is subject to review and editing by the sales/service teams.

7.5 Final Logging (L number)

After the information in the pre-log number is reviewed and deemed accurate, the Sample Custodian or designated client representative will perform the final login procedure. In this step the LIMS assigns a unique serial number (L number) and generates sample labels for each container in the sample delivery group.

7.6 Other Data Entry

Most of the analytical data is entered into the LIMS via automatic data upload from the laboratory instrumentation, or through other LIMS tools such as electronic bench sheets used by the analyst. The custodian or Account Manager must employ manual data entry procedure for a few methods, primarily field data provided by the samplers. Examples include pH, dissolved oxygen, and conductivity.

7.7 Login Folder Review and Client Communication

The Client Representative or team chemist is responsible for accuracy of the information entered into LIMS for accounts, projects, and login numbers. This review must occur prior to release of the samples to the laboratory and is normally performed after the pre-log step of the process. The service team is also responsible for communication and resolution of any discrepancies identified at the time of sample receipt and inspection. Other duties of the service teams are presented in Microbac SOP 44 and Microbac SOP MISDATA01.

8.0 DETAILED LOGGING PROCEDURE

8.1 Log into the LIMS.

8.2 Go to Sample Management; Login; Login ID – enter the correct workorder (B number) or template (T number); Check Pre-Login and click OK.

8.3 Check all products in the matrix for each client ID. **DO NOT CHANGE ANY INFORMATION IN THE B# SCREEN.** Check all projects/products, etc. in this screen to make sure they match the chain of custody. **Read Login information in the top right corner for specific instructions.**

8.4 Go to Copy; Copy Template; Source Template B number; Pre-Login; Collect Date (Change date if needed); then click OK.

8.5 The pre-log (P number) Screen will come up at this time. This is the screen you edit if necessary. For example: If unpreserved VOA samples were received and

the B number does not include the "VOA-Unpreserved" code, it can be added to samples on this screen.

8.6 Place your cursor on the Client ID. Check the matrix below to see if it is correct against the chain of custody and the bottles lined up. If the matrix matches the chain of custody and the bottles, go back to the client ID.

- You may have to change the client ID to match the bottle or chain of custody ID.
- Tab over to the received date; enter 0 and tab for the current date and add the time received from the printed barcode label that the ROR system generates.
- Tab over to the collect date; the date should be filled in but you will need to add the time. If no time is given, default to 00:01.
- Tab over to the TAT; do not change this number; if the TAT is different from the chain of custody, put this information in the "Prelim" button, not the TAT.
- Tab to the end of the line checking for any errors and also to check comments and QC tags.

8.7 Move your cursor down to the next fraction (line) and start over with these instructions from Section 8.6. When tabbing to the receive and collect dates/times use the F3 key and the dates/times will copy from the above fraction ***or*** you can also "Copy Duplicate Fractions" or "Copy Products" by going to the Copy tab on the Toolbar, and clicking on one or the other. To delete fractions or products, shift F6.

8.8 If nothing else needs added or deleted go to Copy; order fractions; click on the Auto Calc due dates button and the F10 to save. This will automatically fill in the due dates and client dates. Click on the add prods button at the bottom of the page. Be sure to add prods on LAST fraction.

8.9 At this point, your folder which is now a P number needs to go through Peer Review or given to the client representative for review. When the folder is returned to you after review, you may make corrections if needed and then take it to an L number. Be sure to take care of discrepancies if needed.

8.10 In order to take a P number to an L number go to copy; source template P# _____; Login.

8.11 The L number screen will come up at this time. If a green button comes up at the top of the screen, click on it at this time.

8.12 Click on Reports; The L number will come up automatically at this time in the Login Reports screen; go to Login Labels; Printer; Go and your labels will print with

address labels attached. Watch for the ICOC on label. Use pen to color labels for help with archiving.

8.13 Label the bottles and put them away in their appropriate walk-in, refrigerator or freezer.

8.14 Place the client Chain of Custody form, and the extra client address labels in a manila master folder.

8.15 Place the client address label on the folder tab and in the bottom right hand side, on the front of the folder, facing out (sideways).

8.16 If the TAT is less than 7 days or these are short hold, make a note on the outside of the master folder to help in prioritizing review.

8.17 Folders are then taken to the Service team.

9.0 CALIBRATION AND QUALITY CONTROL

9.1 IR Gun Calibration

9.1.1 The IR Temperature Guns are calibrated once a year by Cole-Parmer and a certificate is supplied and are maintained in the QA office. The guns are calibrated quarterly (every 3 months) by Wet Lab personnel.

9.1.2 The calibration of the IR temperature guns are checked once daily, in the morning before use against the ROR system Quality Control probe thermometer in the 1005/WI walk-in cooler by Support Service personnel. Readings are recorded in the temperature log books. The IR gun must read within 0.5° C of the probe reading. If this is unsuccessful, the IR Temperature Gun is returned to the manufacturer for maintenance.

9.2 Coolers are received into the Login area. A temperature blank is removed and held near the IR Temperature Gun. (If a temperature blank is not available, any size container can be used.) A temperature reading is taken for each cooler or bottle if required, see 6.3.4.

9.2.1 The IR Temperature Gun is pointed at the bottom of the bottle. The trigger on the gun is pulled immediately and a reading appears on the readout of the gun. The temperature is then recorded in the Cooler Inspection section of the ROR.

9.2.2 If the temperature exceeds 6° C, the Account Manager is notified so that the client may be advised that their samples were received with the temperature out of range. All sample containers in a cooler with a temperature exceeding the 6ºC when required or received frozen must be listed in the Discrepancy section of the inspection report and on Form #1957(Figure 4). Clients will then decide if the samples are to be analyzed or not. (Acceptance criteria can be adjusted at the request of the client). If the samples are received frozen, then this is noted in the discrepancy section of the Cooler Inspection Form and the Account Manager is notified.

9.3 Procedure for Tracking When Cooler Temperature is not Within Guidelines

9.3.1 Login Staff

When cooler or bottle temperature is determined to be out of the regulatory guidelines:

1. Label each container from the cooler with a "Cooler Temp Out" or "Bottle Temp Out" sticker.
2. If the cooler is to be unpacked at a later time, label the cooler with a "Cooler Temp Out" sticker.
3. Place a "Cooler Temp Out" sticker on the front of the project folder to notify project management.

The temperature discrepancy still needs noted in the receipt and inspection forms.

9.3.2 Analysts

When analyzing a sample with a container labeled with a "Cooler Temp Out" or "Bottle Temp Out" sticker.

1. Indicate on your bench sheets and extraction logs the sample being analyzed was received out of regulatory guidelines with "CT1" in the comment section.

9.3.3 Data Reviewers and Supervisors

1. Data must be qualified at the analyte level for samples received in coolers with the temperatures outside of regulatory guidelines.
2. Address the temperature out of regulatory guidelines in the case narrative using the appropriate phrase below.

9.3.4 Case Narrative Comments (Helpful Hints)

1. The temperature at receipt was XX C, but samples were received within 15 minutes of collection.
2. The temperature at receipt was XX C, exceeding the regulatory guidelines for testing.
3. Although samples were on ice, the temperature at receipt was XXXC, exceeding the regulatory guidelines of 0°-10°C for microbiological testing.
4. Although samples were received on ice, the temperature at receipt was XX C, exceeding the regulatory guidelines of 0°-6°C for chemical testing.

9.4 Temperature of Storage Units

The temperatures of the walk-in cooler, archive walk-in, V1 storage refrigerator, and the F-1 freezer are checked every four hours via electronic temperature probes. This system sends an email alert to the login supervisor and staff if a temperature is out of range. In the event of a temperature excursion for the walk-in coolers, Microbac notes the discrepancy and takes appropriate actions to relocate the samples if the problem persists.

