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Supplemental Remedial Investigation Phase 3 QAPP

Environmental Restoration Support at Former Defense Depot Memphis, Tennessee

Contract W91278-16-D-0061, Task Order 0002

Revision 1 April 2018





Table of Contents

Acronyms and Abbreviations	iii
QAPP Worksheet #1 & 2: Title and Approval Page	1
QAPP Worksheet #3 & 5: Project Organization and QAPP Distribution	3
QAPP Worksheet #4, 7 & 8: Personnel Qualifications and Sign-off Sheet	
QAPP Worksheet #6: Communication Pathways	7
QAPP Worksheet #9: Project Planning Session Summary	9
QAPP Worksheet #10: Conceptual Site Model	. 10
QAPP Worksheet #11: Project Data Quality Objectives	. 17
QAPP Worksheet #12: Measurement Performance Criteria	.20
QAPP Worksheet #13: Secondary Data Uses and Limitations	.22
QAPP Worksheet #14/16: Project Tasks & Schedule	.23
QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits	;28
QAPP Worksheet #17: Sampling Design and Rationale	. 30
QAPP Worksheet #18: Sampling Locations and Methods/SOP Requirements	. 32
QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times	. 33
QAPP Worksheet #20: Field QC Summary	.34
QAPP Worksheet #21: Field SOPs	.35
QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection	. 36
QAPP Worksheet #23: Analytical SOPs	.37
QAPP Worksheet #24: Analytical Instrument Calibration	. 38
QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and	
Inspection	
QAPP Worksheet #26 & 27: Sample Handling, Custody, and Disposal	
QAPP Worksheet #28: Analytical Quality Control and Corrective Action	
QAPP Worksheet #29: Project Documents and Records	
QAPP Worksheet #31, 32 & 33: Assessments and Corrective Action	
QAPP Worksheet #34: Data Verification and Validation Inputs	
QAPP Worksheet #35: Data Verification Procedures	
QAPP Worksheet #36: Data Validation Procedures	
QAPP Worksheet #37: Data Usability Assessment	
References	.58

Tables

Table 1.	Soil Samples Exceeding Remediation Goals	12
Table 2.	Phase 3 Well Locations	30

Tables (Attached)

- Table 3. Planned Well Locations and Depth
- Table 4.Existing LTM Wells

Figures

- Figure 1. Main Installation Unit Boundaries
- Figure 2. MIP Survey and Soil Sample Locations
- Figure 3. Main Installation LTM Wells
- Figure 4. Fluvial Aquifer PCE Concentrations
- Figure 5. Fluvial Aquifer TCE Concentrations
- Figure 6. Intermediate Aquifer PCE Concentrations
- Figure 7. Intermediate Aquifer TCE Concentrations
- Figure 8. Fluvial Aquifer Groundwater Elevations
- Figure 9. Intermediate Aquifer Groundwater Elevations
- Figure 10. Recommended Well Locations

Appendices

- Appendix A. Field Standard Operating Procedures
 - SOP 1 General Procedures for Field Personnel
 - SOP 2 Drilling and Soil Sampling
 - SOP 3 Well Installation, Development and Abandonment
 - SOP 4 Groundwater Sample Collection
 - SOP 7 Sample Control and Documentation
 - SOP 8 Sample Packing and Shipping
 - SOP 9 Equipment Decontamination

Appendix B. Analytical Standard Operating Procedures

- L1 Microbac-Analysis of Volatile Organic Analytes by Method 8260
- L2 Microbac-Sample Receiving and Login
- L3 Chemtech-Volatile Organic Compounds by GC/MS SW 846 Method 8260B/C
- L4 Chemtech-Sample Preparation for Toxicity Characteristics Leachate Procedure
- L5 Chemtech-Sample Receipt, Sample Acceptance Policy, Sample Login, Sample Identification, and Sample Storage
- Appendix C. Responses to USEPA Comments, USEPA and TDEC Approval Letters

Acronyms and Abbreviations

	American Casisty for Tasting and Materials
ASTM	American Society for Testing and Materials
BEC	BRAC Environmental Coordinator
BFB	4-bromofluoro-benzene
bgs	below ground surface
BRA	Baseline Risk Assessment
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
Chemtech	Chemtech Consulting Group
CoC	chain-of-custody
COR	Contracting Officer's Representative
СТ	carbon tetrachloride
CVOC	chlorinated volatile organic compound
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
ft	feet/foot
FTL	Field Team Leader
GC/MS	gas chromatography/mass spectrometry
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDR	HDR Environmental, Operations and Construction, Inc.
IAQ	Intermediate Aquifer
ICAL	initial calibration
ICB	initial calibration blank
ICV	initial calibration verification
IS	internal standard
J&E	Johnson and Ettinger
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LIMS	Laboratory Information Management System
LOD	limit of detection
LOQ	limit of quantitation
LTM	long-term monitoring

MAQ	Memphis Aquifer
MCL	maximum contaminant level
MDL	method detection limit
MI	Main Installation
MIP	membrane interface probe
mL	milliliter
MLGW	Memphis Light, Gas & Water
MS	matrix spike
MSD	matrix spike duplicate
NAVD	North American Vertical Datum of 1988
ORP	oxygen reduction potential
OSHA	Occupational Safety and Health Administration
PCB	polychlorinated biphenyl
PCE	tetrachloroethene
PCP	pentachlorophenol
PID	photoionization detector
PM	Project Manager
PQO	Project Quality Objective
PVC	polyvinyl chloride
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
QSM	Quality Systems Manual
RA	remedial action
RAO	remedial action objective
RF	response factor
RG	remediation goal
RL	reporting limit
RPD	relative percent difference
RPM	Remedial Project Manager
RRT	relative retention time
RSD	relative standard deviation
RT	retention time
SCHD	Shelby County Health Department
SMT	Site Management Team
SOP	Standard Operating Procedure
SOW	Statement of Work
SPCC	System Performance Check Compound
SRI	supplemental remedial investigation
SVOC	semi-volatile organic compound
TCE	trichloroethene
TCLP	toxicity characteristic leaching procedure
TDEC	Tennessee Department of Environment & Conservation
ТМ	Technical Manager
TTA	target treatment area
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
VI	vapor intrusion
VISL	Vapor Intrusion Screening Level Calculator
VOC	volatile organic compound
%R	percent recovery
µg/L	micrograms per liter

QAPP Worksheet #1 & 2: Title and Approval Page

Project Name	Environmental Restoration Support, Former Defense Depot Memphis, Tennessee (DDMT)			
Site Location	Memphis, Shelby County, Tennessee			
Contract Number	W91278-16-D-0061			
Task Order	0002			

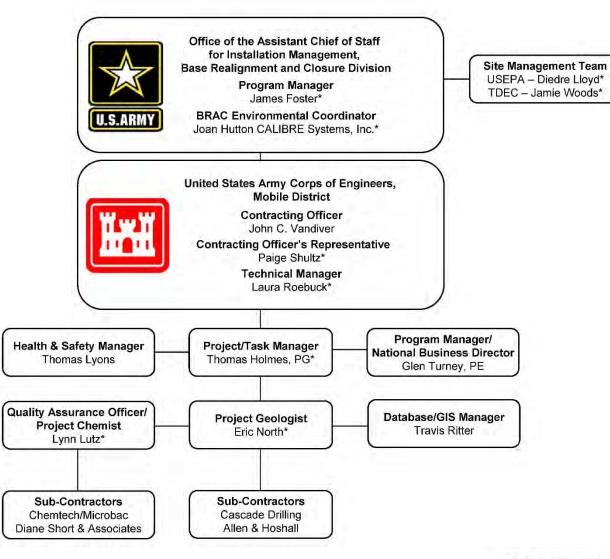
Approvals:

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 Systems, 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 Environmental, Operations and Construction, Inc. (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
Memphis Depot Main Installation Record of Decision Memphis Depot Caretaker, Revision 2.	February 2001	CH2M HILL
Main Installation Source Areas Investigation, Defense Depot Memphis, Tennessee, Defense Logistics Agency, Revision 0.	February 2009	e²M
Main Installation Interim Remedial Action Completion Report, Defense Depot Memphis, Tennessee, Defense Logistics Agency, Revision 1.	February 2010	HDR e ² M
Preliminary Close Out Report, U. S. Defense Depot Memphis Tennessee, TN42100220570.	February 2010	USEPA
Supplemental Remedial Investigation Phase 2 Work Plan, Defense Depot Memphis, Tennessee, Revision 1.	August 2016	HDR
Annual Long-Term Monitoring Report-2016, Defense Depot Memphis, Tennessee, TN42100220570, Revision 0.	April 2017	HDR
Supplemental Remedial Investigation Phases 1 and 2 Report, Defense Depot Memphis, Tennessee, TN4210020570, Revision 0.	October 2017	HDR

QAPP Worksheet #3 & 5: Project Organization and QAPP Distribution



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Jay Caughman	Allen & Hoshall	Surveying PM	jcaughman@allenhoshall.com	901-261-4710	

QAPP Worksheet #4, 7 & 8: Personnel Qualifications and Sign-off Sheet

ORGANIZATION: HDR

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Tom Holmes	PM	MS Geophysics 40 years	Registered Professional Geologist, Georgia	
Lynn Lutz	QA Officer / Project Chemist	BA Chemistry 31 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-hr 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 15 years	OSHA HAZWOPER, 40-hour; OSHA 510 Construction Industry Health and Safety	

ORGANIZATION: Chemtech

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Loreana Davi	Laboratory PM	BA International Business and Management, one year experience		

ORGANIZATION: Microbac

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

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

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. QA supervision for contract activities.
Regulatory Interface	BEC	Joan Hutton	770-317-4323	Communicate with USEPA/TDEC via phone and email as needed, and submit project documents for regulatory review. Monitor compliance with the Federal Facilities Agreement (FFA) (USEPA, 1995) regarding submittals and timing.
USEPA Oversight	USEPA RPM	Diedre Lloyd	404-562-8855	Review and comment on project documents. Participate in monthly Site Management Team calls to discuss recent project activities, document review and project schedule, upcoming documents and issue resolution. Documents for initial review will be submitted electronically by email or on CD via overnight delivery; in either case a paper copy of the text, tables and figures will be submitted via overnight delivery. Final documents will be submitted electronically by email or on CD via overnight delivery; paper copies will not be provided, unless requested. Communications regarding issue resolution will be initiated via phone call or email on the day the potential issue is identified.
TDEC Oversight	TDEC RPM	Jamie Woods	901-371-3041	Review and comment on project documents. Participate in monthly Site Management Team calls to discuss recent project activities, document review and project schedule, upcoming documents and issue resolution. Documents will be submitted electronically by email or on CD via overnight delivery. Communications regarding issue resolution will be initiated via phone call or email on the day the potential issue is identified.
Technical Direction	BEC	Joan Hutton	770-317-4323	Review project documents and represents the BRAC PM.
Manage Task Order activities	HDR PM	Tom Holmes	404-295-3279	Submit task order deliverables; 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 significant changes to field activities due to site conditions or other factors for discussion with USEPA and TDEC RPMs and regulatory buy-in. Communicate by phone or email within at least 4 hours of identifying the required change.
Manage Field Tasks	HDR Project Geologist	Eric North	512-775-7848	Supervise HDR field activities. Communicate with HDR PM and Project Chemist. Provide daily quality control reports (DQCRs) and notification of any work problems.

Communication Driver	Responsible Entity	Name	Contact Information	Procedure (timing, pathway, etc.)
QAPP changes in the field	HDR QA Officer	Lynn Lutz	303-754-4266	Manage and implement in-field QAPP changes. Notify HDR PM of QAPP changes.
Reporting Lab Data Quality Issues	Laboratory PM	Kurt Hummler	908-789-8900 * 3143	Notify the HDR Project Chemist regarding laboratory data quality issues including corrective actions (CAs) and data usability.
Field CAs	HDR Project Geologist/FTL	Eric North	512-775-7848	Issue CAs in writing to the HDR PM for review and submittal to USACE TM and BEC.
Analytical CAs	HDR Project Chemist	Lynn Lutz	303-754-4266	Coordinate laboratory analyses, review deliverables, determine the need for CA on analytical issues and notify the HDR PM. Provide 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 or hard copy, as applicable.

QAPP Worksheet #9: Project Planning Session Summary

No planning sessions were held, but the supplemental remedial investigation (SRI) has been discussed on monthly Site Management Team (SMT) calls and during the August 29, 2017 SMT project review meeting in Memphis.

The Main Installation (MI) SRI was initiated in 2015 and included review of the basis for the selected remedy and field investigation to improve understanding of site hydrogeology, on-site extent of groundwater contamination and potential off-site sources of contamination. The SRI Phase 2 Work Plan (HDR, 2016), which was approved by TDEC in June 2016 and by EPA in August 2016, identified data gaps to be addressed based on review of the Phase 1 findings and the 2015 LTM results. An additional 29 well locations were recommended with 9 wells to be installed during Phase 2 and the remaining 20 wells during Phase 3.

The requirements of SRI Phase 3 were established in the Statement of Work (SOW) for Contract No. W91278-16-D-0061 Task Order Number 0002 (USACE, 2016). The SOW includes installation of 20 monitoring wells on the Main Installation (MI and off-site, groundwater sampling and analysis for volatile organic compounds (VOCs). Following installation and initial sampling, the Phase 3 wells will be incorporated into long-term monitoring (LTM); the wells will be sampled semiannually in LTM events until at least four samples have been collected and a sample frequency can be determined in accordance with the current criteria as described in the 2016 LTM report (HDR, 2017a). An access agreement for one off-site, Phase 2 Intermediate Aquifer (IAQ) well could not be completed; two onsite Fluvial Aquifer wells were installed instead, leaving 19 wells to be installed in Phase 3.

The SRI Phases 1 and 2 Report (HDR, 2017d) is in preparation for submittal to EPA and TDEC in October 2017. The report presents findings from SRI wells and LTM results through April 2017, and includes review of the initial Phase 3 locations.

Preliminary Phase 3 well locations were discussed during the August 2017 meeting. Mr. Foster, Army BRAC Program Manager, suggested the number of Phase 3 wells be based on project requirements rather than a pre-set number, as it would be more beneficial in the long run to provide a comprehensive site analysis as quickly as possible. Ms. Lloyd, EPA RPM, suggested SRI wells be placed where needed for design of remedial activities. This guidance was followed in preparing the Project Data Quality Objectives (DQOs) in Worksheet #11 and Sampling Design and Rationale in Worksheet #17.

QAPP Worksheet #10: Conceptual Site Model

Background Information

Site History

DDMT is located in southeastern Memphis, Tennessee and consists of approximately 632 acres at the MI and Dunn Field. The MI contains approximately 567 acres with open storage areas, warehouses, former military family housing, and outdoor recreational areas. Dunn Field, which is located across Dunn Avenue from the northwest section of the MI, contains approximately 65 acres with former mineral storage and waste disposal areas.

DDMT originated as a military facility in the early 1940s to provide stock control, material storage, and maintenance services for the U.S. Army. DDMT was selected for closure under BRAC in 1995, and storage and distribution activities ceased in September 1997.

During operations from 1942 to 1997, DDMT received, warehoused, and distributed supplies to U.S. military services and civilian agencies. The supplies included hazardous substances; textile products; food products; electronic equipment; construction materials; and industrial, medical, and general supplies.

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, based on the North American Vertical Datum of 1988 (ft NAVD). The highest point is at 312 ft NAVD along Dunn Avenue near the northwest MI and the lowest point is at 267 ft NAVD 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. 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 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 and is located approximately 1 mile west of DDMT.

Downward leakage from the Fluvial Aquifer is widespread in the Memphis area, particularly where the confining unit is thin or absent and in the vicinity of MLGW well fields. An hydraulic connection or 'window' has been identified in the northwestern MI. Sands in the fluvial deposits and in the Upper Claiborne, observed to be in contact at several well borings in the central MI, 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.

Within the northwestern MI, a connection or window between the Fluvial Aquifer and IAQ has been identified through numerous soil borings and water level measurements, in addition to elevated chlorinated volatile organic compounds (CVOCs) in the IAQ monitoring wells within the area. The extent of the "window" indicates that there is also a connection to the underlying MAQ. In addition, Fluvial Aquifer groundwater elevations indicate a sink, due to leakage into the IAQ, is present in the south-central MI near MW-259.

Groundwater flow direction in the Fluvial Aquifer 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 sink in the south-central MI. Groundwater flow in the IAQ on the MI is into and through the window in the northwest MI (HDR, 2017c).

Land Use Considerations

All 567 acres on the MI have been transferred through public benefit and economic development conveyances. Land use controls implemented for the MI prevent residential use, drilling of groundwater wells and production or consumptive use of groundwater except in the eastern section of Functional Unit 6, Administrative and Residential Areas (Figure 1) which is available for

unrestricted use (CH2M HILL, 2004). The MI is primarily used for warehousing in the Memphis Depot Industrial Park and for general operations at Barnhart Crane and Rigging.

Site Contaminants

Types of past activities that could have resulted in the presence of hazardous materials in environmental media at the MI include repackaging hazardous substances 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 CVOCs in groundwater at the MI.

Known or Suspected Contaminants or Classes of Contaminants

The MI remedial investigation (RI) (CH2M HILL, 2000) only found soil contamination in near-surface soils; contaminants consisted primarily of metals, PCBs, semi-volatile organic compounds (SVOCs), and a pesticide, dieldrin. Groundwater contaminants were limited to CVOCs primarily tetrachloroethene (PCE) and trichloroethene (TCE); carbon tetrachloride (CT) and chloroform (CF) were detected in the southeast MI, in addition to PCE and TCE. CVOCs were not detected at high concentrations in soil samples on the MI.

Additional investigation was performed in 2008 to identify potential source areas for the observed groundwater plumes on the MI (e2M, 2009). The field investigation consisted of a membrane interface probe (MIP) survey and confirmation soil samples on grids overlaying the upgradient areas of the plumes. The MIP survey and soil sample locations are shown on Figure 2. Seventy soil samples were collected and the results were compared to the soil remediation goals (RGs) established for Dunn Field, which has similar proposed land use and potential for impacts to groundwater. Only five soil samples contained CVOCs at concentrations above an RG, and three were only slightly above the RGs; the five samples are listed on Table 1 with the CVOC concentrations and the respective RGs. The generally low CVOC concentrations were not considered to warrant remedial action (RA).

-	-		
Sample	счос	RG (mg/kg)	Concentration (mg/kg)
	carbon tetrachloride	0.2150	22.9
TTA2-W-B1-SB-27	chloroform	0.9170	1.07
TTA2-W-B2-SB-17	methylene chloride	0.0305	0.508
TTA2-W-A2 26-27	methylene chloride	0.0305	0.0389
WCP-B3-SB-8	tetrachloroethene	0.1806	0.199
WCP-A4-7.5-9.5	tetrachloroethene	0.1806	0.181

Table 1. Soil Samples Exceeding Remediation Goals

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.

Secondary Contaminant Migration

Secondary contaminant migration was limited to CVOCs on the MI. Following release through spills, contaminants are held in the loess because of limited permeability and adsorption to clay and organic matter in the fine-grained soils. CVOCs migrate into the fluvial soils beneath the loess with infiltration of rainwater and then to groundwater.

Fate and Transport

The contaminants of concern are CVOCs in groundwater, which migrate with groundwater. Groundwater in the Fluvial Aquifer flows onto the MI from all sides with flow off the MI through vertical leakage at the 'window' in the northwest MI and the sink in the south-central MI.

Naturally occurring biodegradation of CVOCs does not appear to be a significant contributor to natural attenuation in the Fluvial Aquifer at the MI based on the aquifer's low natural carbon content and high dissolved oxygen (DO), and the absence of reductive dechlorination products outside of the EBT areas. However, first order decay rates calculated for the 2009 groundwater model had good agreement with PCE and TCE concentrations at wells along the flow paths, which indicate attenuation is occurring through physical processes of dispersion, dilution, sorption and volatilization (HDR, 2017d).

Groundwater contamination in the IAQ and MAQ in the Memphis area is the result of vertical migration of contaminants from the surficial aquifers (alluvial and fluvial); the alluvial aquifer is not present at DDMT. Contaminant concentrations in the MAQ wells at DDMT are below maximum contaminant levels (MCLs); groundwater remediation in the Fluvial Aquifer is considered the most effective approach to protecting water quality in the deeper aquifers.

Potential Receptors and Exposure Pathways

The Baseline Risk Assessment (BRA) conducted during the MI RI (CH2M HILL, 2000) included a human health risk assessment. Health risks to industrial workers were within acceptable levels for future industrial use of the property, except for lead in a limited surface soil area. However, it was concluded that soil contaminants of concern were present at levels that do not allow for unrestricted use and unlimited exposure. The BRA also concluded that contaminated groundwater in the Fluvial Aquifer poses an unacceptable risk through the ingestion pathway; however, land use controls prevent drilling of groundwater wells and production or consumptive use of groundwater on the MI, except in Functional Unit 6 as noted previously.

In July 2017, HDR used the October 2016 groundwater analytical results and the USEPA Vapor Intrusion Screening Level Calculator (VISL) and the Johnson and Ettinger (J&E) vapor intrusion (VI) model to evaluate the VI human health risk from VOCs in groundwater at the MI under commercial exposure scenarios. The cancer and non-cancer risks from VISL were five to ten times greater than the risks calculated with the J&E model. However, both methods identified risks greater than the target 1x10⁻⁶ risk for cancer. Therefore, VOC concentrations in groundwater at the MI may present a human health risk via VI (HDR, 2017e). A separate work plan has been prepared for soil gas sampling on the MI to further evaluate the VI human health risk on the MI (HDR, 2017f).

Nature and Extent of Contamination

Remedial action objectives (RAOs) and the selected remedy for the MI were established in *the Main Installation Record of Decision, Revision 2* (MI ROD) (CH2M HILL 2001). The MI groundwater RAOs include restoration of groundwater quality to levels at or less than MCLs and prevention of off-site migration (horizontally or vertically) of groundwater contaminants in excess of MCLs. The selected groundwater remedy for the MI was enhanced bioremediation treatment (EBT), land use controls and LTM. The remedy required EBT to be implemented in the most contaminated parts of the plume, while untreated parts of the plume were allowed to degrade under natural attenuation processes.

EBT was conducted from September 2006 to March 2009 (EBT-1) and November 2012 to November 2014 (EBT-2). While CVOC concentrations were reduced, the RA was not sufficient to meet the RAOs for the MI.

LTM has been performed at the MI since 2004. MI LTM wells are shown on Figure 3. As noted in the figure legend, the well symbols indicate the aquifer (Fluvial, Intermediate or Memphis) and are color coded by area (Background, B-835, North-Central, South-Central, West-Central, Southeast, target treatment area (TTA)-1 North, TTA-1 South, TTA-2 and Window).

Groundwater contamination above MCLs is documented through semiannual LTM sampling. Although several CVOCs have been detected above MCLs, the CVOCs detected most frequently above MCLs are PCE and TCE. The April 2017 LTM results (HDR, 2017c) showing concentrations at individual wells, concentration isopleths and groundwater elevation contours are presented on Figures 4 to 7.

Recent Findings and Data Gaps

Findings from the 2016 and April 2017 LTM reports (HDR, 2017a; HDR, 2017c) and the SRI Phases 1 and 2 report (HDR, 2017d), and the remaining data gaps are discussed below.

Groundwater Flow

Groundwater flow in the Fluvial Aquifer, which is controlled by the surface of the uppermost clay in the Upper Claiborne, is relatively well defined. Groundwater flow is onto the MI from all sides with flow off the MI through vertical leakage at the window in the northwest MI and the sink in the south-central MI.

Groundwater in the IAQ flows into and through the window in the northwest MI but the hydraulic gradient from the northwest MI is not clearly defined by the existing wells. The gradient in the IAQ may be variable due to the individual sand and clay beds having limited extent. The groundwater flow direction and extent of CVOCs in the IAQ to the north of MW-256 need to be determined.

Groundwater flow direction in the MAQ is also not well-defined; only two wells on the MI and one well on Dunn Field are screened in the Memphis Sand. However, further investigation of the MAQ is not considered necessary for re-evaluation of the selected remedy in the MI ROD.

The hydraulic gradient in the Fluvial Aquifer and the IAQ at the southeast area of the window (area bounded by MW-282, MW-63A/B, MW-207A/B, MW-211 and MW-107) is not well defined. The groundwater elevation contours in the Fluvial Aquifer (Figure 8) indicate flow is toward the area of

MW-39 and MW-208B or toward the sink at MW-259; the only area with Fluvial Aquifer flow clearly into the window is near MW-63A/B. The outlines of the PCE and TCE plumes in the Fluvial Aquifer are generally consistent with these flow paths.

The groundwater elevation contours in the IAQ (Figure 9) indicate flow in the northwest quadrant of the MI is to the northwest; limited IAQ groundwater elevations in the north-central MI indicate flow is to the southeast. The shape of the PCE plume in the IAQ (Figure 6) indicates contaminant migration to the northeast (MW-197A to MW-207A) before the plume alignment turns to the northwest. Since contaminant migration from the Fluvial Aquifer into the IAQ is considered the route for potential impacts to the MAQ, additional wells for groundwater elevations and CVOC concentrations in this area are needed.

CVOC Concentration Trends

CVOC concentrations have been generally stable as shown on trend plots in the annual LTM reports (HDR, 2017a). CVOC concentrations exceeded the MCL in 86 of 146 MI LTM wells in April 2017, mainly for PCE and TCE; the number of wells was the same as the previous event in October 2016 (HDR, 2017c). Of the 86 MI LTM wells with concentrations above the MCL, 20 Fluvial Aquifer wells and 1 IAQ well (MW-107B) exceeded an MCL by a factor of 10 (>50 micrograms per liter [μ g/L] for PCE, TCE and CT and >20 μ g/L for vinyl chloride [VC]).

Decreasing or increasing CVOC concentrations are observed in a number of wells in former EBT areas. Continued reductive dechlorination is indicated in wells with decreasing PCE or TCE and increasing cis-1,2-Dichloroethene (cDCE) or VC (e.g., MW-100B, PMW21-04 and PMW85-05). Lower reductive dechlorination is indicated in wells with the opposite trends, increasing PCE or TCE and decreasing cDCE or VC (e.g., MW-85). Areas near wells with relatively quick rebound of CVOC concentrations following EBT or consistently high concentrations of parent compounds (PCE, TCE and CT) may be near past releases and should be considered for additional investigation prior to implementation of additional RA. These wells include: PMW21-02, PMW21-03, and PMW21-04 in TTA-1 North; MW-101, PMW101-03B and PMW101-04B in TTA-1 South; and DR2-1, MW-92 and PMW85-05 in TTA-2. Additional investigation could include further MIP surveys, soil gas sampling in loess or fluvial deposits and/or additional soil sampling.

Plume Delineation

CVOC concentrations in a number of LTM wells are consistently above MCLs and showed no impact from previous EBT indicating the areal extent of RA will need to be expanded to meet the RAO for concentrations below the MCLs throughout the MI. Delineation of identified plumes will need to be completed during the SRI to effectively determine the extent of remedial activities.

CVOC concentrations at MW-266 indicate there is not an upgradient, off-site source at the eastern end of the TTA-2 plume, but CVOC concentrations at MW-267 indicate potential for an on-site source further north. The PCE concentration of 5.87 μ g/L at MW-280 may represent the upgradient (northeast) extent of the TTA-2 plume.

Upgradient impacts were identified at the North-Central, TTA-1N and TTA-1S plumes (Figures 4 and 5). The upgradient impacts are off-site for the TTA-1N plume and potentially off-site for the North-Central and TTA-1S plumes (HDR, 2017d). Off-site well MW-269, which is upgradient of TTA-1N,

had its highest PCE concentration (64.9 μ g/L) in the April 2017 sample. Migration of CVOCs onto the MI will need to be addressed in planning additional RA and additional upgradient delineation should be evaluated at these areas.

Relatively high concentrations of TCE and cDCE above the MCLs were initially reported at MW-270 on the southern MI boundary, but decreased over time. Phase 2 wells, MW-275 and MW-276, show the extent of CVOCs observed at MW-270 is limited. MW-270 is not downgradient of an identified plume and a discrete, upgradient source is suspected and further investigation is needed.

Groundwater Impacts in the IAQ and MAQ

IAQ well MW-107, which is located in the upgradient section of the 'window', has had variable PCE concentrations, which have ranged from 20 to 80 µg/L since 2011. MW-107 was installed in 2001 to evaluate potential contamination from Building 720, a Former Fuel and Cleaners Dispensing location. PCE is the primary contaminant in the window area of MI IAQ wells. The nearest well with a similar concentration is MW-207B, a fluvial West-Central well with PCE concentrations of 12 to 124 µg/L since 2011. As noted above, additional wells are needed to determine the hydraulic gradient near the southeast area of the window, near MW-107. CVOC concentrations in these wells will also indicate the potential for groundwater contamination at MW-107 to impact the IAQ and MAQ.