10.0 REFERENCES

10.1 Microbac SOP LQAP “Laboratory Quality Assurance Plan”

10.2 Microbac SOP RADSAFETY “Radiation Safety Program Manual”

10.3 Microbac SOP33 “Laboratory Waste Management”

10.4 Microbac SOP44 “Project Management, Technical Service and Subcontracting”

10.5 Microbac SOP MISDATA01”Data Entry, Data Review and Reporting”

Table 1

Sample Containers, Preservation and Hold Times
CONVENTIONALS - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Acidity	100	P, G	Cool, ≤ 6° C	14 Days
Alkalinity	100	P, G	Cool, ≤ 6° C	14 Days
Total Solids	50	P, G	Cool, ≤ 6° C	7 Days
Ash Content @ 750° C	25	P, G	Cool, ≤ 6° C	---
Biochemical Oxygen Demand	500	P, G	Cool, ≤ 6° C	48 Hours
BTU	10	P, G	Cool, ≤ 6° C	---
Formaldehyde	20	G	Cool, ≤ 6° C	3 Days
Chloride	25	P, G	Cool, ≤ 6° C	28 Days
Chloride, Total Residual	100	P, G	Cool, ≤ 6° C	6 Hours
Cyanide (midi)	50	P, G	Cool, ≤ 6° C, NaOH, pH>12	14 Days
Cyanide, Amenable to Chlorination (midi)	100	P,G	Cool, ≤ 6° C, NaOH, pH>12	14 Days
Chemical Oxygen Demand	25	P, G	Cool, ≤ 6° C, H_2SO_4, pH<2	28 Days
Color, Platinum-Cobalt	50	P, G	Cool, ≤ 6° C	48 Hours
Coliform, Fecal	120	Sterile P, G	Cool, < 10° C, $Na_2S_2O_3$	8 Hours
Coliform, Total	100	Sterile P, G	Cool, < 10° C, $Na_2S_2O_3$	8 Hours
Specific Conductance	100	P, G	Cool, ≤ 6° C	28 Days
Corrosivity (pH)	50	P, G	Cool, ≤ 6° C	---
Chromium, Trivalent (calc)	-	P, G	Cool, ≤ 6° C	---
Chromium, Hexavalent	150	P, G	Cool, ≤ 6° C	24 Hours
Dissolved Oxygen	300	G	Cool, ≤ 6° C	6 Hours
Fluoride	25	P, G	Cool, ≤ 6° C	28 Days
Ignitability	75	P, G	Cool, ≤ 6° C	---
Fluoride, Total (Distilled/Non-Distilled)	200	P, G	Cool, ≤ 6° C	28 Days
Hardness	100	P, G	Cool, ≤ 6° C, HNO3, pH<2	6 Months
Surfactants (MBAS)	100	P, G	Cool, ≤ 6° C	48 Hours
Coliform Fecal (MPN)	100	Sterile P, G	Cool, < 10° C, $Na_2S_2O_3$	8 Hours
Nitrogen, Ammonia (Distilled/Non-Distilled)	100	P, G	Cool, ≤ 6° C, H_2SO_4, pH<2	28 Days
Nitrogen, Nitrite	50	P, G	Cool, ≤ 6° C	48 Hours
Nitrogen, Nitrate	75	P, G	Cool, ≤ 6° C	48 Hours
Nitrogen, Nitrate-Nitrite	25	P, G	Cool, ≤ 6° C, H_2SO_4, pH<2	28 Days
Nitrogen, Organic (calc)	100	P, G	Cool, ≤ 6° C, H_2SO_4, pH<2	28 Days
Oil and Grease	1000	G	Cool, ≤ 6° C, HCl, pH<2	28 Days

1. P = Polyethylene (preferred when acceptable)
2. G = Borosilicate glass with Teflon lined cap
3. For more current list of method and preservations, see LIMS tables, Products/Containers

Table1 (continued)
Sample Containers, Preservation and Hold Times
CONVENTIONALS - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Phenolics, Total	100	Amber, G	Cool, ≤ 6° C, H_2SO_4, pH< 2	28 Days
Phosphorus, Total	50	P, G	Cool, ≤ 6° C, H_2SO_4, pH< 2	28 Days
pH Lab	50	P , G	Cool, ≤ 6° C	6 Hours
Orthophosphate	50	P, G	Cool, ≤ 6° C	48 Hours
Reactivity, Cyanide	10	P, G	Cool, ≤ 6° C	---
Reactivity, Sulfide	10	P, G	Cool, ≤ 6° C	---
Sulfite	50	P, G	Cool, ≤ 6° C	6 Hours
Settleable Solids	1000	P, G	Cool, ≤ 6° C	48 Hours
Sulfate	25	P, G	Cool, ≤ 6° C	28 Days
Specific Gravity	50	P, G	Cool, ≤ 6° C	---
Total (Organic) Sulfur	10	P, G	Cool, ≤ 6° C	---
Sulfide	500	P, G	Cool, ≤ 6° C, Zinc Acetate, NaOH, pH> 9	7 Days
Total Dissolved Solids	50	P, G	Cool, ≤ 6° C	7 Days
Total Suspended Solids	200	P, G	Cool, ≤ 6° C	7 Days
Turbidity	50	P, G	Cool, ≤ 6° C	48 Hours
Volatile Dissolved Solids	50	P, G	Cool, ≤ 6° C	7 Days
Total Volatile Solids	50	P, G	Cool, ≤ 6° C	7 Days
Volatile Suspended Solids	200	P, G	Cool, ≤ 6° C	7 Days

1. P = Polyethylene (preferred when acceptable)
2. G = Borosilicate glass with Teflon lined cap
3. For more current list of method and preservations, see LIMS tables, Products/Containers

Table 2
Sample Containers, Preservation and Hold Times
VOLATILE ORGANICS (VOA) - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Gasoline Range Organics	40 mL	G, Septa Caps	Cool, ≤ 6° C, HCl, pH< 2	14 Days
Volatile Aromatics	40 mL	G, Septa Caps	Cool, ≤ 6° C, HCl, pH< 2	14 Days
Volatile Organics (VOA)	40 mL	G, Septa Caps	Cool, ≤ 6° C, HCl, pH< 2	14 Days
VOA – Method 624	40 mL	G, Septa Caps	Cool, ≤ 6° C	7 Days
VOA – Method 624 (chlorinated) *	40 mL	G, Septa Caps	Cool, ≤ 6° C, $Na_2S_2O_3$	7 Days

*Provided upon client request when samples contain chlorine.

Table 3
Sample Containers, Preservation and Hold Times
SEMIVOLATILE ORGANICS - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Diesel Range Organics	1000 mL	G	Cool, ≤ 6° C	7 Days
Pesticides/PCBs	1000 mL	G	Cool, ≤ 6° C	7 Days
Polyaromatic Hydrocarbons	1000 mL	G	Cool, ≤ 6° C	7 Days
Herbicides	1000 mL	G	Cool, ≤ 6° C	7 Days
EDB/DBCP	3.40 mL	G	Cool ≤ 6° C	7 Days
Semivolatile Organics	1000 mL	G	Cool, ≤ 6° C	7 Days

* Sodium thiosulfate is added for DE chlorination when Method EPA 608 is requested.
1. P = Polyethylene (preferred when acceptable)
2. G = Borosilicate glass with Teflon lined cap
3. For more current list of method/preservatives, see LIMS tables, containers and product

Table 4
Sample Containers, Preservation and Hold Times
METALS - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
All Metals (26)	500 mL	P, G	HNO_3, pH< 2	6 Months*
Mercury	50 mL	P, G	HNO_3, pH< 2, ≤ 6° C	28 Days
Furnace Metals	100 mL	P, G	HNO_3, pH< 2	6 Months

Table 5
Sample Containers, Preservation and Hold Times
TCLP - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
TCLP Volatiles	100 mL	G	Cool, ≤ 6° C	14 Days
TCLP Semi-Volatiles	100 mL	G	Cool, ≤ 6° C	14 Days
TCLP Pesticides	100 mL	G	Cool, ≤ 6° C	14 Days
TCLP Herbicides	100 mL	G	Cool, ≤ 6° C	14 Days
TCLP Metals	100 mL	P, G	Cool, ≤ 6° C	6 Months*

* For (1) TCLP parameter 100 mL required; for full TCLP (2) 1000g
* Mercury is 28 days

NOTE:
1. P = Polyethylene (preferred when acceptable)
2. G = Borosilicate glass with Teflon lined cap
3. Triple the volumes above for MS/MSD samples

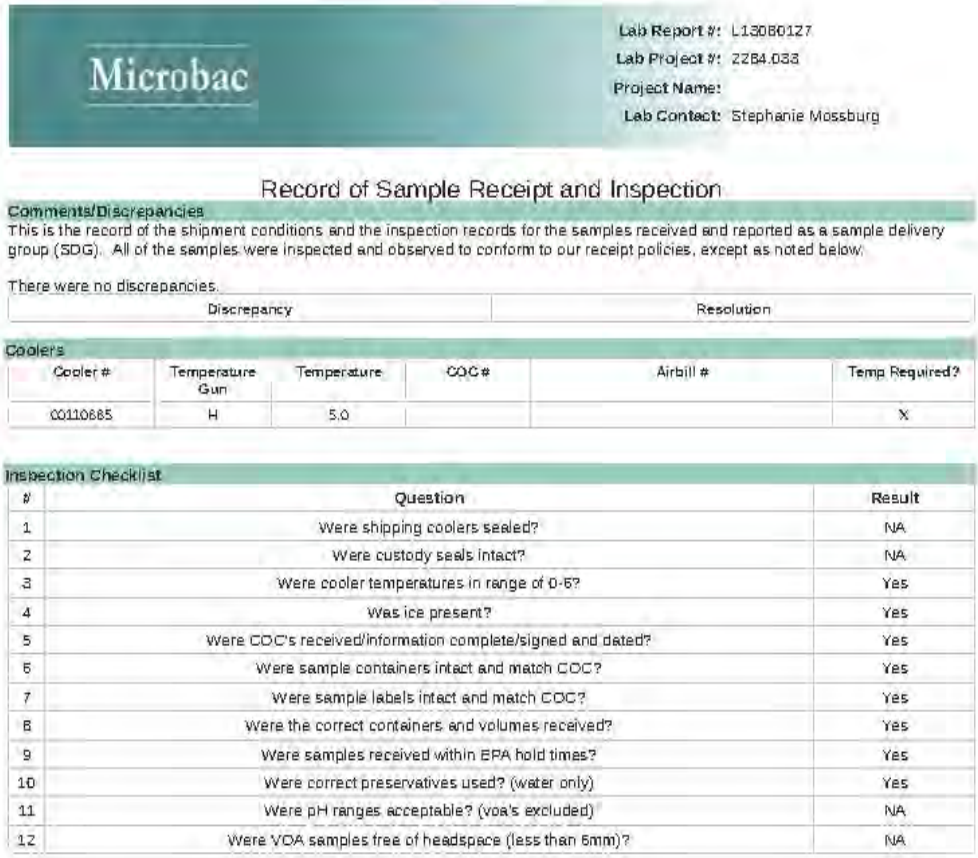
Table 6
Sample Containers, Preservation and Hold Times
SOIL

METHOD	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Coliform Fecal	1g	G	Cool, < 10° C	8 Hours
Chromium, Hexavalent	50g	G	Cool, ≤ 6° C	24 Hours
5035	5 g	P, G	Cool, ≤ 6° C	48 Hours
TCLP-VOA	105g	G	Cool, ≤ 6° C	14 Days
TCLP-SV	105g	G	Cool, ≤ 6° C	14 Days
TCLP-Pest/Herb	105g	G	Cool, ≤ 6° C	14 Days
TCLP-Metals	105g	G	Cool, ≤ 6° C	6 Months *
Total Metals (except Hg)	3g	G	N/A	6 Months
Hg	2g	G	Cool, ≤ 6° C	28 Days
DRO	30g	G	Cool, ≤ 6° C	28 Days
Semi-Volatiles	30g	G	Cool, ≤ 6° C	14 Days
Herbicides	50g	G	Cool, ≤ 6° C	14 Days
Volatiles	1g	G	Cool, ≤ 6° C	14 Days
Conventionals (where applicable)	1g – 100g	G	Cool, ≤ 6° C	14 Days
Petroleum Hydrocarbons	30g	G	Cool, ≤ 6° C	14 Days
Percent Moisture	25 g	P, G	Cool, ≤ 6° C	---
Percent Solids	25 g	P, G	Cool, ≤ 6° C	---
Paint Filter Liquids Test	100 g	P, G	Cool, ≤ 6° C	---

Table 7
Analyses Requiring Unpreserved pH Checks

Test	Method
Alkalinity	SM 2320B
Alkalinity, Automated	EPA 310.2
Acidity	SM 2310B
Chloride	SM 4500CL E
Ferrous Iron	SM 3500FE B
Sulfate	SM 4500S04 E
	EPA 375.4
Solids, Total Dissolved, TDS	SM 2540C
Residue, Volatile	EPA 160.4
Residue, Total TSS	SM 2540B
Residue, nonfilterable TSS	SM 2540D
Reside, Settleable	SM 2540F
IC Anions	EPA300.0
	EPA 9056
Conductivity	SM 2510
	EPA 120.1
MBAS	SM 5540C
Turbidity	SM 2130B
	EPA 180.1
Fluoride, Electrode	SM 4500FC
Chlorine	SM 3400CL G
Perchlorate	EPA 6850
Nitroaromatics/Nitroamines	EPA 8330
Acetate/Formate	SOP- HPLC12
Fluroborate	SOP-K9305

Figure 1



Microbac

Lab Report #: L13080127
Lab Project #: 2284.038
Project Name:
Lab Contact: Stephanie Mossburg

Record of Sample Receipt and Inspection

Comments/Discrepancies

This is the record of the shipment conditions and the inspection records for the samples received and reported as a sample delivery group (SDG). All of the samples were inspected and observed to conform to our receipt policies, except as noted below:

There were no discrepancies.

Discrepancy	Resolution

Coolers

Cooler #	Temperature Gun	Temperature	COC #	Airbill #	Temp Required?
C0110685	H	5.0			X

Inspection Checklist

#	Question	Result
1	Were shipping coolers sealed?	NA
2	Were custody seals intact?	NA
3	Were cooler temperatures in range of 0-6?	Yes
4	Was ice present?	Yes
5	Were COC's received/information complete/signed and dated?	Yes
6	Were sample containers intact and match COC?	Yes
7	Were sample labels intact and match COC?	Yes
8	Were the correct containers and volumes received?	Yes
9	Were samples received within EPA hold times?	Yes
10	Were correct preservatives used? (water only)	Yes
11	Were pH ranges acceptable? (voa's excluded)	NA
12	Were VOA samples free of headspace (less than 6mm)?	NA

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Page 2

L13080127 / Revision: 0 / 8 total pages

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Figure 2

MICROBAC®

Ohio Valley Division

Cleaning the Scanner

Cleaning procedure

To clean all the parts listed below use glass cleaning wipes that are individually packaged. Using the wipe remove the debris. It may be necessary to remove large chunks of toner material and dust from the rollers. If so gently scrape the material off being careful not to damage the rollers.

Parts that require cleaning

1) Rollers

- Open the top compartment of the paper feed for the scanner.
- Locate the white colored rollers on the top of the open compartment door.
- These should be completely white without any black streaks or spots on them.
- Toner can buildup here and cause lines to form on the scans.

2) Glass

- Open the door to the scanning bed.
- On the left side of the scanning bed there is a narrow strip of glass.
- This glass must be completely free of debris / dust / detritus.
- Imperfections on the glass will cause lines to appear on the scan.

3) Additional Preventative Maintenance

If you look on the top of the lid above the glass you cleaned in step 2 you will see a thin white strip of material. This material must remain free of dust and imperfections. The smallest bit of dust can be picked up by the scanning bed and turned into lines on the scan.

A can of compressed air should be used periodically to clean out paper dust and debris. IT or office staff maintain a supply for use by the lab.

If you require assistance please contact the IT support staff.

Document Control Form

Figure 3

Ohio Valley Division MICROBAC®

Water
Short Hold Times

6 Hour	24 Hour	48 Hour
Fecal / MPN*	CR-6	BOD
CI-TRL	I-	Color
DO-L	-	NO3
pH-L	-	NO2
SO3	-	PO4 (orthophosphate)
-	-	Set-5
-	-	Turbidity
-	-	MBAS
-	-	9056 (NO3, NO2 or PO4)
-	-	300

* 8 hour hold time

Additional Priorities for Short Hold Times

Volatiles 7 Day	Semivolatile Waters with 3 days or less remaining of hold time	
Unpreserved 624 or 8260	DRO	8015
RSK175	Pesticides	8081, 608
-	PCBs	8082, 608
-	PAHs	8270
-	Herbicides	8151
-	Semivolatile	8270
-	Formaldehyde	-
-	TDS	-
-	TSS	-
-	TVS	-
-	Total Solids	-

Figure 4

MICROBAC®

COOLER TEMP >6° C LOG

Cooler ID_____

SAMPLE ID	Bottle 1 °C	Bottle 2 °C	Bottle 3 °C	Bottle 4 °C	Bottle 5 °C	Bottle 6 °C

pH Exceptions

pH Lot #_____

SAMPLE ID	Bottle 1	Bottle 2	Bottle 3	Bottle 4	Bottle 5	Bottle 6

Document Control # 1957
Last 10-07-2016

Issued to: Document Master File

ALS Standard Operating Procedure

DOCUMENT TITLE:	SAMPLE RECEIVING, ACCEPTANCE, AND LOGIN
REFERENCED METHOD:	N/A
SOP ID:	SMO-SMPL_REC
REV. NUMBER:	16.0
EFFECTIVE DATE:	04/09/2016

ALS Environmental

SAMPLE RECEIVING, ACCEPTANCE, AND LOGIN

SOP ID:	SMO-SMPL_REC	Rev. Number:	16.0	Effective Date:	04/09/2016

Approved By: _____ Date: 3/22/16
Sample Management Custodian – Krystal Kelpe

Approved By: _____ Date: 3/24/16
QA Manager – Chaney Humphrey

Approved By: _____ Date: 3/20/16
Laboratory Director – Kelly Horiuchi

Archival Date:	_____	Doc Control ID#:	Non-Controlled	Editor:	_____

TABLE OF CONTENTS

DATA AND RECORD ARCHIVING

1) Scope and Applicability

1.1 The purpose of this standard operating procedure (SOP) is to describe the requirements and guidelines necessary for effective sample receiving as well as the documentation associated with this process. Additionally, this document describes the procedures relating to the Sample Management Office for initiating any subcontract documentation.

1.2 This standard operating procedure (SOP) is applicable to all samples delivered to this laboratory and subcontracted out for analysis.

2) Summary of Procedure

2.1 For the purposes of this document sample receiving is considered to be an all-inclusive system, which comprises sample custody transfer, sample acceptance, and sample login.

2.2 This procedure is essential in identifying compromised samples and ensuring the validity of the laboratory's sample data. Improper sample handling affects the credibility and acceptability of analytical results, regardless of their accuracy and precision. Therefore, it is essential that all samples be properly received and handled and that the documentation maintained accurately reflects the integrity and processing of samples.

3) Definitions

3.1 Custody The guardianship or safe keeping of a sample. A sample is considered to be in a person's custody if it is physically in their possession, or it is in their view after being in their possession, or it was in their possession and then locked up or sealed to prevent tampering, or it is in a secure area.

3.2 Chain of Custody (COC) Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses.

3.3 Internal Chain-of-Custody Procedures employed to record the possession of samples from the time of sample receipt until disposal/storage and are performed at the special request of the client. These protocols are handled electronically through LIMS.

3.4 Compromised Samples Those samples which are improperly sampled, insufficiently documented, improperly preserved, collected in improper containers, exceeding holding times and/or not received intact when delivered to a laboratory.

3.5 Holding Times (Maximum Allowable Holding Times) The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid or not compromised. (40 CFR Part 136)

3.6 Preservation Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

3.7 Service Request (SR) / Job File A unique, computer generated laboratory number which is assigned to a sample or group of samples submitted (at the same time) by the client representing one job or project. The job or project includes specific sample management information, analysis data, client correspondence, analysis report and

other pertinent information comprising a single sample submission containing one or more samples in a client's project.

3.8 COC Chain-of-Custody

3.9 SACF Sample Acceptance Check Form

3.10 LIMS Laboratory Information Management System

3.11 SMO Sample Management Office

3.12 PM Project Manager (may be referred to in other lab documents as PC/Project Chemist)

3.13 SMC Sample Management Custodian

3.14 SDG Sample Delivery Group

3.15 EDD Electronic Data Deliverable

4) Health and Safety Warnings

4.1 Handle all samples as potentially hazardous. Gloves should be worn when handling all samples, safety glasses, and a lab coat shall be worn when handling liquid or soil (solid) samples. Always work under a hood when chemically preserving samples. Also place broken or leaking samples under the hood. Get assistance when confronted with any situation that appears to be dangerous.