CVOCs in the Fluvial Aquifer have migrated vertically into the IAQ through the window in the northwestern area of the MI. Increasing CVOC concentrations have been observed in two deeper IAQ wells (MW-140 and MW-202A), but the concentrations are relatively low (10.9 and 5.2 µg/L). CVOC concentrations remain below MCLs in downgradient MAQ wells MW-254 and MW-255. The CVOCs in the deeper IAQ wells are considered to be due to contaminant migration from the Fluvial Aquifer; additional IAQ wells in the window are not considered necessary at present. However, the extent of CVOCs in the IAQ downgradient of the window and north of MW-256 should be investigated.

Leakage from the Fluvial Aquifer to the IAQ at the sink in the south-central MI creates a potential for contaminant migration in to the IAQ. The presence of contamination in the IAQ near the center of the sink should be investigated. An IAQ well in this area will also aid determination of the groundwater flow direction in the IAQ south of the window.

QAPP Worksheet #11: Project Data Quality Objectives

DQOs for Phase 3 well installation and sampling were established using the seven step process developed by the EPA in the *Guidance on Systematic Planning Using the Data Quality Objective Process* (USEPA, 2006). The purpose of these DQOs are to identify the project objective and establish performance and acceptance criteria, which serves as the basis for designing a plan for collecting data of sufficient quality and quantity to support the goals of the study (USEPA, 2006).

Step 1: State the Problem

Due to the number of LTM wells exceeding MCLs and the CVOC concentrations at wells located outside areas impacted by previous EBT, the Army is re-evaluating the selected remedy for the MI. The following data gaps need to be addressed in order to provide a solid basis for the re-evaluation.

- 1. Determine the hydraulic gradient and off-site extent of CVOCs in the IAQ to the north of MW-256.
- 2. Investigate potential contaminant migration into the IAQ at the Fluvial Aquifer sink and determine the IAQ hydraulic gradient in the south-central MI.
- Determine the hydraulic gradient and extent of CVOCs in the Fluvial Aquifer and the IAQ at the southeast area of the window (area bounded by MW-282, MW-63A/B, MW-207A/B, MW-211 and MW-107).
- 4. Assess a potential contaminant source near MW-107 that could impact the IAQ and MAQ due to its location within the upgradient area or the window.
- 5. Complete delineation of identified plumes, including potential upgradient impacts on-site and off-site, where needed to effectively plan remedial activities.
- 6. Investigate upgradient extent of CVOCs at MW-270 on the southern MI boundary.
- 7. Complete well installation in areas without previous investigation as identified in the SRI Phase 2 work plan.
- 8. Investigate vertical heterogeneity in hydraulic parameters in the Fluvial Aquifer and IAQ in order to effectively position well screens for vertical delineation in Phase 4.
- Investigate potential source areas near wells with relatively quick rebound of CVOC concentrations following EBT or consistently high concentrations of parent compounds (PCE, TCE and CT). (PMW21-02, PMW21-03, PMW21-04 in TTA-1 North; MW-101, PMW101-03B and PMW101-04B in TTA-1 South; and DR2-1, MW-92 and PMW85-05 in TTA-2).

Step 2: Identify the Goal of the Study

The goal of this investigation is to collect hydrogeological and analytical data from new monitoring wells in the Fluvial Aquifer and the IAQ to address identified data gaps. Data Gaps 8 (vertical heterogeneity) and 9 (potential source areas) are outside the scope of work for the current task order and will be addressed later.

Step 3: Identify Information Inputs

Additional groundwater investigation will provide soil lithology from boring logs, ground water elevations and VOC concentrations to address data gaps. The data will be used with LTM data to determine groundwater flow direction within and across lithological units, areas where MCLs are exceeded, trends in CVOC concentrations, and potential source areas/receptors.

Step 4: Define the Boundaries of the Study

The study area is defined by the former DDMT property boundary for the MI and groundwater plumes migrating onto or away from the property; the area is approximated by the LTM wells shown on Figure 3.

An initial groundwater sample will be collected and water level measured from each well following installation. The new wells will be incorporated into MI LTM and sampled semiannually until sufficient data is available to revise the sample frequency.

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

The groundwater contaminants at DDMT are CVOCs, but sample analyses include all Target Compound List VOCs to identify degradation products and potential off-site impacts. Groundwater samples will be analyzed for VOCs by Method SW8260.

The findings of the SRI will be used with related studies including an updated risk assessment, groundwater modeling and a VI study to evaluate the RAOs and determine the appropriate RA.

Lithology at each boring will be described using the Unified Soil Classification System (American Society for Testing and Materials [ASTM] D2487-83).

Groundwater elevations will be determined from water level measurements referenced to a surveyed benchmark elevation.

Initial samples will be collected by low-flow sampling with measurement of water quality parameters. Passive diffusion bag samples will then be installed in the middle of the saturated screen for sampling during LTM.

Groundwater concentrations above the MCLs will be used to determine where RA is necessary.

Step 6: Specify Performance or Acceptance Criteria

Groundwater samples will be collected from new monitoring wells and analyzed at a United States Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) laboratory for VOCs by Method SW8260.

Performance criteria for analytical data are presented in Worksheet #12. Acceptance criteria will be based on compliance with the task descriptions in Worksheet #14/16 and the applicable Standard Operating Procedures (SOPs).

A Tennessee-registered land surveyor will establish horizontal and vertical control for all new monitoring wells. Vertical control will be established to 0.01 ft and will be based on the North American Vertical Datum of 1988. Horizontal control will be established to 0.1 ft and will be based on the Tennessee State Plane Coordinate System.

Well installation and development, groundwater sampling, laboratory analysis, and data validation will be conducted in accordance with this work plan and the 2017 Uniform Federal Policy – Quality Assurance Project Plan (UFP-QAPP) (HDR, 2017b).

Step 7: Develop the Plan for Obtaining Data

The project schedule and task summaries are provided on Worksheet #14/16, and sampling design and rationale are provided on Worksheet #17.

QAPP Worksheet #12: Measurement Performance Criteria

Matrix: Groundwater, Investigation Derived Waste (IDW) Soil, Toxicity Characteristic Leaching Procedure (TCLP) Analytical Group: 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

*Samples of water and soil IDW will be collected at the completion of field work or before if storage capacity is reached; field quality control (QC) samples are not required by the disposal facility and will not be collected.

Matrix: IDW Soil (total) Analytical Group: VOCs Concentration Level: Low/Medium

Data Quality Indicators	QC Sample and / or Activity Used to Assess Measurement Performance*	Measurement Performance Criteria
Precision-lab	LCS/LCSD	≤ 20% RPD
Accuracy/bias	Surrogate spike recoveries	QSM v.5.1 App. C Table 23 (or lab if not in QSM) control limits
Accuracy/bias	LCS/LCSD recoveries	QSM v.5.1 App. C Table 23 (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

* Samples of soil IDW will be collected at the completion of field work or before if storage capacity is reached; field quality control (QC) samples are not required by the disposal facility and will not be collected.

QAPP Worksheet #13: Secondary Data Uses and Limitations

Source	Data Type	Data Uses for Current Project
National Weather Service http://w2.weather.gov/climate/index.php?wfo=meg	Meteorological - Historical monthly rainfall, barometric pressure, and long-term averages	Rainfall and barometric pressure during field activities
e ² M, <i>Main Installation Source Areas Investigation, Defense Depot Memphis, Tennessee, Defense Logistics Agency, Revision 0.</i> . February, 2009.	Site assessment and soil sample results	Background information
HDR. April 2017 Long-Term Monitoring Summary Report, Defense Depot Memphis Tennessee, TN4210020570, Revision 0. August 2017	Recent groundwater elevation and analytical data	Background information and selection of well locations.
HDR. Fourth Five-Year Review, Defense Depot Memphis Tennessee, Revision 0. September 2017.	Latest Five-Year Review	Background information
HDR, Supplemental Remedial Investigation Phases 1 and 2 Report, Defense Depot Memphis, Tennessee, TN4210020570, Revision 0. October 2017 (in preparation).	Description of previous investigations; recent lithology, groundwater elevation and analytical data	Background information and selection of well locations.

QAPP Worksheet #14/16: Project Tasks & Schedule

The tasks and schedule for SRI Phase 3 and submittal of deliverables for regulatory agency review are shown below.

Activity	Responsible party	Planned start date	Planned completion date	Deliverable(s)	Planned Deliverable due date
Draft Work Plan	HDR	8/15/17	11/15/17	Phase 3 QAPP, Revision 0	11/15/17
Regulatory Review	USEPA & TDEC	11/16/17	1/15/18	Comments or Approval Letter	1/15/18
Final Work Plan	HDR	1/16/18	2/15/18	Phase 3 QAPP, Revision 1	2/15/18
Well Access Agreements	Army	10/15/17	1/15/18	Final agreements to file	1/15/18
SRI Phase 3 Field Tasks	HDR/ Cascade	3/12/18	5/16/18	Field reports, boring logs and well diagrams to file	5/16/18
Well Sampling	HDR	5/21/18	5/30/18	Samples to laboratory, field reports to file	5/30/18
Phase 3 Sample Analysis	Chemtech	6/1/18	6/22/18	Level 4 report and Electronic Data Deliverable	6/22/18
Data Validation	DSA	6/25/18	7/9/18	Data validation narrative report	7/9/18
Report Preparation	HDR	6/1/18	9/15/18	SRI Phase 3 Summary Report Revision 0	9/15/18
Phase 3 Well LTM Sampling	HDR	10/1/18	4/30/19	Results included in LTM reports	-

The Phase 3 field, analytical and data review tasks are summarized below. Additional details are provided in the SOPs.

Well Access:

Access for well locations on the MI will be cleared with the property manager and tenants/owners by HDR.

Access for off-site locations will be obtained from the property owners by the Army with assistance from HDR, as requested.

Well Installation:

Permits for well installation will be obtained from Shelby County Health Department (SCHD). Proposed well locations will be cleared for utilities prior to drilling.

Borings for Fluvial Aquifer wells will be advanced by rotasonic drilling to the uppermost clay beneath the water table. Each boring will be backfilled to the top of clay and the well installed using Schedule 40 polyvinyl chloride (PVC) casing and screen.

Borings for IAQ wells will be advanced by rotasonic drilling 5-10 ft into the clay at the base of the Fluvial Aquifer. Surface casing will be installed to the bottom of the boring and grouted in place; boring will then be advanced to the target depth and the well installed using Schedule 80 PVC casing and screen.

Wells will be developed no sooner than 24 hours after completion.

Soil cuttings from all well borings will be contained in a roll-off or on plastic sheeting for testing prior to disposal. Cuttings with suspected contamination due to elevated photoionization detector (PID) readings or visible staining will be placed in a covered drum after logging and screening the soil core. Wastewater generated during drilling, well installation and development will be contained for sampling and analysis to determine disposal requirements.

Horizontal and vertical coordinates for each new well will be determined by Tennessee-registered land surveyor after installation.

Project-specific requirements for well installation are provided in the attachment at the end of this worksheet.

Well Abandonment/Repair:

Monitoring well MW-257 will be abandoned in accordance with SCHD requirements. A permit will be obtained from SCHD.

The bollards will be removed from MW-96, which is located on the golf course. The work will be scheduled during dry weather and coordinated with the property manager. The bollard holes will be back-filled with clean soil and grass-seed applied.

Sampling:

Water levels will be measured at the new wells and nearby LTM wells during a 1-day sweep. Measurements will be made at least 24 hours after well development or sampling.

Initial groundwater samples will be collected using bladder pumps for low-flow sampling; if the saturated thickness or recharge is insufficient for a bladder pump, samples will be collected using disposable bailers. Water quality parameters will be measured to confirm well stabilization prior to sampling. PDBs will be installed after the initial sample.

Samples will be collected from IDW (soil cuttings by HDR and wastewater by Cascade Drilling) to determine disposal requirements. Additional samples will be collected from cuttings with suspected contamination.

Analysis:

Analyze groundwater samples for VOCs. Analyze soil IDW samples for VOCs by standard analysis and TCLP. Analyze wastewater samples per analytical requirements of the CERCLA-approved disposal facility.



Quality Control:

Follow SOP 1-General Procedures for Field Personnel; SOP 2-Drilling and Soil Sampling; SOP 3-Well Installation, Development and Abandonment; SOP 4-Groundwater Sample Collection; SOP 7-Sample Control and Documentation; and SOP 8-Sample Packing and Shipping. Field quality control (QC) samples are described on Worksheet #20-Field QC Summary and laboratory QC on Worksheet #28-Analytical Quality Control and Corrective Action.

Data Management:

Laboratory will provide complete analytical data package including raw data (Level 4) for groundwater samples in accordance with Appendix E, SW-846 Reporting Requirements, of the DoD Quality Systems Manual Version 5.0 (July 2013).

Analytical data will be added to the DDMT database after validation.

Documentation and Records:

All well locations will have final coordinates documented with surveyor stamped plats. Field measurements and sample data will be noted in field records and maintained in project files.

Groundwater sample analytical results will be provided in summary tables for analytes detected and in tables showing complete analytical results in an appendix. The complete laboratory analytical reports and data validation will also be provided in appendices to the SRI report.

Assessment/Audit Tasks:

Field sampling procedures are reviewed in biennial audits by QA officer.

Annual laboratory audits are performed through National Environmental Laboratory Accreditation Program.

Data Review Tasks:

Laboratories will verify that data are complete for samples received. All data package deliverables requirements will be met. Data will be reviewed by DSA at the Step I (Verification)/Steps IIa and IIb (Validation) level as described in the *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual* (Intergovernmental Data Quality Task Force, 2005). Achievement of project-specific measurement performance criteria specified in the QAPP and data validation criteria will be evaluated during the data verification and validation, and the analytical measurement error will be assessed. A Steps I/IIa/IIb Data Validation Report will be produced for each Sample Delivery Group.

Validated data and related field logs/notes/records will be reviewed to assess total measurement error and determine overall usability of the data for project purposes. Data limitations will be determined and data will be compared to Project Quality Objectives (PQOs) and required Action Limits. CA is initiated, as necessary. Final data are placed in a database, with any necessary qualifiers, and tables, charts, and graphs are generated.

Attachment – Well Installation

Borings will be advanced using rotasonic drilling methods and continuous soil cores will be collected from ground surface to the termination depth of each boring. Drilling and well installation will be performed by Cascade Drilling, a licensed Tennessee well driller. An HDR field geologist will record field observations and log the soil core. Headspace analysis with a PID will be made on one grab sample selected from each 10-ft soil core based on field observations; the PID reading will be recorded on the boring log.

Fluvial Aquifer Wells

A 6-inch borehole will be advanced up to 5 ft into the uppermost clay beneath the water table. After presence of the clay is confirmed, the boring will be back-filled with bentonite pellets to just below the top of clay.

Well casings will be new, unused, decontaminated, 10-ft sections of 2-inch I.D. Schedule 40 PVC pipe with internal flush, threaded joints. Well screens will be one 10-ft section of Schedule 40 PVC continuous slotted 0.010-inch screen. A threaded PVC cap or point will be placed at the bottom of the screen. Centralizers will be used at the top of the screened section, and every 30 ft along the riser.

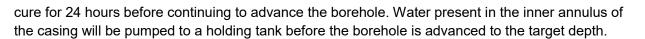
A filter pack of clean, inert, hard, well-rounded coarse sand (8-16 or 10-20 sieve with less than 2% flat particles) will be installed from approximately 1 ft below to 5 ft above the well screen. A minimum 5-ft-thick bentonite seal will be placed above the filter pack. Cement-bentonite grout will be placed in the annular space above the bentonite seal to approximately 6 inches bgs.

Wells will generally be set as flush-mounted completions with brightly painted bollards used where appropriate. The number and location of bollards required at each well will be determined by the FTL. Each well pad will be 3 by 3 ft square and 4 inches thick constructed using Quikcrete® crack resistant (No. 1006) or equivalent concrete mix. The manhole will be a Morrison Bros. Co. 519 9-inch diameter powder-coated single-bolt water tight manhole (or equivalent) placed in the center of the pad and approximately 1-inch higher than the edge of the pad. The manhole will have an identification brass label stamped to clearly show the well ID. Wells will be secured as soon as possible after drilling with corrosion resistant locks.

The wells will be developed by surging the well with the development pump. Well development will be initiated no sooner than 24 hours following grout installation. 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.

Intermediate Aquifer Wells

A 12-inch borehole will be advanced 5 to 10 ft into the uppermost clay at the top of the Jackson Formation/Upper Claiborne Group (base of Fluvial Aquifer). An 8-inch diameter, threaded Schedule 80 PVC or steel surface casing will be installed. After placing the surface casing, the driller will lower a 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



Well casings will be new, unused, 2-inch I.D. Schedule 80 PVC pipe with internal flush, threaded joints. The well screens will be one 10-ft section of new, unused, 2-inch I.D. Schedule 80 PVC continuous slotted 0.010-inch screen. A threaded PVC cap or point will be placed at the bottom of the screen. Centralizers will be used at the top of the screened section and every 30 ft along the riser. The well casing, screen, cap and centralizers will be decontaminated prior to use.

A filter pack of clean, inert, hard, well-rounded sand (less than 2% flat particles) will be installed from approximately 1 ft below to 5 ft above the top of the well screen. The filter pack material will be selected based on the soil type in the zone to be screened; coarse sand (10-20 sieve) or medium sand (20-40 sieve) will be used. The bentonite seal, cement-bentonite grouting, surface completion and well development for IAQ wells will be as described for Fluvial Aquifer wells.

Investigation Derived Waste

IDW consisting of soil cuttings from the borings, wastewater from equipment decontamination and groundwater from well development will be stored for analysis prior to disposal.

Soil cuttings from the well borings will be transported to Dunn Field and placed on plastic sheeting; the cuttings will be covered by plastic sheeting held in place by perimeter weights. Upon completion of drilling, one soil sample will be collected for approximately each 5 cubic yards of soil cuttings. At each sample location, approximately 6 inches of surface soil will be removed and samples will be collected for VOC analysis by standard extraction and by TCLP extraction. Soil samples for standard extraction will be collected with an Encore[™] sampler. Soil samples for TCLP extraction, which requires greater sample volume, will be collected by completely filling a 4-ounce jar for each location. If standard analysis demonstrates the VOC concentrations are less than the Dunn Field RGs, the soil cuttings will be spread on Dunn Field. If the soil concentrations exceed the RGs, TCLP VOC results will be reviewed to confirm the soil meets requirements for disposal as non-hazardous waste at a Comprehensive Environmental Response, Compensation and Liability Act (CERCLA)-approved facility in the Memphis area.

Water IDW will be stored in a Baker frac tank at a location to be determined during mobilization. The tank will be properly labeled with the water source (equipment decon and well development) and the start/end dates. After well installation, development and sampling are completed, grab samples of wastewater will be collected by Cascade Drilling for complete TCLP analysis and disposal as non-hazardous waste at a CERCLA-approved facility in the Memphis area.

QAPP Worksheet #15: Project Action Limits and Laboratory-Specific Detection/Quantitation Limits

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

	Project Action	Project	Achievable Lat	ooratory Limits ²
Analyte	Limit (MCL)	Quantitation Limit Goal	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

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. Project Action Limits for groundwater on the MI are the current (2016) USEPA MCLs.

Matrix: MI Groundwater Analytical Method: VOCs - Microbac Concentration level: Low/Medium

	Project Action	Project	Achievable Lat	ooratory Limits ²
Analyte	Limit (MCL)	Quantitation Limit Goal	MDLs	RLs
	(µg/L)	(µg/L)	(µg/L)	(µg/L)
Carbon tetrachloride	5	5	0.25	5
Chloroform	80	5	0.125	5
Tetrachloroethene	5	5	0.25	5
Trichloroethene	5	5	0.25	5
Vinyl chloride	2	10	0.25	10
cis-1,2-Dichloroethene	70	5	0.25	5

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: Soil IDW Analytical Method: VOCs, total - Chemtech Concentration level: Low

Project Action	Project	Achievable Lab	ooratory Limits ²
Limit	Quantitation Limit Goal	MDLs	RLs
(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)
0.215	0.005	0.0005	0.005
0.917	0.005	0.0005	0.005
0.1806	0.005	0.0005	0.005
0.182	0.005	0.0005	0.005
0.0294	0.005	0.0005	0.005
0.755	0.005	0.0005	0.005
	(<i>mg/kg</i>) 0.215 0.917 0.1806 0.182 0.0294	Project Action Limit Quantitation Limit Goal (mg/kg) (mg/kg) 0.215 0.005 0.917 0.005 0.1806 0.005 0.182 0.005 0.0294 0.005	Project Action Limit Quantitation Limit Goal MDLs (mg/kg) (mg/kg) (mg/kg) 0.215 0.005 0.0005 0.917 0.005 0.0005 0.1806 0.005 0.0005 0.182 0.005 0.0005 0.0294 0.005 0.0005

Project Action Limits for Total VOCs - Dunn Field RGs

Matrix: Soil IDW Analytical Method: VOCs, TCLP - Chemtech Concentration level: Low

	Project Action	Project	Achievable La	poratory Limits ²
Analyte	Limit	Quantitation Limit Goal	MDLs	RLs
	(mg/L)	(mg/L)	(mg/L)	(mg/L)
Benzene	0.5	0.025	0.0016	0.025
Carbon tetrachloride	0.5	0.025	0.0010	0.025
Chlorobenzene	100.0	0.025	0.0025	0.025
Chloroform	6.0	0.025	0.0017	0.025
1,2-Dichloroethane	0.3	0.025	0.0024	0.025
1,1-Dichloroethene	0.7	0.025	0.0024	0.025
Methyl ethyl ketone (MEK) (2-Butanone)	200.0	0.130	0.0066	0.130
Tetrachloroethene	0.7	0.025	0.0014	0.025
Trichloroethene	0.5	0.025	0.0014	0.025
Vinyl chloride	0.2	0.025	0.0017	0.025

Project Action Limits for TCLP VOCs – Resource Conservation and Recovery Act TCLP regulatory limits (hazard characteristic)

QAPP Worksheet #17: Sampling Design and Rationale

The sampling activities will be conducted in accordance with the field SOPs listed on Worksheet #21 and provided in Appendix A. The number of samples and the analytical parameters are summarized in Worksheet #18. Samples to be collected consist of groundwater samples and soil cuttings from well borings.

Physical Boundaries for the Area

The sampling area is defined by the former DDMT property boundary for the MI and groundwater plumes migrating onto or away from the property; the extent is approximated by the LTM wells shown on Figure 3. The area may be expanded as the SRI proceeds.

Basis for the Placement and Number of Sample Locations

Well locations were selected to address the data gaps listed in Worksheet #11 based on review of analytical results and groundwater elevation contours from April 2017 LTM, which included the SRI Phases 1 and 2 wells. Isopleths for primary contaminants (PCE and TCE), groundwater elevation contours and proposed locations are shown on Figure 10. The rationale and general location for each well is in Table 2 below. The planned well construction data for Phase 3 wells including location coordinates, aquifer to be screened, and estimated depths for groundwater, top of clay, surface casing (where needed) and top of screen are listed on Table 3. The planned screen length is 10 feet for all wells. Construction data for existing MI wells are provided on Table 4.

Rationale	Well ID and Location
Offsite extent of CVOCs and groundwater flow direction in IAQ at northwest MI	I-1: N of MW-256
Potential CVOC contamination and groundwater flow direction in IAQ at sink in south-central MI	I-2: Central area of sink, near MW-259
Extent of CVOCs and groundwater flow direction in Fluvial aquifer and IAQ at the window; assess potential contaminant source at MW-107	F-1 and I-3: N of MW-207A/B.
	F-2: at IAQ wells MW-214A/B.
	F-3: at Bldg 720, near IAQ well MW-107.
Off-site extent of CVOCs upgradient of TTA-1S	F-4: WSW of MW-279.
Off-site extent of CVOCs upgradient of TTA-1N	F-5: NW of MW-269.
	F-6: S of MW-269.
Extent of CVOCs upgradient of TTA-2 West (Near MW-267)	F-7: Side-gradient (ESE) of MW-267.
	F-8: Upgradient (N) of MW-267.
	F-9: Adjacent to IAQ well MW-268.
Extent of CVOCs in North-Central plume	F-10: Upgradient (off-site) NE of MW-263.
	F-11: Mid-section of plume, E of MW-260.

Table 2. Phase 3 Well Locations



Rationale	Well ID and Location
	F-12: Side gradient, near downgradient end of plume, ENE of MW-281.
	F-13: Side gradient, SSE of MW-258.
Extent of CVOCe in Building 825 plume	F-14: Upgradient (NW) of MW-143.
Extent of CVOCs in Building 835 plume	F-15: Upgradient (WNW) of MW-62.
	F-16: Downgradient (E) of MW-97
Extent of CVOCs in S-C plume; replace PZ-03	F-17: E of MW-261, near PZ-03.
	F-18: Between PZ-03 and MW-271
Extent of CVOCs at MW-270	F-19: E of MW-270.
Area of central MI without previous investigation,	F-20: N of Bldg 560 center.
extent of CVOCs and groundwater flow direction in Fluvial aquifer and IAQ	I-4: adjacent to F-20

Soil IDW samples will be collected upon completion of drilling. One soil sample will be collected for approximately each 5 cubic yards of soil cuttings. At each sample location, approximately 6 inches of surface soil will be removed prior to sampling. Water IDW samples will be collected after well installation, development, and sampling are completed; grab samples of wastewater will be collected by Cascade Drilling for TCLP analysis per requirements of the disposal facility.

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 chain-of-custody (CoC) form. The sample ID will include the location (MW-xxx) and sampling event (SRI3), for example MW-285-SRI3.

IDW samples will include the medium (S for soil and W for water), the sequence and sample event, for example IDW-Sxx-SRI3).

Prefixes will replace the location for trip blanks (TB-x) and rinse blanks (RB-x) and suffixes will identify the locations of MSs or duplicates (MW-xxx-SRI3-MS). Additional detail is provided in SOP-7, *Sample Control and Documentation*.

QAPP Worksheet #18: Sampling Locations and Methods/SOP Requirements

Sampling Location	Number of Locations	Matrix	Depth (ft)	Analytical Group	Concentration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference ¹	Rationale for Sampling Location
SRI Phase 3 Fluvial Aquifer Wells	20	GW	75-150	VOCs	Low	20 field, 2 duplicate	SOP 4	Determination of hydraulic gradient and contaminant extent
SRI Phase 3 IAQ Wells	4	GW	140-215	VOCs	Low	4 field, 1 duplicate	SOP 4	Determination of hydraulic gradient and contaminant extent
Soil cuttings IDW	1 per 5 cubic yard soil cuttings	Soil	NA	VOCs, TCLP VOCs	Low	1 per 5 cubic yard soil cuttings	SOP 2	Determination of waste classification for disposal
Water IDW	1 per full Baker tank	Water	NA	per disposal facility requirements	Low	1 per full Baker tank	SOP 4	Determination of waste classification for disposal

¹ Specify the appropriate letter or number from the Project Sampling SOP References (Worksheet #21). GW: Groundwater

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times

Analytical Laboratory (Name, sample receipt address, point of contact, e-mail, and phone numbers): Chemtech, 284 Sheffield Street, Mountainside, New Jersey 07092, Kurt Hummler, 908-789-8900, <u>kurt@chemtech.net</u>

Required accreditations/certifications: DoD ELAP

Back-up Laboratory: Microbac Laboratories, Inc., 158 Starlite Drive, Marietta, Ohio 45750, Michelle Taylor, 800-373-4071, michelle.taylor@microbac.com, 800-373-4071

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, water	USEPA SW846 5030B/8260B (Chemtech L-3) (Microbac L-1)	Chemtech: 10/20/2018 Microbac: 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 5030B/8260B (Chemtech L-3) (Microbac L-1)	Chemtech: 10/20/2018 Microbac: 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)	1 week for Level 3; 3 weeks for Level 4
VOCs	IDW Soil (total)	USEPA SW846 5030B/8260B (Chemtech L-3)	Chemtech: 10/20/2018	Terracore kit	Chill ≤ 6 degrees Celsius (°C)	14 Days	1 week for Level 3; 3 weeks for Level 4
VOCs	IDW Soil (TCLP) prep	USEPA SW846 1311 (Chemtech L-4)	Chemtech: 10/20/2018	8 ounce glass jar Teflon®-lined septum	Chill ≤ 6 degrees Celsius (°C)	14 Days	1 week for Level 3; 3 weeks for Level 4

Matrix ¹	Analyte/ Analytical Group	Field Samples	Field Duplicates ²	Matrix Spikes²	Matrix Spike Duplicates ²	Field Blanks²	Equipment Blanks ²	Trip Blanks ²	Other	Total # analyses
Groundwater - Initial	VOCs	24	3	2	2	0	2	4	0	37
Groundwater - LTM	VOCs	24	3	2	2	0	0	2	0	33

Note:

1) Initial samples will be collected using low-flow method over 4 days. Following the initial samples, passive diffusion bags will be installed for sampling during LTM. LTM samples will be collected over 1 to 2 days.

2) Samples of soil and water IDW will be collected at completion of field work or before if storage capacity is reached; field quality control (QC) samples are not required by the disposal facility and will not be collected.