4.2 In the event of broken liquid or soil samples, SMO needs to cleanup using one of the following procedures:

- Liquids: Broken glass is handled carefully using disposable gloves and disposed of in the Glass Disposal Box. Remaining sample and cleanup materials are disposed of in accordance with the *SOP for Waste Disposal*.
- Soils: Broken glass is disposed of in the Glass Disposal Box, and the soil is disposed of into the 55-gallon soil drum. This information is noted on the Service Request Form and the PM is notified. Soil that is still intact in a glass jar may be salvaged with client's approval.

5) Personnel Qualifications and Responsibilities

5.1 All employees involved with sample receiving, acceptance and login must ensure the procedures described in this document are followed. More specifically, SMO personnel, Project Managers and the Sample Management Custodian are responsible for complying with and implementing the procedures listed in this document.

6) Procedure

6.1 Upon sample receipt, the condition, including any abnormalities or departures from normal or specified conditions as described in the test method or method standard operating procedure must be recorded. All of the information including any other observances must be recorded on the Sample Acceptance Check Form (Attachment 2) and other associated documentation as detailed in the following procedures. Refer to Section 6.4 for the necessary procedures and documentation requirements dictated by abnormalities or departures.

6.2 Sample Custody

Upon delivery to the laboratory, the sample(s) must be transferred (as soon as possible) to a Sample Management Custodian (SMC) or a representative of the

laboratory who accepts and assumes custody of the sample(s). Samples are transported to the laboratory by a number of means including courier, common carrier, sampler or client representative. The acceptance of a sample is achieved by presenting a signature, date and time of receipt in accordance with the requirements of the transmitter and client such as an electronic board (i.e. FedEx) and Chain of Custody Form. Sample shipping containers are examined for the presence and condition of custody seals, locks, shipping waybills, etc. After opening shipping containers, remove any other documents in order to evaluate login priority (see note below) and continue processing the samples.

Note: Rush requests and samples with short holding times are given top priority for processing. Sample Custodian alerts Project Manager and analysts by calling them and distributing copies of the COC and any other pertinent documentation. Refer to Appendix F in the Quality Assurance Manual for Sample Preservation and Holding Times which list maximum allowable hold times.

6.2.1 Shipping Receipts and Chain of Custody (COC) Forms

6.2.1.1 Packing Slips

A copy of the packing slip must be kept, whenever possible, as part of the permanent chain of custody process and placed in the job file.

6.2.1.2 Chain of Custody Forms

These forms may be identical to the one issued by the laboratory (see Figures 11-1 and 11-2 in the Quality Assurance Manual) or clients may submit samples using a similar form. The SMC or designee shall sign the COC and add the date and time of receipt. In addition, the service request number must be added to the COC form at the time of sample login.

6.2.2 Legal/Internal Chain of Custody (COC)

When samples are logged in using LIMS, the system automatically generates an internal chain of custody each time a sample is scanned into possession for use within the laboratory. This internal COC may be accessed anytime during the laboratory procedures and is provided to the client upon request.

6.3 Sample Receipt and Login

In order to evaluate the state of a sample upon receipt, the laboratory must evaluate certain parameters including container type, volume and preservation (thermal and chemical). Compare the findings against the specified criteria in Sample Preservation and Holding Times Tables in the most recent Quality Assurance Manual (Appendix F). Refer to Section 6.4 for the discrepancy/exception and the rejection of samples procedures.

Important: For odorous samples, refer to Section 6.8.1 for the handling procedure.

6.3.1 Service Request Form

A Service Request form (Attachment 3) shall be completed in LIMS for all samples received by the laboratory using the information provided on the sample receipt documentation (e.g., COC) and data collected by the SMC. A copy of this completed form shall accompany the sample(s). The following includes a description of the key components.

1. Service Request Number: Client's job file number (automatically assigned)
2. Report Name: Name of Client that shall be on report.

3. Reporting Address: Address of the Client that will be on the report.
4. Project Name: Client's referenced study or project name.
5. Project Number: Client's reference study or project number.
6. ISR Number (if applicable): Internal Service Request (between laboratories in the network using the same LIMS system)
7. Date Received: Date the laboratory actually received samples.
8. Purchase Order: Client's purchase order number or verbal notation (default).
9. Project Manager: The PM responsible for all client activity for job file.
10. TAT: Sample turnaround time (normal TAT, if not specified).
11. Initials: Initials of SMC or alternate logging in the sample(s).
12. Sample Type: Type/container of sample submitted by client.
13. Comments: Any comments concerning the sample or samples being submitted including short hold times.
14. Tier: QC level if one is given on the ISR or COC.
15. EDD: If EDD is required or not.
16. Method: Specified method for the samples to be analyzed.
17. Sample ID: Client's specified sample identification.
18. Test(s) Required: Number of methods for analysis on the samples.
19. Date Collected: Sampling date for each sample.
20. Time Collected: Sampling time for each sample.
21. Sample Type: Sample matrix for each sample.

Note: Some of the information (client's project name or number) may not be provided and will not be included on the form.

6.3.2 LIMS Login

Prior to sample arrival, the Project Manager may create a sample delivery group (SDG) in LIMS based on project information and in accordance with the *SOP for Project Management*. Analysis information associated with each sample is stored in this SDG. When samples arrive, the custodian uses this SDG as a template to create a job folder specific to the samples received. The custodian could either manually search SDG information from LIMS or find it by scanning the barcode of the bottle order form (also known as Bottle Order \ Sample Supplies Summary form).

Once the correct SDG has been selected, a sample template is chosen from the SDG template that best matches the analyses stated on the COC for each sample included on the COC. Once all the samples are chosen the custodian creates a unique job folder. Job folder is then edited as necessary (e.g., project name and number, date and time of sample collection, and client sample IDs).

Each sample container for a sample is given a unique lab code by the LIMS system. This lab code is express in the format of PYYJJJJ-sss.ccc.

Where:

"P" is the current lab ID code for Simi Valley,
"YY" is the two-digit year code (e.g., 15 for Y2015),
"JJJJJ" is the five-digit job number (e.g., 00001 for the first project),
"sss" is a three-digit sample ID number;
"ccc" is the three-digit container ID number.

An example for the second container of the first sample for the first job of year for 2015 would be P1500001-001.002. The alphanumeric code before the dash is the job number, the number after the dash is sample ID and the number after the period is container ID.

6.3.3 Sample Acceptance Check Form The SMC shall complete and generate a Sample Acceptance Check form (Attachment 2) based on the information specified in this section. This form is given to the PM and electronically accessible so that Chemists may input additional preservation check information.

Once the samples have been checked and the SACF produced, the form is to be saved at G:\\STARLIMS\Sample Acceptance Check form (as SR#_Client_Project) so that additional information such as pH may be added.

6.3.3.1 Sample Acceptance Policy Sample containers are removed and organized according to the COC identification and analyses. The sample conditions are checked to ensure sample integrity has not been compromised. These steps are listed to complete the criteria for the acceptance or rejection of samples but they do not necessarily occur in this order. Each point is an evaluation requirement which must be used to complete the Sample Acceptance Check form.

- Sample submission documents are properly used, fully completed (in ink) and shall include the client, sample identification, project name or location, date and time of collection, collector's name, sample type, preservation type (if applicable) and any special remarks concerning the sample.
- Proper sample labeling is considered: unique sample identification (ID), durable labels (labels that are not easily removed) and the use of ink.
- Sample containers checked for integrity (broken, leaking, Tedlar® bags are received flat, under inflated or with the valve open, Summa canisters are received under an unacceptable vacuum or with the valve open, etc.). Reject samples with broken or leaking containers.
- Sample container labels and/or tags agree with the sample documentation (ID, required analyses, etc.).
- Adherence to specified holding times (see Appendix F in the Quality Assurance Manual)
- Appropriate containers (size, type) are received for the requested analyses (see Appendix F in the Quality Assurance Manual).
- Proper temperatures of sample containers, if applicable (see Appendix F in the Quality Assurance Manual).
- Adequate sample volume (see Appendix F in the Quality Assurance Manual)
- Assessment of proper sample preservation, where applicable (see Appendix F in the Quality Assurance Manual). Reject samples preserved with the inappropriate preservatives for which the

requested analysis has been compromised (e.g., cyanide samples preserved with acid).
- Any notation made by other persons accepting the sample and any evaluations made and noted on the associated documentation.

Once the samples have been checked against the Sample Acceptance Policy, the sample custodian must generate a Sample Acceptance Check form, sample identification labels, and Service Request form (optional). The Project Manager is responsible for generating and emailing the Sample Receipt Acknowledgment form (Section 6.5) if requested. The sample login forms and labels must be completed to properly track laboratory samples.

6.3.3.2 Measurement of Temperature The temperature of all coolers containing samples requiring thermal preservation shall be taken using a verified thermometer calibrated against NIST standards and the data recorded (with correction factor applied) on the Sample Acceptance Check form (Attachment 2).

A reading shall be taken by placing the thermometer in the cooler so as to give an accurate reflection of the cooler temperature (i.e. not directly on ice or blue ice and at approximate sample level or in the temperature blank, if supplied). The lid must be closed to allow enough time for the thermometer to reach equilibrium (i.e., a minimum of five minutes) before the temperature reading is taken and recorded. The arrival temperature check is considered acceptable if the following is adhered to:

- Samples have a temperature of +/-2˚C of the required temperature or the method specified range; or
- Samples with a required temperature of 4˚C have a temperature ranging from just above freezing of water to 6˚C; or
- IMPORTANT: The US EPA has published revisions to the Code of Federal Regulations at 40 CFR 136 and 40 CFR 141. These revisions, known as the Method Update Rule (MUR), became effective 4/11/07 and contains a revised approved methods tables and temperature requirements. A number of the methods have been updated and for those methods the temperature requirement has been updated to ≤6˚C. Refer to Appendix F in the most recent Quality Assurance Manual for the specific methods that are affected.