QAPP Worksheet #21: Field SOPs

Method/ SOP Reference	Title, Revision Number and Date	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)
SOP1	General Procedures for Field Personnel, Rev. 2, August 2017	HDR	Log books, first aid kit, personal protective equipment	Ν
SOP2	Drilling and Soil Sampling, Rev. 3, August 2017	HDR	Drilling equipment, sample jars, disposable scoops	Ν
SOP3	Well Installation, Development and Abandonment, Rev. 1, November 2014	HDR	Well construction materials, cement/bentonite grout, concrete	Ν
SOP4	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	Ν
SOP7	Sample Control and Documentation, Rev. 3, August 2017	HDR	Sampling log book, DQCR forms, digital camera, CoC forms	Ν
SOP8	Sample Packing and Shipping, Rev. 2, October 2016	HDR	Sample bottles, bubble wrap, ice, zip lock bags, coolers, tape, custody seals	Ν
SOP9	Sampling Equipment Decontamination, Rev. 2, August 2017	HDR	American Society for Testing and Materials (ASTM) Type II water (supplied by lab) or distilled water, pesticide-grade methanol, Alconox detergent, brushes	Ν
L-2	Standard Operating Procedure Sample Receiving and Login, Revision 19, September 2017	Microbac	Thermometers, hood, pH strips, IR temperature guns, Geiger counter, disposable gloves	Ν
L-5	Sample Receipt, Sample Acceptance Policy, Sample Login, Sample Identification, & Sample Storage, Revision 21, July 2017	Chemtech	Thermometers, hood, pH strips, IR temperature guns, Geiger counter, disposable gloves	Ν

Note: The drilling subcontractor, Cascade Drilling, will collect and submit the IDW water samples per their SOP and the disposal facility requirements.

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection

Field Equipment	Activity	SOP Reference	Title or position of responsible person	Frequency	Acceptance Criteria	Corrective Action
GeoXH	Field Global Positioning System 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], 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. Send 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

QAPP Worksheet #23: Analytical SOPs

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 2	Drilling and Soil Sampling, Revision 2, February 2015	NA	Soil sampling	Terracore sampling kit, glass jars with Teflon [®] lined lids	Ν
SOP 4	Groundwater Sample Collection, Revision 1, November 2014	Screening	Field water quality (pH, conductivity, DO, ORP, temperature, turbidity)	Field multimeter (YSI, Horiba, Lamotte)	Ν
L-1 Microbac	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	Ν
L-3 Chemtech	VOCs by gas chromatography (GC)/mass spectrometry (MS) - SW 846 Method 8260B/C (M8260B/C-SWGCMSVOA) Rev. 22 – 05/05/2017	Definitive	VOCs in Water and Soil	-Hewlett Packard 5890 GC -Hewlett Packard 5971/5972 mass selective detectors MS -Hewlett Packard MSD Chemstation Software	Ν
L-4 Chemtech	Sample Preparation for Toxicity Characteristics Leaching Procedure (M1311-TCLP) Rev. 10 – 06/23/2017	Definitive	ZHE extraction of soils for TCLP VOCs	-Rotary agitator -ZHE extraction vessels -filter apparatus	Ν

QAPP Worksheet #24: Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference ¹
	Mass spectral ion intensities with 4- bromofluoro- benzene (BFB)	Every 12 hours prior to initial calibration (ICAL), initial calibration verification (ICV) or continuing calibration verification (CCV)	$\begin{array}{l} \underline{Mass-Ion Abundance Criteria} \\ 50-15-40\% of mass 95 \\ 75-30-60\% of mass 95 \\ 95-base peak, 100\% relative abundance \\ 96-5-9\% of mass 95 \\ 173-<2\% of mass 174 \\ 174->50\% of mass 95 \\ 175-5-9\% of mass 174 \\ 176->95\% and <101\% of mass 174 \\ 177-5-9\% of mass 176 \\ \hline (Established criteria in Table 4 of SW-846 \\ 8260B) \end{array}$	Retune instrument and repeat BFB check. Flagging criteria are not appropriate.	Analyst	L-1 L-3
GC/MS for VOCs in Water, Soil SW-846 8260B	Initial multipoint calibration for all analytes (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. Relative Standard Deviation (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. Flagging criteria are not appropriate.	Analyst	L-1 L-3

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Instrument	Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference ¹
GC/MS for VOCs in Water, Soil SW-846 8260B (continued)	CCV	Once per each 12 hours, prior to sample analysis (criteria for these checks must be met prior to sample analysis)	1. Average Relative RF 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).	 Evaluate system and take CA. Rerun calibration check. If still out, prepare new calibration curve for any analyte not meeting criteria. Reinject any samples analyzed after criteria were exceeded. 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 L-3
	Retention time (RT) window position establishment for each analyte and surrogate		NA	Analyst	L-1 L-3	

⁴Specify the appropriate reference letter or number from the Analytical SOP References (Worksheet #23).

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection

	Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference ¹
١	GC/MS for /OCs in Vater, Soil	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) M8260B/C- SWGCMS VOA (L-3)

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

QAPP Worksheet #26 & 27: Sample Handling, Custody, and Disposal

Sampling Organization: HDR Laboratory: Chemtech 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 SHIP	MENT	
Sample Collection	HDR Field Team	HDR SOP 2, HDR SOP 4
Sample Packaging	HDR Field Team	HDR SOP 8
Coordination of Shipment	HDR Field Team and Project Chemist, Laboratory (Chemtech) PM	HDR SOP 8
Type of Shipment/Carrier: Cardboard carton (air sample in Summa [™] canister), shipped via FedEx Ground.	HDR Field Team	HDR SOP 8
Type of Shipment/Carrier: Cooler with ice (soil samples in Terracore kit of three 40-mL vials), shipped via FedEx next morning delivery.	HDR Field Team	HDR SOP 8
SAMPLE RECEIPT AND ANALYSIS		
Sample Receipt	Laboratory (Chemtech, Microbac) sample custodian	Microbac SOP L-2, Chemtech SOP L-5
Sample Custody and Storage	Laboratory (Chemtech, Microbac) sample custodian	Microbac SOP L-2, Chemtech SOP L-5
Sample Preparation	Laboratory (Chemtech, Microbac) sample preparation chemist or analyst	Microbac SOP L-1, Chemtech SOPs L-3, L-4
Sample Determinative Analysis	Laboratory (Chemtech, Microbac) sample analyst	Microbac SOP L-1, Chemtech SOP L-3

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 (Chemtech, Microbac) sample custodian.	Microbac SOP L-2, Chemtech SOP L-5
Sample Extract/ Digestate Storage (No. of days from extraction/digestion): 60 days from data package report	Laboratory (Chemtech, Microbac) sample custodian	Microbac SOP L-2, Chemtech SOP L-5
SAMPLE DISPOSAL		
Number of Days from Analysis: 60 days from data package report	Laboratory (Chemtech, Microbac) sample custodian	Microbac SOP L-2, Chemtech SOP L-5

QAPP Worksheet #28: Analytical Quality Control and Corrective Action

Matrix: Groundwater, Soil Analytical Group: VOCs by GC/MS Analytical Method/SOP: SW-846 8260B / Microbac L-1 & Chemtech L-3

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 relative retention time (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
Internal standard (IS) –RT and area response checked from daily calibration check	Every field sample, standard, and QC sample.	RT ± 30 seconds and extracted ion current profile (EICP) area within - 50% to +100% of <i>the mid-point</i> <i>standard in the ICAL</i> for each IS compound.	 Inspect MS and GC for malfunctions. Take appropriate CAs. Reanalyze samples analyzed while system was malfunctioning. 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 <i>the mid-</i> <i>point standard in the</i> <i>ICAL</i> 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.	 Recalculate results. Locate and fix the source of the problem. 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 ≤ 30%	 Review lab QC data to determine if they are in control. Qualify data. Use data to evaluate whether proper collection procedures were followed. Determine further CA. 	Analyst	Precision- overall	RPD ≤ 30%

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 Table G-4. RPD ≤ 30 %	Qualify data.	Analyst	Precision- overall and accuracy/ bias	In-house control limits (or, if not established, control limits in DoD QSM 5.1 Table G-4. RPD ≤ 30 % 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 Table G-4. Up to 3 marginal exceedances if full list of 66 analytes is run.	 The analytical batch must be reprocessed. Reprep and analyze LCS and affected samples. 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 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 Table G-3.	 Recalculate result, and reanalyze sample if still out. Re-extract and reanalyze sample, if still out. Report both analyses and document in report that steps 1 and 2 were performed. Qualify the data. 	Analyst	Accuracy/ bias	In-house control limits (or, if not established, control limits in DoD QSM 5.1 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.	 Take and document appropriate CA Reanalyze all samples processed with a contaminated blank. 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	 Review lab QC data to determine if there is a laboratory problem. If same compounds are found in field samples at similar concentrations, qualify the data. OR 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	 Review lab QC data to determine if there is a laboratory problem. If same compounds are found in field samples at similar concentrations, qualify the data. OR 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.

QAPP Worksheet #29: Project Documents and Records

Sample Collection and Field Records							
Record	Generation	Verification	Storage location/archival				
Field Notes, and Logbooks	HDR FTL	HDR PM, Chemist	HDR Network Drive Project Folder				
GIS files	HDR FTL	HDR PM	HDR Network Drive Project Folder				
CoC Forms	HDR FTL	HDR PM, Chemist	HDR Network Drive Project Folder				
Airbills	HDR FTL	HDR PM, 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/archiva							
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)			
Laboratory Information Management System (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 (IDW Water and IDW Soil)				
CoC Forms	X	Х				
Equipment Logs	Х	Х				
Sample Prep Logs	X	Х				
Analytical Run Logs	X	Х				
CA Forms	X	Х				
Extraction Records	Х	Х				
Reported Sample Results	X	Х				
Raw Data Printouts	Х					
LIMS Reports	X	Х				
Data Packages and Checklists	Х	Х				

QAPP Worksheet #31, 32 & 33: Assessments and Corrective Action

Assessments:

Assessment Type	Responsible Party & Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable due date
Off-Site Laboratory TSA	DoD ELAP personnel or contractor	Annual	NA	ELAP annual certification audit report	NA
Laboratory Performance Audit	HDR Data reviewer/ validator, HDR Project Chemist	Ongoing with data package data validation	NA	Email from 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 findinas	Assessment Response Documentation	Timeframe for Response	Responsibility for Implementing Corrective Action	Responsible for monitoring Corrective Action implementation	
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

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

QAPP Worksheet #35: Data Verification Procedures

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 Chemist 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.	HDR Project Chemist

QAPP Worksheet #36: Data Validation Procedures

Data Validator: Project Chemist, HDR

Analytical Group/Method	Organics (VOCs in Soil, VOCs in Air)
Data Deliverable Requirements:	Level 4 data package including all instrument raw data
Analytical Specifications:	Per method and this QAPP
Measurement of Performance Criteria:	DQOs in this QAPP
Percent of Data Packages to be Validated:	100%
Percent of Raw Data to be Reviewed:	10%
Percent of Results to be Recalculated:	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
Validation Code:	S2bVM (100%), S3VM (10%)
Electronic Validation Program/Version:	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

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

%R for LCS

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

For Precision:

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

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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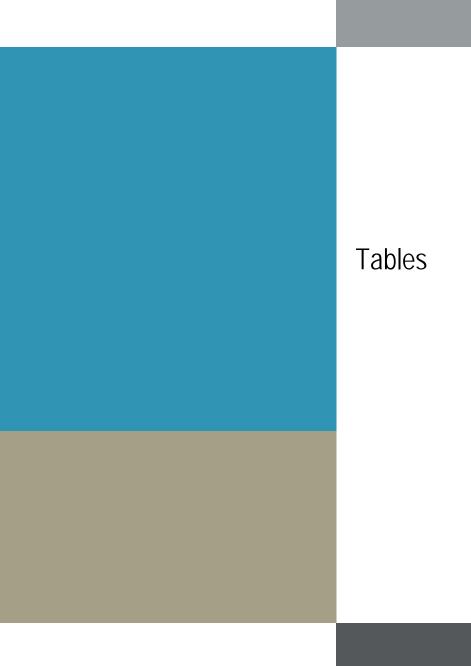


TABLE 3 PROPOSED WELL LOCATIONS AND DEPTHS SRI PHASE 3 WORK PLAN Main Installation - Defense Depot Memphis, Tennessee

								Top of		Surface	Top of	
			Ground	Groundwater	Top of Clay	Surface	Water	Clay	Saturated	Casing	Screen	Total Well
	Easting	Northing	Elevation	Elevation	Elevation	Casing Depth	Depth	Depth	Thickness	Depth	Depth	Depth
Location	(ft)	(ft)	(ft, NAVD)	(ft, NAVD)	(ft, NAVD)	(ft, bgs)	(ft, bgs)	(ft, bgs)	(ft)	(ft, bgs)	(ft, bgs)	(ft, bgs)
F-1	803201	278087	303	207	148	NA	96	155	59	NA	101	111
F-2	803886	277907	300	215	167	NA	85	133	48	NA	90	100
F-3	803084	278429	304	206	147	NA	98	157	59	NA	103	113
F-4	800254	275875	306	211	177	NA	95	129	34	NA	100	110
F-5	800060	276467	295	211	189	NA	84	106	22	NA	89	99
F-6	800119	276255	299	211	183	NA	88	116	28	NA	93	103
F-7	806338	276987	304	226	209	NA	78	95	17	NA	83	93
F-8	806042	277409	303	230	214	NA	73	89	16	NA	78	88
F-9	805257	277204	303	212	185	NA	91	118	27	NA	96	106
F-10	806050	279130	288	238	217	NA	51	71	20	NA	56	66
F-11	804968	278382	300	231	209	NA	69	91	22	NA	74	84
F-12	803960	278220	300	221	175	NA	79	125	46	NA	84	94
F-13	804531	277863	300	222	197	NA	78	103	25	NA	83	93
F-14	801079	278428	290	210	190	NA	80	101	20	NA	85	95
F-15	801553	278346	292	208	193	NA	84	99	15	NA	89	99
F-16	802435	276082	300	206	175	NA	94	125	31	NA	99	109
F-17	802858	276346	296	205	175	NA	91	121	30	NA	96	106
F-18	803356	276365	300	204	180	NA	95	120	24	NA	100	110
F-19	805292	275515	273	206	195	NA	67	78	11	NA	72	82
F-20	803750	277027	301	204	152	NA	97	149	52	NA	102	112
I-1	801231	279461	285	173	220	140	112	65	NA	73	108	118
I-2	804482	276239	298	195	155	110	103	143	NA	151	166	176
I-3	803251	278085	302	206	148	140	96	154	NA	162	177	187
1-4	803801	277026	301	202	152	110	99	149	NA	157	172	182

Note:

1) Depths and elevations for Phase 3 wells are estimated based on elevation contour maps for ground surface, groundwater and top of clay.

2) All monitoring wells will have 10-foot screens.

3) Estimated groundwater depths are for the Fluvial Aquifer at wells F-1 to F-20 and the Intermediate Aquifer at wells I-1 to I-4.

NA: Not applicable

ft: feet

bgs: below ground surface

btoc: below top of casing

NAVD: North American Vertical Datum of 1988

								Surface			
					Top of Casing	Ground	Depth to	Casing	Riser	Screen	Total Well
			Northing	Easting	Elevation	Elevation	Top of Clay	Depth	Length	Length	Depth
Well	Aquifer	Area	(ft)	(ft)	(ft, NAVD)	(ft, NAVD)	(ft, bgs)	(ft, bgs)	(ft)	(ft)	(ft, btoc)
DR1-1	Fluvial	TTA-1S	276300	800856	293.14	293.42	141.5	NA	121.7	20	141.7
DR1-1A	Fluvial	TTA-1S	276307	800863	293.00	293.37	-	NA	89.2	20	109.2
DR1-2	Fluvial	TTA-1N	276537	801153	290.00	291.39	115.0	NA	97.7	20	117.7
DR1-3	Fluvial	TTA-1S	276527	801416	290.93	291.11	126.5	NA		20	129.7
DR1-4	Fluvial	TTA-1S	276231	801400	292.78	293.00	126.0	NA	106.3	20	126.3
DR1-5	Fluvial	TTA-1S	276080	800828	294.46	294.86	144.0	NA	124.7	20	144.7
DR1-5A	Fluvial	TTA-1S	276087	800835	294.51	294.87	-	NA	90.0	20	110.0
DR1-6	Fluvial	TTA-1S	276044	801103	293.17	293.50	136.0	NA	114.4	20	134.4
DR1-6A	Fluvial	TTA-1S	276035	801104	293.28	293.58	-	NA	90.9	20	110.9
DR1-7	Fluvial	TTA-1N	276791	801441	289.15	289.46	129.5	NA	108.3	20	128.3
DR1-8	Fluvial	TTA-1N	276752	800875	290.09	290.47	111.0	NA	92.7	20	112.7
DR2-1	Fluvial	TTA-2	276772	806498	304.90	305.08	94.0	NA	73.9	20	93.9
DR2-2	Fluvial	TTA-2	276771	806659	304.30	304.67	92.0	NA		15	93.4
DR2-3	Fluvial	TTA-2	276539	806203	303.44	303.66	106.0	NA	93.0	20	113.0
DR2-4	Fluvial	TTA-2	276456	806633	303.55	303.96	106.5	NA	88.1	20	108.1
DR2-5	Fluvial	TTA-2	276831	806180	305.41	305.72	99.0	NA		15	99.5
DR2-6	Fluvial	TTA-2	276644	805861	304.70	304.92	116.0	NA	94.6	20	114.6
MW-16	Fluvial	Background	278838	807100	299.86	300.19	75.0	NA	57.6	15	72.6
MW-19	Fluvial	Background	278946	800782	290.57	290.86	93.5	NA	83.1	10	93.1
MW-21	Fluvial	TTA-1N	276473	800602	295.00	295.30	-	NA	92.1	15	107.1
MW-22	Fluvial	TTA-1S	275912	800702	298.04	298.49	-	NA	95.4	10	105.4
MW-23	Fluvial	Background	275791	801817	298.99	299.24	-	NA	101.2	10	111.2
MW-24	Fluvial	Background	275616	803539	299.51	299.81	114.7	NA	97.3	15	112.3
MW-25A	Fluvial	TTA-2	275975	805521	269.88	270.13	-	NA	73.0	10	83.0
MW-26	Fluvial	TTA-2	276508	805962	303.69	303.89	110.0	NA	97.6	10	107.6
MW-34	Intermediate	Window	279411	801918	299.97	300.80	158.3	NA	136.6	20	156.6
MW-38	Intermediate	Window	279141	802450	307.45	308.45	155.0	NA	139.9	15	154.9
MW-39	Fluvial	W-C	277281	802598	296.28	296.58	-	NA	95.5	20	115.5
MW-39A	Upper Claiborne	W-C	277278	802608	298.61	298.70	165.0	NA	148.1	20	168.1
MW-50	Fluvial	TTA-2	276456	807065	298.82	299.32	126.0	NA	115.0	10	125.0
MW-52	Fluvial	SE	275372	805897	279.26	279.71	104.0	NA	94.0	10	104.0
MW-53	Fluvial	Background	279177	805136	306.38	305.58	83.0	NA	72.5	10	82.5
MW-55	Fluvial	Background	279301	801205	292.08	292.48	74.0	NA	64.0	10	74.0
MW-62	Fluvial	B-835	278290	801858	293.71	293.90	97.0	NA	86.1	10	96.1
MW-63A	Fluvial	N-C	278200	803573	305.96	306.33	140.0	NA	130.0	10	140.0
MW-63B	Fluvial	N-C	278201	803558	305.78	306.22	-	NA	115.0	10	125.0

								Surface			
					Top of Casing	Ground	Depth to	Casing	Riser	Screen	Total Well
			Northing	Easting	Elevation	Elevation	Top of Clay	Depth	Length	Length	Depth
Well	Aquifer	Area	(ft)	(ft)	(ft, NAVD)	(ft, NAVD)	(ft, bgs)	(ft, bgs)	(ft)	(ft)	(ft, btoc)
MW-64	Fluvial	TTA-2	276952	805006	304.21	304.46	113.0	NA		10	112.0
MW-66A	Fluvial	TTA-1N	276626	799793	284.22	284.34	94.0	NA	74.6	20	94.6
MW-85	Fluvial	TTA-2	276704	806065	304.13	304.50	111.5	NA	95.9	15	110.9
MW-88	Fluvial	TTA-2	276879	806513	305.15	305.47	97.0	NA	82.0	15	97.0
MW-89	Intermediate	Window	278287	802555	303.98	304.38	94.5	NA	147.0	30	177.0
MW-90	Intermediate	Window	278284	802540	304.19	304.64	-	NA	115.0	30	145.0
MW-92	Fluvial	TTA-2	276614	806490	304.41	304.78	107.9	NA	93.0	15	108.0
MW-93	Fluvial	Background	275542	804440	294.08	294.31	107.0	NA	92.0	15	107.0
MW-94A	Fluvial	W-C	276806	803086	303.00	303.23	117.5	NA	109.6	10	119.6
MW-96	Fluvial	TTA-2	276310	806320	289.02	289.67	95.5	NA	75.5	20	95.5
MW-97	Fluvial	S-C	276074	802139	297.44	297.70	117.5	NA	97.5	20	117.5
MW-98	Fluvial	W-C	276891	802573	294.43	294.93	147.0	NA	137.0	10	147.0
MW-99	Fluvial	Background	277443	801115	285.33	285.69	111.5	NA	91.5	20	111.5
MW-100B	Fluvial	TTA-1N	276601	800854	290.92	291.47	127.5	NA	107.4	20	127.4
MW-101 ¹	Fluvial	TTA-1S	276204	801110	291.74	291.98	134.0	NA	89.0	15	104.0
MW-102B	Fluvial	Background	275761	800708	311.40	312.07	140.5	NA	120.5	20	140.5
MW-103	Fluvial	N-C	278691	805160	301.37	301.89	90.0	NA	70.0	20	90.5
MW-104	Fluvial	N-C	278676	805417	291.98	292.15	90.5	NA	66.7	20	87.2
MW-107 ¹	Upper Claiborne	Window	278419	803010	304.92	305.18	158.0	NA	128.0	15	143.0
MW-108	Upper Claiborne	W-C	277658	802986	303.07	303.25	170.0	NA	160.0	10	170.0
MW-113	Fluvial	TTA-2	276685	806279	304.81	304.92	105.0	NA	96.0	10	106.0
MW-140	Memphis	Window	279061	801716	298.12	298.16	76.0	NA	224.6	20	244.6
MW-141	Intermediate	Window	278019	802571	303.71	303.70	110.0	NA	148.7	20	168.7
MW-142	Fluvial	B-835	278056	801629	291.18	291.49	98.5	NA	85.0	20	105.0
MW-143	Fluvial	B-835	278301	801201	290.66	290.90	92.5	NA	78.6	20	98.6
MW-197A	Upper Claiborne	W-C	276975	802042	291.64	291.54	176.0	NA	161.7	15	176.7
MW-197B	Fluvial	W-C	276973	802037	291.43	291.43	-	NA	93.8	15	108.8
MW-198	Fluvial	B-835	277776	802142	291.78	292.20	105.0	NA	90.3	15	105.3
MW-199A	Intermediate	B835	277756	802574	301.90	301.84	117.0	NA	146.1	15	161.1
MW-199B	Fluvial	B-835	277752	802576	302.06	302.07	-	NA	104.6	15	119.6
MW-200	Fluvial	W-C	277006	802859	300.18	300.51	117.0	NA	103.2	15	118.4
MW-202A	Intermediate	Window	278686	802111	299.67	299.69	86.0	NA	176.2	15	191.2
MW-202B	Intermediate	Window	278693	802112	299.92	299.74	88.0	NA	118.8	15	133.8
MW-203A	Upper Claiborne	W-C	276842	801740	290.70	290.80	167.0	NA	142.9	20	162.9
MW-203B	Fluvial	W-C	276821	801742	290.87	291.10	-	NA	93.0	20	113.0
MW-204A	Fluvial	W-C	276725	802168	292.59	292.49	149.0	NA	133.3	15	148.3

								Surface			
					Top of Casing	Ground	Depth to	Casing	Riser	Screen	Total Well
			Northing	Easting	Elevation	Elevation	Top of Clay	Depth	Length	Length	Depth
Well	Aquifer	Area	(ft)	(ft)	(ft, NAVD)	(ft, NAVD)	(ft, bgs)	(ft, bgs)	(ft)	(ft)	(ft, btoc)
MW-204B	Fluvial	W-C	276708	802167	292.71	293.00	-	NA	94.9	15	109.9
MW-205A	Upper Claiborne	W-C	277157	802277	292.30	292.40	158.0	NA	141.3	15	156.3
MW-205B	Fluvial	W-C	277173	802278	292.16	292.30	-	NA	97.3	15	112.3
MW-206A	Fluvial	W-C	277219	802792	300.32	300.35	144.5	NA	127.3	15	142.4
MW-206B	Fluvial	W-C	277201	802795	300.30	300.12	-	NA	96.7	15	111.7
MW-207A	Upper Claiborne	W-C	277653	803192	304.05	304.45	167.0	NA	149.9	15	164.9
MW-207B	Fluvial	W-C	277665	803193	304.06	304.42	-	NA	108.5	15	123.5
MW-208A	Upper Claiborne	W-C	277382	802799	302.21	302.40	198.0	NA	183.4	15	198.5
MW-208B	Fluvial	W-C	277397	802815	302.13	302.08	-	NA	106.7	15	121.7
MW-209A	Intermediate	B-835	277574	802507	298.45	298.36	205.0	NA	189.0	15	204.0
MW-209B	Fluvial	B-835	277582	802520	298.89	298.72	-	NA	102.3	15	117.3
MW-210A	Intermediate	W-C	277239	801958	289.61	289.70	139.0	NA	177.0	15	192.0
MW-210B	Fluvial	W-C	277228	801952	289.54	289.83	-	NA	97.0	15	112.0
MW-211	Intermediate	Window	278001	802974	304.14	304.09	99.0	NA	166.3	15	181.3
MW-212	Fluvial	B-835	278028	802225	295.74	295.68	100.0	NA	85.3	15	100.3
MW-213	Fluvial	B-835	278427	801669	294.22	294.20	92.0	NA	77.3	15	92.3
MW-214A	Upper Claiborne	N-C	277878	803907	304.01	303.96	137.0	NA	119.1	15	134.1
MW-214B	Upper Claiborne	N-C	277876	803922	304.10	303.96	-	NA	101.6	15	116.6
MW-215A	Upper Claiborne	N-C	277298	804164	304.97	304.86	147.0	NA	128.8	15	143.8
MW-215B	Fluvial	N-C	277298	804177	305.03	304.98	-	NA	105.4	15	120.4
MW-216	Fluvial	S-C	276025	801996	297.72	297.63	119.0	NA	99.9	15	115.0
MW-217	Fluvial	TTA-2	276671	805214	304.65	304.51	116.0	NA	101.8	15	116.8
MW-218	Fluvial	TTA-2	276937	805628	306.07	306.00	114.0	NA	98.9	15	114.0
MW-219	Fluvial	TTA-1N	276429	800461	295.13	295.00	110.0	NA	98.0	15	113.0
MW-229	Intermediate	Window	279294	802837	311.78	312.09	93.0	NA	188.4	20	208.4
MW-252	Intermediate	Window	278789	801365	294.16	294.40	87.0	97.0	126.1	20	146.1
MW-253	Intermediate	Window	278287	801191	290.47	290.80	107.0	117.0	118.3	20	138.3
MW-254	Memphis	Window	279334	800858	292.84	293.28	75.0	85.0	285.8	20	305.8
MW-255	Memphis	Window	279305	801227	291.84	292.38	72.0	82.0	284.7	20	304.7
MW-256	Intermediate	Window	279302	801244	292.68	293.40	69.0	79.0	127.1	20	147.1
MW-258	Fluvial	N-C	278126	804427	304.37	304.83	105.0	NA	79.3	20	99.3
MW-259	Fluvial	TTA-2	276279	804451	290.77	291.44	136.0	NA	98.6	20	118.6
MW-260	Fluvial	N-C	278398	804376	304.16	304.45	96.0	NA	68.0	20	88.3
MW-261	Fluvial	S-C	276391	802592	293.52	293.79	135.0	NA	90.0	20	110.3
MW-262	Intermediate	Window	279334	800833	293.22	293.50	82.0	87.0	154.4	10	164.6
MW-263	Fluvial	N-C	278945	805817	291.40	291.78	79.0	NA	69.1	10	79.3