Note: Samples that are hand delivered to the laboratory immediately following collection may not meet these criteria. This is considered acceptable if there is evidence that the chilling process has begun such as arrival on ice. Include a notation on the Sample Acceptance Check Form.

6.3.3.3 Chemical Preservation A pH measurement may be required on certain tests, the pH value shall be documented on the Sample Acceptance Check Form. Perform this check in accordance with the applicable method SOP and the *SOP for Laboratory Storage, Analysis, and Tracking.*

The pH of the sample shall be checked with a narrow ranged pH indicator strip (preferable). Take a small aliquot of the sample with a transfer pipette and place a few drops onto the pH indicator strip. Ensure that a new pipette is used for every sample container to prevent

cross-contamination. Refer to Section 6.7 on specific information for subcontracted jobs.

6.3.3.4 Headspace Check for headspace in VOA vials. Pay close attention to samples that are opaque; bubbles may not be easily observed. Samples with heavy sediments may stick to the vial, making it appears to have no bubble when the vial is inverted. Any bubble in the sample should not exceed 5-6 mm.

6.3.3.5 Reusable media The pressure of each Summa canister and glass bottle shall be checked and recorded to ensure the sample has the appropriate volume. Initial and final pressures are noted on the Service Request Form and on the back of the sample tag. Refer to the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters* for additional details.

At the time of sample submission, ambient air sampling canisters will likely have a vacuum (negative pressure). If the canister has a negative pressure, the gauge will read in inches of Mercury (inHg) or pounds per square inch (psig) depending on the gauge used. If the reading is inHg, the value must be converted to psig (A conversion chart may be used and is located in the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters* Attachment B). Vacuum readings entered in inHg to the system will be automatically converted to psig.

Returned canisters and glass bottles that are not samples are logged in and handled following the same procedures. Canisters received at an initial pressure *lower than* -9.8 psig (-20.0 inHg) are shelved on a canister rack outside SMO (P-101) for the canister department to clean. Canisters received that have an initial pressure *higher than* -9.8 psig (-20.0 inHg) are placed on a canister rack in SMO for screening before they are returned to the canister department for cleaning. This procedure must be performed in accordance with the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters.*

PUFs and PUF/XAD cartridges that are not samples are logged in according to their etched ID and the analysis is marked cancelled.

Thermal desorption tubes that are not samples are logged in according to their bar code ID and the analysis is marked cancelled.

6.3.3.6 Sample Login Contingency Plan This section is designed to detail the sample custody and receipt procedure for samples that are delivered to the laboratory late in the day or when the SMC or designee is not present. If sample(s) are delivered under thermal preservation, laboratory personnel shall evaluate the cooler temperature per Section 6.3.3.2. The temperature must be noted on the COC form along with the date and initial of the person making the notation. Refer to the Quality Assurance Manual for information on preservation requirements, which are listed by method and sample type. The person, following acceptance, evaluation and analysis (if performed), should place the samples in the appropriate storage location in accordance with the *SOP for Laboratory Storage, Analysis, and Tracking* and submit the paper work to SMO in order for the login process to be completed.

6.3.3.7 Short Hold Times When samples are delivered to the laboratory with little remaining on the hold time it may be possible for the analysis to

proceed prior to the login process. The following are circumstances where this is allowed.

- Tedlar bag samples only
- If there is no time for sample(s) evaluation and login prior to hold time expiration and an analyst is able to analyze the sample(s) immediately.

However, there are requirements that must be followed by the analyst(s) if the samples are to be analyzed prior to sample login.

- At a minimum, the analyst shall review/compare the chain of custody with the samples received to ensure that the sample identifications, etc. are correct.
- It is imperative that the client sample ID be referenced on all laboratory analytical documentation.
- Also, the analyst should check the integrity (i.e. leaking or flat Tedlar bag) of the samples and make any notations on the associated documentation.
- Additionally, once the samples have been analyzed they are to be immediately delivered back to SMO for the sample acceptance and login procedures detailed in this SOP.

6.3.3.8 Sample Identification Labels After samples have been logged into the computer and the lab ID assigned, the SMC shall print labels for each sample container received. Each computer-generated label is affixed to the appropriate sample container, where possible. Certain sample containers, such as solid adsorbent cartridges, are placed in a sealed bag identified with the job number and all the laboratory ID codes associated with each sample in the bag.

6.3.3.9 Sample Login/Labeling Verification After labels have been applied to the corresponding sample containers they should be verified by a second person to ensure proper labeling. Place all associated documentation into the job file and submit to the Project Manager.

- Once the documentation has been generated and the labeling verification has been performed, the custodian must complete the first section of the LIMS Sample Login Verification Form (Attachment 4).
- The Project Manager responsible for the project verifies login information. This process is documented on the LIMS Sample Login Verification form. It is only after this secondary review that the job folder is released out of the login console to the job in progress area, making the analysis information available to the analysts.

6.4 Discrepancy / Sample Rejection Procedures

Any discrepancies or concerns are noted on the Sample Acceptance Check Form (per Sample Acceptance Policy, see Section 6.3.3.1) and immediately communicated to the appropriate Project Manager. If and when there is any doubt as to the suitability of a sample to be tested such as a leaking valve, broken container, etc. the SMC shall inform the PM. Regardless of the discrepancy, the PM shall be responsible for coordinating all correspondences and consulting with the client for further instructions before the laboratory may proceed. However, when there are short holding time

constraints, the laboratory may complete the sample analysis, where possible for all samples in the client's job file including the sample in question.

6.4.1 Chemical Preservation for Water and Soil Samples Contact the PM and if the PM approves adding preservative to bring sample within the proper range, be sure to record the specific sample container identifications, preservative added, including type, lot number(s), and final pH on the Sample Acceptance Check form (Attachment 2) (even if subcontracting). Refer to Section 6.7 for information on sub-contracting and splitting samples, where appropriate. When chemical preservation is performed in the laboratory the Preservative Tracking Log (Attachment 2, *SOP for Media Request Fulfillment*) must be utilized for documentation purposes.

6.4.2 Login Revisions Changes to SR forms may be made by anyone authorized for sample login and Project Management capabilities; however, it is recommended that whenever possible documentation of the reasons for the changes and the person making those corrections is documented and any copies of the original must be retained and marked as obsolete.

6.5 Sample Receipt Acknowledgment

An acknowledgment form (Attachment 5) may be accessed and emailed to the client, along with a PDF of any other requested documentation.

6.6 Job File and LIMS Documentation

The sample documentation shall be maintained in each client's job file in accordance with current procedures and shall at a minimum include:

- Original chain of custody form (if utilized) with the laboratory job number
- Service Request Form
- Preservation Tracking Log, if utilized
- Sample Acceptance Check Form
- Sample Login Verification Checklist
- Any documentation including memos or transmittal forms, which are transmitted to the laboratory by the common carrier, courier, sampler, or client.
- Any internal documentation which is pertinent to the handling and/or analysis of the samples.

Note: The original and all copies and revised versions of documentation must be kept in the associated job file.

Once the samples have been received, accepted (or rejected) and logged into the laboratory system, a job file (referencing the corresponding service request number) must be created and all receipt, acceptance and login documentation included. The COC is to be scanned into a PDF and attached to the LIMS job file. The job file must be submitted to the appropriate Project Manager for approval. The job file will be kept in a designated area for the inclusion of all the remaining documentation for the project including analytical data, invoices, etc.

6.7 Sample Transfer between Laboratories

The following must be adhered to for all samples, extracts, digestates and split samples that are transferred, carried or shipped from one laboratory to another (between In-Network laboratories and to laboratories outside of the Network). Samples are generally prepared for shipping by packing bubble wrapped glass containers in a cooler filled with blue ice (or ice). Custody seals are signed and dated and placed on the front of the cooler. The cooler is then sealed with packaging tape. For specific

information on sample transfer to the off site preparation facility, refer to the *SOP for Laboratory Storage, Analysis, and Tracking* (Also, refer to Section 6.7.1.3).

Samples not analyzed at the laboratory are subcontracted to pre-approved laboratories (internal and or external). Samples are logged in for the required tests and assigned a subcontract lab (as assigned by the PM in the SDG, by flagging the team column of the folder with the appropriate sublab). A subcontract COC is printed from LIMS once the login has been completed. The subcontract COC is then placed in the job folder after a copy of document is made.

Note: If LIMS does not have the appropriate test or sub-contract laboratory code, a Request for Test Code or "Sublab" form is filled out and submitted to Kelso IT. In addition, if the sublab is not specified in the SDG, it will automatically be flagged and a subcontract lab must be selected. Contact the PM, if this occurs.

6.7.1 In-Network Sample Transfer This laboratory, when transferring samples to an In-Network laboratory, could either initiate a new chain of custody record or use a photocopy of the original chain of custody record. The SR number from the originating laboratory may remain the same when subcontracting to a laboratory within LIMS; and any documentation generated by the laboratory would be included in that job file.

6.7.1.1 A new chain of custody record may be initiated if the number of samples or analyses is small enough so that it is not too time consuming to write out the new chain of custody record. The sample custodian at this laboratory must accurately transfer the entire client and sample information to the new chain of custody record and sign and date relinquishing it and the samples.