								Surface			
					Top of Casing	Ground	Depth to	Casing	Riser	Screen	Total Well
			Northing	Easting	Elevation	Elevation	Top of Clay	Depth	Length	Length	Depth
Well	Aquifer	Area	(ft)	(ft)	(ft, NAVD)	(ft, NAVD)	(ft, bgs)	(ft, bgs)	(ft)	(ft)	(ft, btoc)
MW-264	Upper Claiborne	N-C	278411	804590	303.72	304.00	117.0	NA	104.8	10	115.0
MW-265	Fluvial	N-C	278112	804710	305.15	305.61	95.0	NA	85.8	10	96.0
MW-266	Fluvial	TTA-2	277092	806686	304.68	305.10	87.0	NA	77.1	10	87.3
MW-267	Fluvial	TTA-2	277161	806001	303.84	304.30	90.0	NA	71.9	10	82.1
MW-268	Upper Claiborne	TTA-2	277204	805284	304.59	304.92	120.0	NA	109.5	10	119.7
MW-269	Fluvial	TTA-1N	276369	800127	290.05	290.50	102.0	NA	92.2	10	102.4
MW-270	Fluvial	SE	275483	805042	281.74	282.20	88.0	NA	78.4	10	88.6
MW-271	Fluvial	S-C	276315	803774	294.91	295.50	145.0	NA	134.7	10	144.9
MW-272	Fluvial	Background	275880	804037	293.27	293.70	122.0	NA	112.8	10	123.0
MW-273	Intermediate	Window	279713	800122	284.73	285.00	85.0	91.0	128.1	10	138.3
MW-274	Fluvial	Background	275726	806543	294.30	294.60	100.0	NA	89.3	10	99.5
MW-275	Fluvial	Background	275232	805306	272.31	272.59	89.0	NA	80.5	10	90.7
MW-276	Fluvial	Background	275564	804697	288.68	288.91	97.0	NA	87.5	10	97.7
MW-277	Fluvial	Background	275532	803998	301.67	301.96	113.0	NA	102.3	10	112.5
MW-278	Fluvial	TTA-1N	276294	799814	292.18	292.46	102.0	NA	91.0	10	101.2
MW-279	Fluvial	TTA-1S	275982	800579	299.89	300.17	123.0	NA	112.0	10	122.2
MW-280	Fluvial	TTA-2	277390	806313	306.36	306.57	87.0	NA	76.0	10	86.2
MW-281	Fluvial	N-C	278155	804123	304.56	305.03	101.0	NA	81.7	10	91.9
MW-282	Fluvial	Background	278710	804033	307.81	308.14	87.0	NA	76.0	10	86.2
MW-283	Fluvial	Background	278176	806074	304.34	304.87	88.0	NA	77.0	10	87.2
PMW21-01	Fluvial	TTA-1N	276533	800600	294.76	295.00	110.0	NA	88.4	20	108.4
PMW21-02	Fluvial	TTA-1N	276575	800701	292.98	293.19	108.0	NA	91.3	20	111.3
PMW21-03	Fluvial	TTA-1N	276573	800743	292.11	292.72	109.0	NA	90.3	20	110.3
PMW21-04	Fluvial	TTA-1N	276602	800772	291.87	292.20	109.0	NA	89.0	20	109.0
PMW21-05	Fluvial	TTA-1N	276628	801130	288.53	288.92	-	NA	94.3	20	114.3
PMW85-01	Fluvial	TTA-2	276802	806146	305.08	305.39	-	NA	93.2	10	103.2
PMW85-05	Fluvial	TTA-2	276752	806222	305.12	305.32	103.0	NA	93.2	10	103.2
PMW92-02	Fluvial	TTA-2	276667	806476	304.17	304.35	99.0	NA	94.8	10	104.8
PMW92-03	Fluvial	TTA-2	276679	806439	303.91	304.17	103.0	NA	92.5	10	102.5
PMW101-02A	Fluvial	TTA-1S	276282	801145	292.00	292.29	138.0	NA	117.7	20	137.7
PMW101-02B	Fluvial	TTA-1S	276286	801145	291.98	292.24	-	NA	97.8	20	117.8
PMW101-03A	Fluvial	TTA-1S	276348	801198	291.61	291.99	141.0	NA	119.2	20	139.2
PMW101-03B	Fluvial	TTA-1S	276353	801194	291.55	291.82	-	NA	99.3	20	119.3
PMW101-04A	Fluvial	TTA-1S	276299	801182	291.07	291.43	138.0	NA	117.9	20	137.9
PMW101-04B	Fluvial	TTA-1S	276296	801187	291.47	291.75		NA	98.6	20	118.6
PMW101-06A	Fluvial	TTA-1S	276192	801187	292.13	292.72	139.0	NA	120.0	20	140.0

								Surface			
					Top of Casing	Ground	Depth to	Casing	Riser	Screen	Total Well
			Northing	Easting	Elevation	Elevation	Top of Clay	Depth	Length	Length	Depth
Well	Aquifer	Area	(ft)	(ft)	(ft, NAVD)	(ft, NAVD)	(ft, bgs)	(ft, bgs)	(ft)	(ft)	(ft, btoc)
PMW101-06B	Fluvial	TTA-1S	276195	801184	292.17	292.40	-	NA	99.3	20	119.3
PMW101-07A	Fluvial	TTA-1S	276143	801172	292.20	292.52	138.0	NA	117.9	20	137.9
PMW101-07B	Fluvial	TTA-1S	276142	801177	292.36	292.70	-	NA	98.0	20	118.0
PZ-03	Fluvial	S-C	276379	802941	298.51	298.98	-	NA	108.9	10	118.9

Notes:

1: MW-101 has three screened sections at the following depths (ft, btoc): 89-104, 109-119 and 124-134.

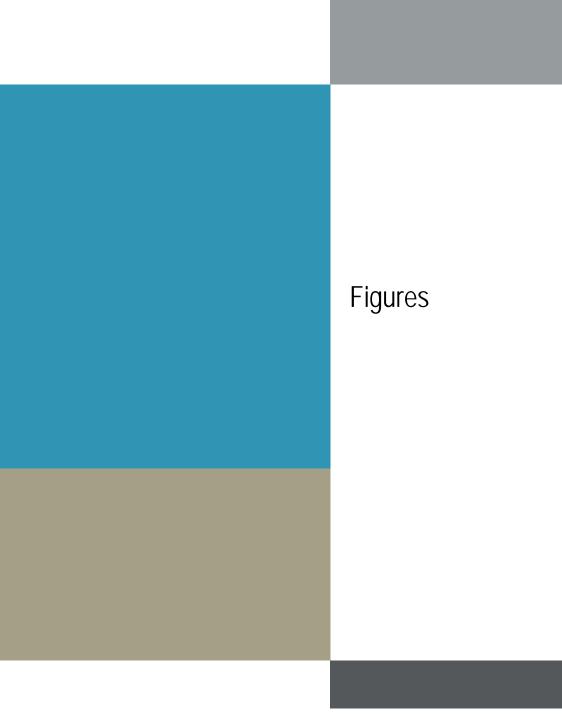
2: MW-107 has two screened sections at the following depths (ft, btoc): 128-143 and 148-158.

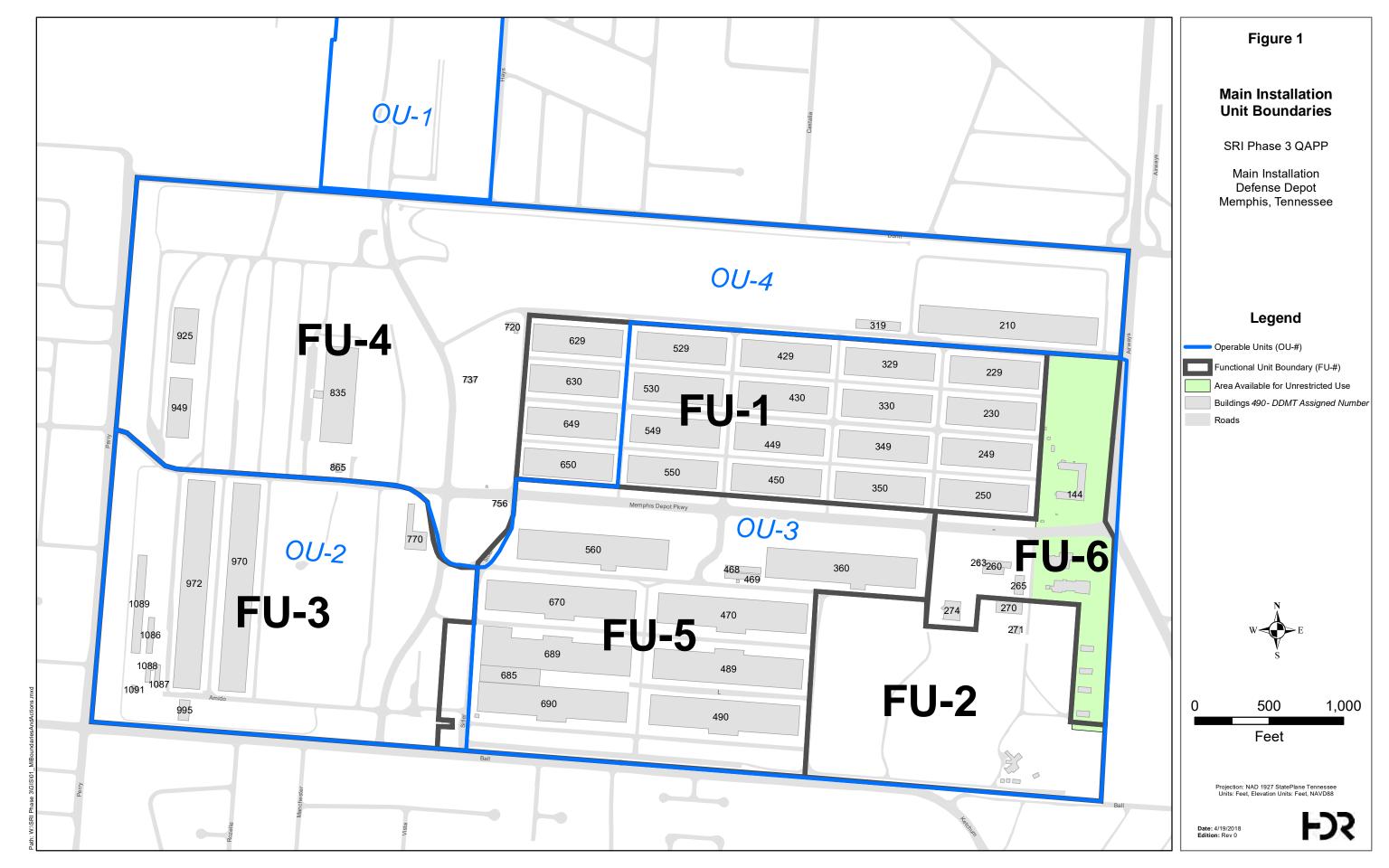
ft: feet

bgs: below ground surface

btoc: below top of casing

NAVD: North American Vertical Datum of 1988







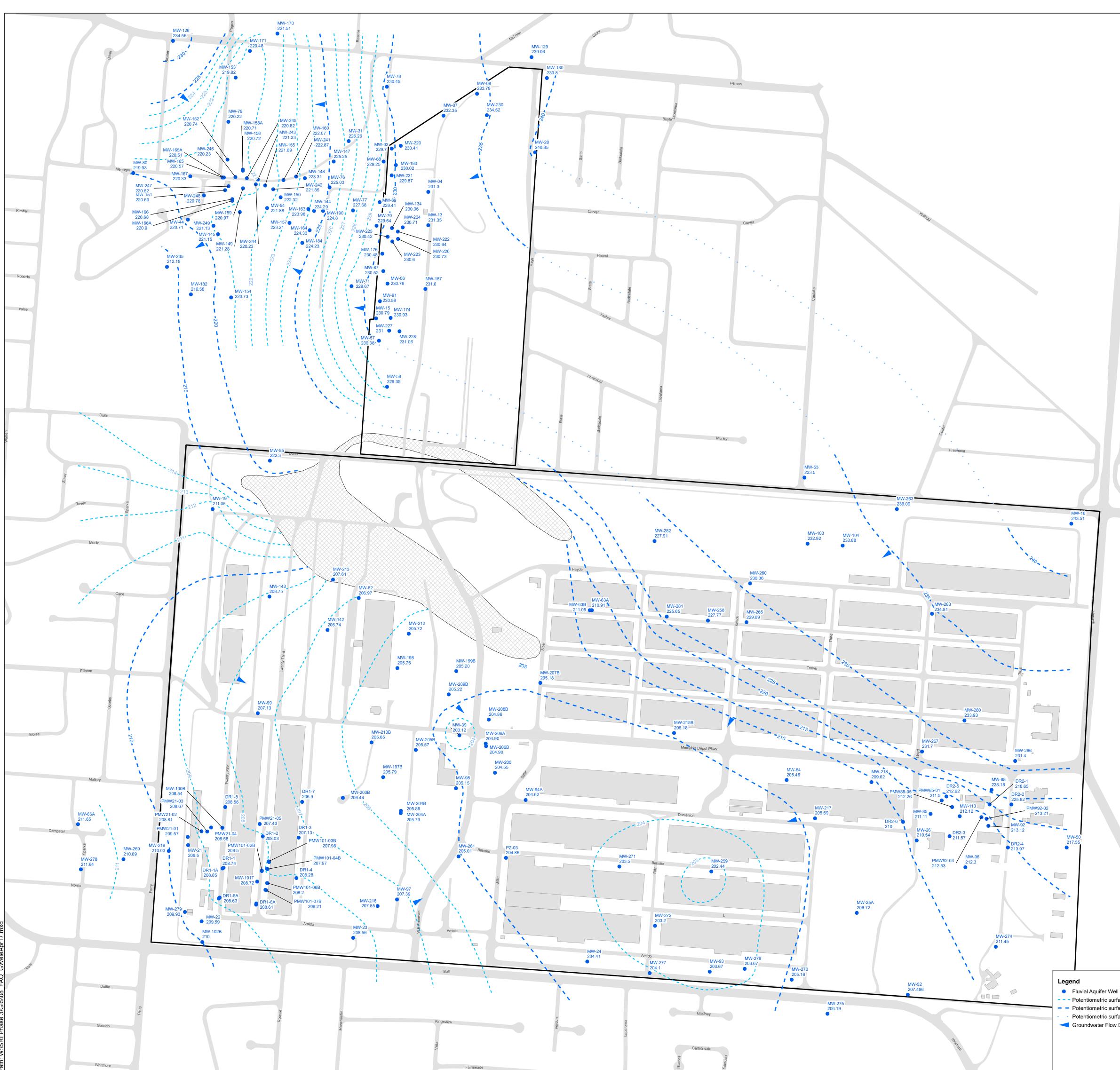












_	Property Boundary	Figure 8		Ň
uifer Well	Roads			W
netric surface of the Fluvial Aquifer 1-ft. contour	Buildings	Fluvial Aquifer		V S
netric surface of the Fluvial Aquifer 5-ft. contour	Clay Elevation Exceeds Groundwater Elevation	Groundwater Elevations	0	250
ater Flow Direction MW-03 229.70	Blue: Fluvial Aquifer, used for groundwater contours (4/18/17)	SRI Phase 3 QAPP		Feet