6.7.1.2 A photocopy of the original chain of custody record may be used when the number of samples or analyses is large or the chain of custody record is complicated and it would take a lot of time to rewrite the client and sample information on a new chain of custody record. On the chain-of-custody-record-photocopy, the sample custodian preferably using blue ink must:

- Indicate which samples have been sent by crossing out the samples retained;
- Correct the number of sample containers actually being transferred by crossing out the number and writing the number of bottles sent;
- Indicate which analyses the subcontract network laboratory will be performing by highlighting the analyses to be performed and/or crossing out the analyses not subcontracted;
- Write the service request number of the originating laboratory on both the original chain of custody record and on the chain-of-custody-record-photocopy; and
- Sign the chain-of-custody-record-photocopy relinquishing it and the samples.

A photocopy of this completed document shall be placed in this laboratory's project file. The receiving network laboratory should treat this photocopied chain of custody record as its official chain of custody record for their project file. This chain-of-custody-record-photocopy must be signed, preferably using blue ink, when the samples are received and logged in at the receiving network laboratory. It will be retained by the receiving network laboratory and a photocopy returned to the originating network laboratory with the final analytical report.

6.7.1.3 Off-Site Extraction Facility Samples are received at the main laboratory and transported to the off-site extraction facility located at 2360 Shasta Way, Unit G, Simi Valley, CA utilizing LIMS for custody relinquishment.

- Samples (PUF, PUF/XAD-2 cartridges and filters) must be transported, wrapped in aluminum foil in tightly sealed glass jars and maintained at <4˚C with blue ice.
- VOA vials must be wrapped in bubble wrap and transported in a cooler with blue ice to adhere to the temperature requirement of 4˚C+/-2˚C.

The technician must use LIMS when receiving the samples to relinquish extracts to the analyst for storage and analysis.

6.7.2 Sample Transfer to an Out of Network Laboratory (Interlaboratory Transfer)

The originating laboratory, when transferring samples to a laboratory outside the network, must initiate a new chain of custody record. This will help to protect the identity of our customer from the outside laboratory and maintain client confidentiality. The sample custodian will indicate that this laboratory is the client on this new chain of custody record and must accurately transfer all the sample and analysis information. Also, the purchase order number is to be included on the new chain of custody record. The new chain of custody record must be signed and dated relinquishing it and the samples.

6.7.2.1 pH Adjustment Certain methods require a pH check and adjustment to be recorded on the Sample Acceptance Check form. After performing pH adjustment place a yellow tape with the words “pH Check” and “date and time” of adjustment across the top of the bottle. Measure pH after 16 hours; adjust pH if necessary, and repeat the process until proper pH is obtained. The analyst will perform the pH check at the time of analysis.

If received within two weeks of collection, acid preserve upon receipt in the laboratory to lower pH to <2. Following acidification, the sample should be mixed, held for 16 hours, and then verified to be pH <2 just prior to sending out to sub-contract network or out of network laboratory. If for some reason such as high alkalinity, the sample pH is verified to be >2, more acid must be added, and the sample held for 16 hours until verified to be pH <2.

6.7.3 Splitting Samples Avoid splitting whole volume analysis samples; e.g., BNA, pesticides, PCBs. Make appropriate sample splits by pouring sample into containers with appropriate preservative already added.

6.8 Storage and Documentation Distribution

When all samples have been labeled and verified, they are to be placed in the designated storage areas per the *SOP for Laboratory Storage, Analysis, and Tracking*. Where necessary, there are refrigerators and freezers dedicated for specific storage requirements (e.g., Wet Chem, SVOA, etc.) and specific locations entered in the Sample Location module of LIMS.

All documentation (e.g. COCs, Sample Acceptance Check Form, Sample Login Verification, etc.) are to be placed inside the Job Folder and given to the PM. The PM will then distribute the folder to the appropriate department.

6.8.1 Odorous Sample Storage Odorous samples (ex., Tedlar bags or VOAs for sulfur) are to be placed in the SMO hood for login and labeled with a “HIGH SULFUR

CONCENTRATION" caution sticker. The PM is to be contacted so that the best course of action may be taken to prevent any laboratory contamination. Following login, every possible precaution is to be taken when storing the samples; therefore, wherever they are stored must minimize any cross-contamination between stored samples and into the lab air for possible contamination into laboratory systems. Segregation of samples must be performed as necessary to ensure that no contamination occurs between samples, extracts, and standards. After analysis, the odorous samples are returned to the SMO hood for disposal the next day upon PM approval.

7) Equipment and Supplies

7.1 Documentation and Records

Forms, Checklists and other required documentation to be maintained are listed in Section 6.6.

8) Quality Assurance and Quality Control

8.1 Internal system audits shall be performed by the Quality Assurance Manager to assess adherence to the guidelines described in this SOP.

9) Summary of Changes

Table 9.1

Revision Number	Effective Date	Document Editor	Description of Changes
16.0	04/09/2016	C. Humphrey	Updated approval page
			Section 3.2 – removed reference to NELAC
			Section 3.6 – removed reference to NELAC
			Section 6.3.3.5 - Revised
			Section 10.5 – Updated reference
			Attachment 2 – Updated
			Attachment 3 – Updated
			Attachment 5 – Updated

10) References and Related Documents

10.1 TNI 2009 Standards.

10.2 US EPA Methods Update Rule (MUR), effective 4/11/07.

10.3 *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 5.0, July 2013.

10.4 *General Requirements for the Competence of Testing and Calibration Laboratories*, ISO/IEC 17025, second edition, 2005-05-15.

10.5 AIHA-LAP, LLC Policy Document – Module 2A Revision 14: Effective Date: July 1, 2015.

10.6 Minnesota Administrative Rules, *Department of Health*, Chapter 4740, Laboratories; Accreditation Requirements.

11) Attachments

11.1 Attachments

Attachment 1	Training plan for Sample Receiving
Attachment 2	Sample Acceptance Check Form
Attachment 3	Service Request Form
Attachment 4	Sample Login Verification Form (also included in the *SOP for Project Management*)
Attachment 5	Sample Acknowledgement Form

Note: Forms are examples and may be modified as long as the minimum requirements of this document are met.

Attachment 1

Training Plan for Sample Receiving

Training Plan for Sample Receiving, Acceptance, and Login

Trainee _____ Trainer _____ Date _____

1. Read SOP — Trainer ____ Trainee ____ Date _____
2. Read Holding Time, Matrix Table (Appendix F of QA Manual) — Trainer ____ Trainee ____ Date _____
3. Demonstrated understanding of — Trainer ____ Trainee ____ Date _____

   Sample Acceptance Check Form & Chain of Custody Form
4. Demonstrated familiarity with related SOPs — Trainer ____ Trainee ____ Date _____

   SOP for Making Entries onto Analytical Records
   SOP for Laboratory Sample Storage, Analysis, and Tracking
   SOP for Nonconformance and Corrective Action
   SOP for Media Request Fulfillment
5. Sample Receipt — Trainer ____ Trainee ____ Date _____

   ☐Understands & knows Sample Hold Times for different methods, media and matrices (or where to find info.)
   ☐Understands Sample Receipt Procedures during business hours as well as after hours
   ☐Knows acceptable temperature for cooler/samples received and how to evaluate and document information
   ☐Knows how to check liquid samples for air bubbles and how to document information
   ☐Knows how to check samples for integrity & if they are compromised (& what this means), how to document
   ☐Knows appropriate containers for samples received according to requested analyses
   ☐Knows adequate sample volume for the analyses requested
   ☐Knows the proper preservation of samples received according to the requested analyses
   ☐Knows when & why the project manager needs to be notified
   ☐Knows how to check canister pressures
6. Sample Login — Trainer ____ Trainee ____ Date _____

   ☐Understands procedure of login and can successfully accomplish the task
   ☐Understands every field of the SR form
   ☐Able to generate a completed project/job – SR form
   ☐Understands the Sample Acceptance Check Form and how to utilize it for different media
   ☐Understands the Sample Receipt Acknowledgment Form and how to utilize it
   ☐Understands the SR form "Draft" copy and know when to utilize it
   ☐Understands the notes that are required at the top of the SR form (i.e., pressurize with helium) and why
   ☐Understands the documentation that must accompany canisters to pressurization
   ☐Understands when an NCAR must be generated in SMO
   ☐Able to submit hardcopy project requirements and how to document special requirements (& what is important to include) on SR form
   ☐Knows steps in documenting samples received outside of hold time
   ☐Know how to properly label samples during login
7. Freezer and Refrigerator Temperature Readings — Trainer ____ Trainee ____ Date _____

   ☐Read *SOP for Calibration and Use of Laboratory Support Equipment*
   ☐Logbooks (Calibration logbook & Freezer / Fridge Temperature logbook)
   ☐Knows required temperatures
   ☐Understands what to do if a temperature exceeds the required temperature (i.e., documentation, notification of QA)
   ☐Ability to calibrate thermometers using appropriate NIST traceable thermometer
   ☐Understands how to apply correction factors to applicable laboratory thermometers
   ☐Reset digital thermometers when appropriate

Attachment 2

Sample Acceptance Check Form

ALS Environmental
Sample Acceptance Check Form

Client: _____ Work order: _____
Project: _____
Sample(s) received on: _____ Date opened: _____ by: _____

Note: This form is used for all samples received by ALS. The use of this form for custody seals is strictly meant to indicate presence/absence and not as an indication of compliance or nonconformity. Thermal preservation and pH will only be evaluated either at the request of the client and/or as required by the method/SOP.

		Yes	No	N/A
1	Were **sample containers** properly marked with client sample ID?	☐	☐	☐
2	Did **sample containers** arrive in good condition?	☐	☐	☐
3	Were **chain-of-custody** papers used and filled out?	☐	☐	☐
4	Did **sample container labels** and/or tags agree with custody papers?	☐	☐	☐
5	Was **sample volume** received adequate for analysis?	☐	☐	☐
6	Are samples within specified holding times?	☐	☐	☐
7	Was proper **temperature** (thermal preservation) of cooler at receipt adhered to?	☐	☐	☐
8	Were **custody seals** on outside of cooler/Box/Container?	☐	☐	☐
	Location of seal(s)? _____ Sealing Lid?	☐	☐	☐
	Were signature and date included?	☐	☐	☐
	Were seals intact?	☐	☐	☐
9	Do containers have appropriate **preservation**, according to method/SOP or Client specified information?	☐	☐	☐
	Is there a client indication that the submitted samples are **pH** preserved?	☐	☐	☐
	Were **VOA vials** checked for presence/absence of air bubbles?	☐	☐	☐
	Does the client/method/SOP require that the analyst check the sample pH and if necessary alter it?	☐	☐	☐
10	**Tubes:** Are the tubes capped and intact?	☐	☐	☐
11	**Badges:** Are the badges properly capped and intact?	☐	☐	☐
	Are dual bed badges separated and individually capped and intact?	☐	☐	☐

Lab Sample ID	Container Description	Required pH *	Received pH	Adjusted pH	VOA Headspace (Presence/Absence)	Receipt / Preservation Comments

Explain any discrepancies: (include lab sample ID numbers): _____

RSK - MEEPP, HCL (pH<2); RSK - CO2, (pH 5-8); Sulfur (pH>4)

Attachment 3

Service Request Form

Service Request Summary

Folder #: **P1505661**
Client Name: ALS Environmental - Simi Valley
Project Name: IHPAT
Project Number: ROUND 204
P.O. Number:

Report To: Chaney Humphrey
ALS Environmental - Simi Valley
2655 Park Center Drive, Suite A
Simi Valley, CA 93065
Phone Number: 805-526-7161
Cell Number:
Fax Number:
E-mail: chaney.humphrey@alsglobal.com

Project Chemist: Chaney Humphrey
Logged By: KKELPE
Date Received: 12/31/15
Archive? N
Internal Due Date: 1/26/16
QAP: LAB QAP
Qualifier Set: Lab Standard
Formset: Lab Standard
Merged?: Y
Report to MDL?: N
PC Approved?: Y
State of Sampling Location:
EDD: No EDD Specified

9 - 1 each-Tube Charcoal (50/100)

Location: P-04

Lab Samp No.	Client Samp No.	Matrix	Collected	SVG OSHA 07/ OV C Tube CS2
P1505661-001	CFM-DCE-TCE-204-1	Air	12/22/15 0000	I
P1505661-002	CFM-DCE-TCE-204-2	Air	12/22/15 0000	I
P1505661-003	CFM-DCE-TCE-204-3	Air	12/22/15 0000	I
P1505661-004	CFM-DCE-TCE-204-4	Air	12/22/15 0000	I
P1505661-005	BLANK	Air	12/22/15 0000	I
P1505661-006	BLANK	Air	12/22/15 0000	I
P1505661-007	BLANK	Air	12/22/15 0000	I
P1505661-008	BLANK	Air	12/22/15 0000	I
P1505661-009	BLANK	Air	12/22/15 0000	I

Folder Comments:

Industrial Hygiene PT Samples (PAT Round)

Test Comments:

Group	Test/Method	Samples	Comments
Semivoa GC	OV C Tube CS2/OSHA 07	1-9	Chloroform, 1,2-dichloroethane, Trichloroethylene

Attachment 4

Sample Login Verification Form

Sample Login and Verification Checklist

Service Request Number, Client & Project Name	(place folder label here)	SDG Used	PM

Sample(s) delivered by: (circle) Client / ALS Emp. / DHL / GSO / FedEx / UPS / Other_____

Yes	No	N/A	SMO Verification	
			Project number has been correctly entered.	
			Sample IDs from the COC have been correctly entered.	
			Sample date and time collected for each sample has been entered correctly.	
			Date received is correct.	
			Container tags are reconciled and applied to correct containers.	By:
			Container tags have been verified by a second person.	By:
			The analyst and PM have been alerted of Short HT or Rush samples.	Notified:
			Sample receipt discrepancies have been noted on Sample Acc. Check Form.	
			Login Completed	By: Date:

Yes	No	N/A	Client Services Login Verification	
			Folder due date is correct.	
			Project Number, Dates, Times, and Sample IDs are correct.	
			Pricing and Rush charges are correct.	
			The subcontract containers have been tagged and sub COC has been generated.	Sub Lab:
			Samples requiring an MS/MSD are properly indicated in the folder.	
			All non-analytical tasks (encores, EDDs, etc.) are logged in and priced correctly.	
			Client has been notified regarding holding time exceedences and sample receipt discrepancies. Notified by email □ verbally □ voicemail □	By: Date:
			Login Approved (red button)	By: Date:

Yes	No	N/A	Client Services Folder Approval	
			Pricing is correct and approved. (Prepaid work is properly indicated with check or credit card.)	
			Hazardous waste designation has been set properly for each sample.	
			Report and/or EDD are complete.	
			Folder Release	By: Date:

Comments:

Attachment 5

Sample Acknowledgement Form

ACCREDITED IN ACCORDANCE WITH nelac

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

Confirmation of Sample Receipt

To:	Chaney Humphrey	**From:**	Chaney Humphrey
Email:	chaney.humphrey@alsglobal.com	**Email:**	Chaney.Humphrey@alsglobal.com
Fax:		**Fax:**	805-526-7270
Phone:	805-526-7161	**Phone:**	805-526-7161 x2084

Samples for analysis have been received by ALS Environmental on 12/31/15 and assigned our Service Request number **P1505661**. **Please verify the following information and notify me of any corrections as soon as possible.**

The estimated completion date for this work is: **2/1/16**

Client: ALS Environmental - Simi Valley
Project: IHPAT/ROUND 204

EDD Required: No

Tier: I

Report To: Chaney Humphrey
ALS Environmental - Simi Valley
2655 Park Center Drive, Suite A
Simi Valley, CA 93065

Billing Address: Chaney Humphrey
ALS Environmental - Simi Valley
2655 Park Center Drive, Suite A
Simi Valley, CA 93065

Comments: Industrial Hygiene PT Samples (PAT Round)

Thank you for your business!

A - Test is Authorized H - Test is On Hold HP - Test is On Hold Pending Input P - Test is Authorized for Prep Only C - Test has been Cancelled * - Test has assigned QC

				OSHA 07 OV C Tube CS2
P1505661-001	CFM-DCE-TCE-204-1	Air	12/22/15 0000	A
P1505661-002	CFM-DCE-TCE-204-2	Air	12/22/15 0000	A
P1505661-003	CFM-DCE-TCE-204-3	Air	12/22/15 0000	A
P1505661-004	CFM-DCE-TCE-204-4	Air	12/22/15 0000	A
P1505661-005	BLANK	Air	12/22/15 0000	A
P1505661-006	BLANK	Air	12/22/15 0000	A
P1505661-007	BLANK	Air	12/22/15 0000	A
P1505661-008	BLANK	Air	12/22/15 0000	A
P1505661-009	BLANK	Air	12/22/15 0000	A

Test Comments:

Group	Test/Method	Samples	Comments
Semivoa GC	OSHA 07/OV C Tube CS2	1-9	Chloroform, 1,2-dichloroethane, Trichloroethylene

Appendix C
Responses to Regulatory Agency Comments

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 4
ATLANTA FEDERAL CENTER
61 FORSYTH STREET
ATLANTA, GEORGIA 30303-8960

March 6, 2018

UPS NEXT DAY AIR
RETURN RECEIPT REQUESTED

Mr. James C. Foster
Assistant Chief of Staff for Installation Management
Base Realignment and Closure Division (ACSIM-ODB)
2530 Crystal Drive (Taylor Building), Room 5000
Arlington, VA 22202-3940

Dear Mr. Foster:

The U.S. Environmental Protection Agency (EPA) has reviewed the Department of Army Responses to EPA Comments on the Defense Depot of Memphis, Tennessee, Uniform Federal Policy-Quality Assurance Project Plan, Revision 0, dated May 12, 2017.

EPA approves the above mentioned report. Should you have any questions or concerns, please feel free to call me on my cell number 404-229-9500.

Sincerely,

Diedre Lloyd
Remedial Project Manager
Restoration & Sustainability Branch
Superfund Division

cc: Mr. Jamie A. Woods, PG, Tennessee, Department of Environment and Conservation, Memphis Environmental Field Office, 8383 Wolf Lake Drive, Bartlett, TN 38133-4119
Ms. Joan Hutton, CALIBRE, 3898 Mountain View Road, Kennesaw, GA 30152
Mr. Thomas Holmes, HDR Environmental, P.O. Box 728, Highlands, NC 28741

Responses to Comments from
U.S. Environmental Protection Agency (EPA) Region 4 on:
Uniform Federal Policy-Quality Assurance Project Plan
Defense Depot Memphis, Tennessee
May 2017
Comments Received: 22 September 2017

EPA General Comments

1. The 2017 Uniform Federal Policy-Quality Assurance Project Plan, Revision 0, dated May 12, 2017 (UFP-QAPP) does not identify quality assurance (QA) personnel or show the independence of the QA function on this project. Please revise the QAPP to include this information and please ensure the QAPP includes all personnel involved in this investigation.

Response: Lynn Lutz will be designated the QA officer as well as the project chemist on WS #1&2; this information will also be added to the organizational chart in WS #3&5, and the table in WS #4,7&8.

2. QAPP Worksheet #12 references Version 5.0 of the Department of Defense Quality Systems Manual (DoD QSM), and Worksheet #28 references DoD QSM Version 4.2. However, a newer version is available (Version 5.1, dated January 2017). Please revise the UFP-QAPP reference to use the newer version.

Response: All references to the DoD QSM throughout the document will be updated to version 5.1.

3. QAPP Worksheets #19, #30 and #20 list the collection of investigation derived waste (IDW) water samples for volatile organic compound (VOC) analysis; however, no additional information about these samples is provided (e.g., quality control [QC] acceptance criteria). Please revise the UFP-QAPP to include all required information for the collection and analysis of IDW water for VOCs.

Response: WS# 20 Note 3 states “IDW water samples are collected as necessary, usually annually; no field quality control (QC) samples are collected.” A summary of the IDW water waste stream, sampling procedures and reporting will be added to WS #17.

4. QAPP Worksheets #12 and #28 list the measurement performance criteria (MPC) for groundwater VOC matrix spike/matrix spike duplicate (MS/MSD) samples as ≤ 30% relative percent difference (RPD). However, Table 4 in Appendix B of the DoD QSM lists the acceptance criteria as ≤ 20% RPD. Table 2 in Microbac standard operating procedure (SOP) #MSV01also lists the RPD as 20%. Please revise the UFP-QAPP to provide the correct MPC.

Response: The correct MPC (20%) will be entered into WS #12 and #28.

5. The data management, reduction and reporting discussion is insufficiently detailed. For example, it is unclear where hardcopy project documents will be stored and how long these documents and the database will be stored before archival/disposal. It is also unclear how analytical data will be entered into the database, if the entry will be reviewed, and how data qualifiers will be added to the final reports. Please revise the UFP-QAPP to provide greater detail regarding the data management, reduction and reporting tasks as per Section 3.5, Data Management Tasks, of the *Uniform Federal Policy Quality Assurance Project Plan Manual*, dated March 2005 (UFP QAPP Manual).

Response: The data management, reduction and reporting discussion in Worksheets #35 and #36 and SOP-10 will be revised to add greater detail per the comment.

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EPA SPECIFIC COMMENTS

1. **Worksheet #6, Communication Pathways, Page 7:** This worksheet does not include EPA in any communication pathways. In addition, the communication procedures do not always specify the form of communication or timeframe for the notifications. Please revise the worksheet to specify that the EPA will be notified when significant corrective actions or changes occur and also revise the worksheet to include the form of communication and timeframe for notification for all communication drivers.

Response: All communication with EPA is through Army, usually the BEC, as noted for 'Regulatory Interface'.
Will add 'EPA Oversight' and 'TDEC Oversight' as separate drivers with RPMs the 'Responsible Entities' and the 'Procedure' as "Review and comment on project documents, participation in monthly Site Management Team calls to discuss recent project activities, document review and project schedule upcoming documents and issue resolution".
Will add "monitoring compliance with the FFA" to the 'Procedure' for the Regulatory Interface driver.
Will add "Field Decisions" as a 'Driver', with HDR PM as 'Responsible Entity'; 'Procedure' Notify BEC of planned changes to field activities due to site conditions or other factors for discussion with USEPA and TDEC RPMs and regulatory buy-in".

2. **Worksheet #17, Sampling Design and Rationale, Pages 30-31:** Worksheet #17 states, **"**Sample frequencies in 2017 for wells by aquifer are:
   Main Installation (MI)
   - 105 Fluvial Aquifer - semiannual (73), annual (20) and biennial (12);
   - 30 IAQ/UC - semiannual (18), annual (8) and biennial (4); and
   - 3 MAQ - annual (3).

   Dunn Field
   - 80 Fluvial Aquifer - semiannual (37), annual (29) and biennial (14);
   - 4 IAQ/UC - annual (3) and biennial (1); and
   - 1 MAQ – biennial"

   However, it is unclear which wells are specifically scheduled for semiannual, annual, and biennial sampling at both the Main Installation (MI) and Dunn Field. Please revise the UFP-QAPP to include a list of the long term monitoring (LTM) wells to be sampled along with the sampling frequency on which they will be sampled.

Response: The LTM wells are listed on Tables 1 and 2, which are referenced in Worksheet #17. Tables 1 and 2 will be revised to include wells installed in 2016 and 2017 and new Tables 3 and 4 listing current sample frequency will be added. The tables will be referenced in Worksheets #11 and #17. Text will be added to Worksheet #17 stating that sampling frequency for each well is reviewed and revised as necessary per established criteria in the annual LTM report; the criteria will also be provided.

3. **Worksheet #21, Field SOPs, Page 36:** This worksheet notes that several standard operating procedures (SOPs) will be modified for project work, but it is unclear how these SOPs will be altered. Please revise this worksheet to identify how the SOPs will be altered for the current investigation.

Response: The QAPP was developed as a site-wide, generic document for general description of the site conditions and activities; separate project-specific QAPPs are prepared as needed. The entries on Worksheet #21 under "Modified for Project Work?" were meant to indicate that the SOPs may be modified

for project-specific QAPPs. The SOPs were not modified for the activities in the 2017 UFP-QAPP; the "Y" entries will be changed to "N".

4. **Worksheet #21, Field SOPs, Page 36:** Field SOP Technical Bulleting for the Trimble GEO-XH is not included in UFP-QAPP Appendix A. Additionally, SOP 11, Field Sampling Technical Systems Audit, is included in UFP-QAPP Appendix A but is not referenced in any of the UFP-QAPP worksheets. Please revise the UFP-QAPP to reference all SOPs required for this investigation, and ensure that all SOPs are provided in the UFP-QAPP appendices.

Response: The QAPP will be revised to reference all required SOPs and to include all referenced SOPs in the appendices.

5. **Worksheet #36, Data Validation Procedures, Pages 66 to 67:** The UFP-QAPP does not indicate what will be included in the data verification/validation reports. Revise the UFP-QAPP to ensure that data validation and verification reports will present a discussion of the following:
   - all QC parameters evaluated,
   - the acceptance criteria used to evaluate each QC parameter,
   - a list of all QC exceedances as well as the extent of the exceedance,
   - the samples associated with each exceedance, and
   - what qualifiers are applied.

Response: WS #36 will be revised to include the requested information.

6. **Appendix A, SOP-4, Groundwater Sample Collection, Section 5.2.1, Water Level Sweep, Page 4-3**: The procedures listed in Section 5.2.1 do not include opening the well and allowing for aquifer equilibration prior to collecting a depth-to water measurement. Please revise Section 5.2.1 of SOP-4 to include allowing the aquifer to equilibrate prior to collecting depth-to-water measurements or provide a rationale as to why this is not necessary.

Response: SOP-4, Section 5.2.1 will be revised to state water levels will be allowed to equilibrate prior to measurement after removing sealing caps. There are no set guidelines for equilibration times. Since the LTM wells at DDMT are installed in sand or sand/gravel layers, they should equilibrate quickly. The measurement will be collected approximately 3 minutes after the well cap is removed. If positive or negative pressure is noted when the well cap is removed, at least two measurements will be made.

7. **Appendix A, SOP-4, Groundwater Sample Collection, Section 5.2.1, Water Level Sweep, Page 4-3:** The procedures listed in Section 5.2.1 do not state the accuracy at which depth-to-water measurements will be collected (i.e. to the nearest 0.01ft). Please revise Section 5.2.1 to include the accuracy at which depth-to-water measurements will be collected during the water level sweep.

Response: The SOP will be revised to state measurements will be recorded to the nearest 0.01 feet.

8. **Appendix A, SOP-4, Groundwater Sample Collection, Section 5.2.2, Water Quality Measurements, Page 4-4:** Section 5.2.2 states, "Groundwater samples will be collected when water quality indicators of dissolved oxygen (DO), redox potential (ORP), pH, specific conductivity, and turbidity stabilize;" however, stabilization of the water column is not included as a parameter that needs to be met prior to collecting groundwater samples. Please revise Section 5.2.2 to include the criteria by which the water column is considered stable prior to groundwater sample collection.

Response**:** The SOP will be revised to state water levels will be used to confirm stabilization when samples are collected using the low-flow sampling procedure. There should be only a slight and stable

drawdown of the water column after pumping begins. If drawdown does not stabilize, the sample will be collected after at least three well casing volumes are removed.

9. **Appendix A, SOP-4, Groundwater Sample Collection, Section 5.2.3, Page 4-4:** SOP-4 in Appendix A states, "Water levels will generally be measured before and during sampling. For wells with dedicated pumps, water levels will be measured only if the water is above the top of the pump. The pump will not be removed in order to obtain a water level;" however, the UFP-QAPP does not designate if wells at either the MI or at Dunn Field have dedicated pumps. Please revise the UFP-QAPP to note the sampling method (i.e. dedicated pumps, passive diffusion bags, bailers, etc.) is planned to be used at each well location.

Response: SOP 4 will be updated to describe current sampling methods. Samples at most LTM wells are collected using PDBs, and no wells currently have dedicated pumps. Tables 3 and 4 which are to be added per the response to Comment #2 will indicate the usual sample method at each well.

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August 9, 2017

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Subject: **2017 Uniform Federal Policy-Quality Assurance Project Plan, Rev. 0**
Defense Depot Memphis, Tennessee
TDoR ID # 79-736

Mr. Foster,

TDEC-DoR has reviewed the contents of the **2017 Uniform Federal Policy-Quality Assurance Project Plan (Rev. 0)** as submitted by T. Holmes (HDRInc), and approves of the document's contents. If there are questions or concerns, please contact me at (901) 371-3041 or at jamie.woods@tn.gov .

Regards,

Jamie A. Woods, P.G.
Project Manager
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cc: Thomas C. Holmes (HDRINC)
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