Dwight

Main Installation Defense Depot Memphis, Tennessee

500 Projection: NAD 1927 StatePlane Tennessee Units: Feet, Elevation Units: Feet , NAVD88 FJS Date: 4/20/2018 Edition: Rev 0



aiborne Well
ontours
ection (Intermedia
l for 18/17)

—— Property Boundary Roads ate) Buildings Clay Elevation Exceeds Groundwater Elevation

Dwight

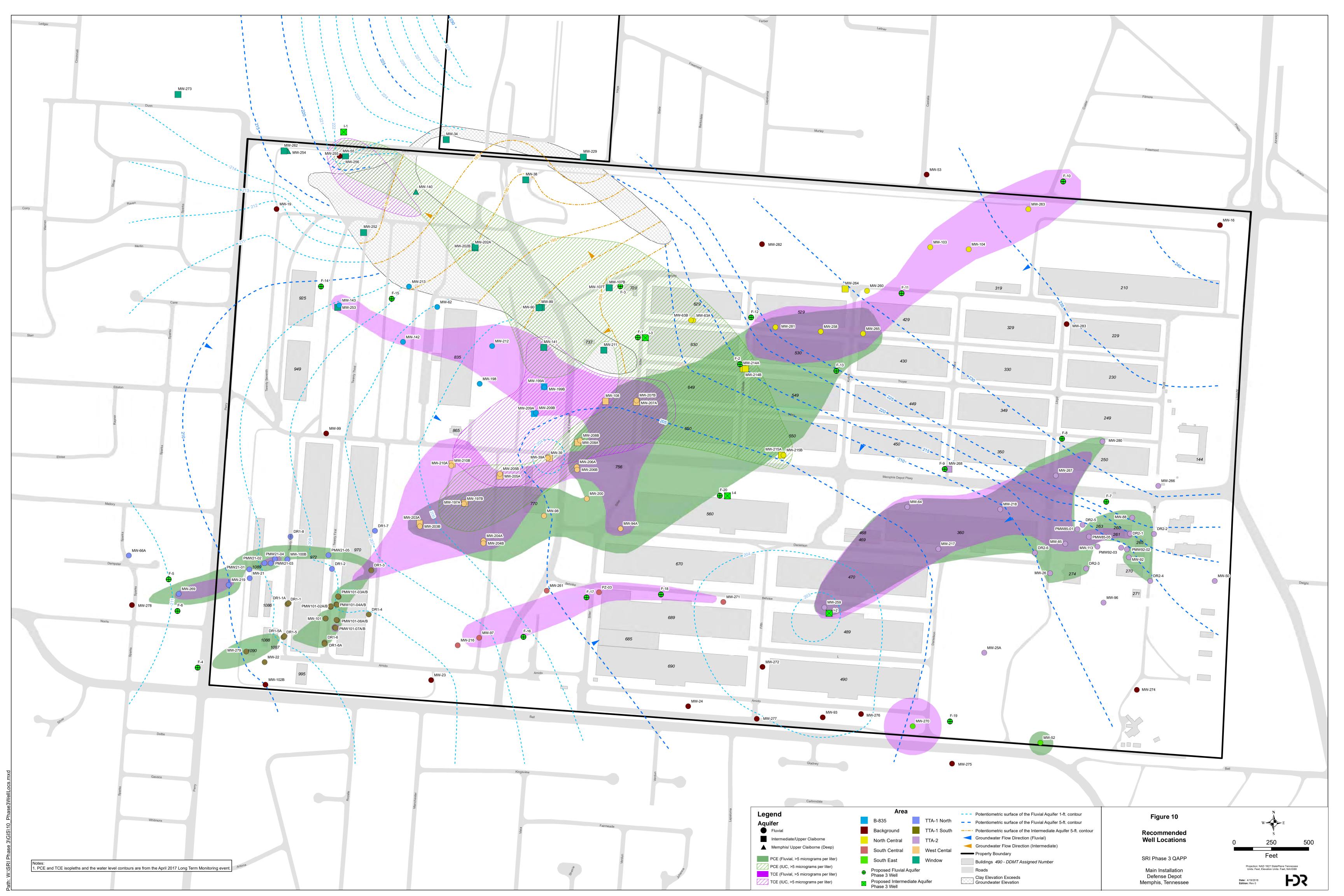
Figure 9

Intermediate Aquifer **Groundwater Elevations**

W E 250 500 0 Feet Projection: NAD 1927 StatePlane Tennessee Units: Feet, Elevation Units: Feet, NAVD88 FJS Date: 4/19/2018 Edition: Rev 0

SRI Phase 3 QAPP

Main Installation Defense Depot Memphis, Tennessee



Appendix A Field Standard Operating Procedures

STANDARD OPERATING PROCEDURE 1 - GENERAL PROCEDURES FOR FIELD PERSONNEL

Lead Organization: <u>Department of the Army (DA)</u> Preparing Organization: <u>HDR</u> SOP Approved by: Field Team Leader: Eric North Project OA Officer: Lynn Lutz

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:
 - o Site Name: Former Defense Depot Memphis Tennessee

- EPA ID (TN4210020570)
- o Project Location: 2241 Truitt Street, Memphis, TN 38114
- The first entry for each field event will list the following information: log books:
 - o Project Name and Number
 - FTL (full name) and initials
 - o 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):
Nater/Ice cooler
Sample cooler

Final Check

- 1. All required equipment/tools received and condition checked
- Yes <u>No</u> Comment:
- 2. Initial equipment calibration completed
- Yes <u>No</u> Comment:
- 3. Vehicles inspected
- Yes <u>No</u> Comment:
- 4. Field locations reviewed
- Yes ____ No ____ Comment:
- 5. Weather forecast checked
- Yes <u>No</u> Comment:
- 6. Staff documents (OSHA, DL) checked
- Yes <u>No</u> Comment:
- 7. Review of project plans confirmed and activities discussed
- Yes <u>No</u> Comment:
- 8. Initial Safety Meeting held and SSHP signed
- Yes <u>No</u> 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 <u>No</u> Comment:
- 2. Equipment/tools decontaminated
- Yes <u>No</u> Comment:
- 3. Rental equipment shipped to supplier
- Yes <u>No</u> Comment:
- 4. Rental vehicles returned
- Yes <u>No</u> Comment:
- 5. DDMT equipment and tools properly stored
- Yes <u>No</u> Comment:
- 6. List damaged equipment
- Yes <u>No</u> Comment:
- 7. Replacement supplies ordered
- Yes <u>No</u> Comment:
- 8. Field locations inspected and trash/debris removed
- Yes <u>No</u> 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 Tennesseelicensed 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 directpush 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: <u>Department of the Army (DA)</u> Preparing Organization: <u>HDR</u> SOP Approved by: Field Team Leader: Justin Bills

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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, premanufactured 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. A reference point will be marked on the well casing for use in groundwater level and well depth measurements. This reference point will marked by the HDR geologist/engineer using a permanent marker for PVC wells, or by notching the top of casing for stainless steel wells. By convention, this marking is usually placed on the north side of the top of casing.

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. The top of casing elevation will be made at the reference point on the north side of the top of casing; the surveyor will not mark the wells in any way. 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 \pm 0.1 for pH, \pm 5% for specific conductance, \pm 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 nonbuoyant 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.
- 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.</p>
- 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.
- 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.</p>
- 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.

Water Level Measurement and Well Assessment Record

Sample Event: _____

	Previous Measurement			
	3/30/2011			
Well I.D	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			

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)	рН	COND (mS/m)	TURB (NTU)	DO (mg/L)	TEMP (Cº)	ORP (mV)	Water Depth (ft, btoc)

Flow Rate_____

General Comments:

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 without 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.

<u>Custody Seals</u>: 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.

EXAMPLE: Sample Plan Detail

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

		,	Parameter Method	VOCs 8260B
			Container	40 mL VOA
			Preservative	HCI to pH<2
				Cool to 4°C
#	Well ID	Sample ID	Additional	No. of
#	Weirid	Sample ID	Auunionai	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

EXAMPLE: Sample Labels for Groundwater Samples

Vorkorder: P55816	
)амріе ID: ТВ-5-ООРМ-9)ate:/ Тіме: Гакеп Ви:	851110268
Preservative: HCL pH <2 09/20/2011 Natrix: Water Tests: VOC_8260	
MICROBAC LABORATORIES INC.	
Vorkorder: P55816	
Jamele ID: TB-5-00PM-9 Jate:/ Time: Taken By:	B320011497
^o reservative: HCL pH <2 09/20/2011 1atrix: Water Fests: VOC_8260	
MICROBAC LABORATORIES INC.	
Vorkorder: P55816	
Sample ID: TB-5-00PM-9 Date:/ Time: Faken By:	8920111480
Preservative: HCL pH <2 09/20/2011 Matrix: Water ests: VOC_8260	
MICROBAC LABORATORIES INC.	

EXAMPLE: Sample Labels for Air Samples

	ALS	
Simi \	Center Drive, Ste. A /alley, CA 93065 1 +1 805 526 7270 (fax)	
Canister .	Sampling Information	
DO NOT adhere a DO NOT over tigh replace the brass ca	ny type of label to the caniste ten the valve and remember t ap.	r. 10
Field	d Readings:	
Pi	Pf	
Initials:	Date:	
Client Name:		-
Sample ID:		-
Analysis:		-
Date / Time:	Sampler's Int.:	
Comments:		_

1.0	
	A
	(ALS)
	And the second second
Pres	ssure / Initials / Date
Psmo:	
Pi1:	
Pf1:	
Pi2:	
Pf2:	
TB:	1
TB Witness:	
	HZA / He / N ₂

0

Attachment 7-4

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

Barcode	Client ID	Teata	Collect Date	Beg. Dopth	End. Depth	Noles
0420111	MW-91-0018-9	NOC_9200-	04/25/2011 18:00	8 yu		
0420112	MW-91-00L9-1	-VOC_8260	04/25/2011 10:00	91421		
0420113	MW-91-GDL8-3	VOO_8280	04725/2041-10:09*	94042		
0420111	04/25/11-TB-1-ODPM-8	VOC_8260	04/25/2011 10:09	111		
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_B260	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-251-00PM-8	VOC 8260	04/25/2011 10:42			
04201111	MW-251-00PM-8	VOC_8260	04/25/2011 10:42			
04201112	MW-251-00PM-8	VOC_8260	04/25/2011 10:42	1.5		
04201113	MW-54-ODPM-8 *	VOC 8260	04/25/2011 11:32			
04201114	MW-54-ODPM-8	VOC 8260	04/25/2011 11:32	- 0		
04201115	MW-54-ODPM-8	VOC 8260	04/25/2011 11:32	1.1.0		
04201116	MW-70-ODPM-8	VOC 8260	04/25/2011 13:23			
04201117	MW-70-ODPM-8	VOC 8280	04/25/2011 13:23			
04201119	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			
04201122	MM 70 OPPMA AND	V/00 8080		KSYA	1	
04201125	MW-76-ODPM-8	VOC 8260	04/25/2011 13:07	-5 7 14		
04201126	MW-76-ODPM-8	VOC_8260	04/25/2011 13:07			
04201127	MW-76-ODPM-8	VOC 8260	04/25/2011 13:07			1.13
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	1.1.1		
04201131	MW-79-ODPM-8	VOC 8260	04/25/2011 11:17	1		
04201132	MW-79-ODPM-8	VOC_8260	04/25/2011 11:17		Received	

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

Samples Collected on: 04/25/2011 by jbsperry

(signed)

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

COC No. A 23953 158 Starlite Drive Marietta, OH 45750 c						CHAIN-OF-CUSTODY RECORD							Fax: 740-373-4835										
Company Name:																			Τ				Program
Project Contact: Contact Phone #:																							
Turn Around Requirements: Location:							VERS																DOD
Project ID:							CONTAINERS														SE)		AFCEE
Sampler (print): Signature:							R OF CO														(LAB US		ADDITIONAL
Sample I.D. No.	Comp	Grab	Date	Tim	e	Matrix*	NUMBER OF	Hold													TOTAL # (LAB USE)		REQUIREMENTS
	-			_			+			-	-			-	-		-	+	+	+		+	
	-						-			-	-				-		-	-	+	-		-	
-																			+			1	
			-																-	-			
							+			-	-		-	-	-	++	+	+	+	+	-	+	
			0																				
	-			1 - 1			-			-	-		_	-	-	+	-	-	+	+		+	
						-																	
						E.c.		-						_				_	-	-			
					-		+		\vdash	-			-	-			+	+	+	-		t	
	-				_		-			-	-			-	-		-	-	+	+			
Relinquished by: Date Time (Signature)			Time		eceived by: ignature) Relinquished by: (Signature)						by:		Date Tir			Tim	e Received by: (Signature)						
Relinquished by: (Signature)		Date	Time	Rece (Sign	eived for Labora nature)	tory by	bry by: Date					Time			Remarks:								

EXAMPLE: ALS Chain-of-Custody Form

	Air - Chain of Custody Record & Analytical Service Request Page 2655 Park Center Drive, Suite A Simi Valley, California 93065											
(ALS)	Phone (805)			Requested Turn	around Time in I	circle	ALS Project No.					
	Fax (805) 52			1 Day (100%) 2 D	ay (75%) 3 Day (-Standard	-					
						ALS Contac	st:					
Company Name & Address (Report	ing Information)		Project Name								
						Analysis	s Method					
				Project Number								
Project Manager				P.O. # / Billing Infor	mation	-						
				1.0. #7 Dialing into	mation			Comments				
Phone	Fax			1				e.g. Actual				
								Preservative or				
Email Address for Result Reporting	•			Sampler (Print & Sign)	I	1		specific instructions				
Client Sample ID	Laboratory ID Number	Date Collected	Time Collected	Canister ID (Bar code # - AC, SC, etc.)	Flow Controller ID (Bar code # - FC #)	Canister Start Pressure "Hg	Canister End Pressure "Hg/psig	Sample Volume				
				Í Í								
								<u> </u>				
-	Report Tier Levels - please select										Project	
Tier I - Results (Default if not specified) Tier II (Results + QC Summaries)				mmaries) Surcharge	EDD required Yo Type:	Custody Sea BROKEN		Requirements (MRLs, QAPP)				
Relinquished by: (Signature)			Date:	Time:	Received by: (Signa		Date:	Time:				
Relinquished by: (Signature)			Date:	Time:	Received by: (Signa	ture)		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

 $\hfill\square$ 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.

<u>Sampling Equipment</u>: 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. <u>Label Sample Container</u> Label the sample container as outlined in SOP 7 Sample Control and Documentation.
- b. <u>Collect Sample</u> 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. <u>Custody</u>, <u>Handling and Shipping</u> - Complete the procedures as outlined in SOP 7 – Sample Control and Documentation and SOP 8 - Sample Packing and Shipping</u>.

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

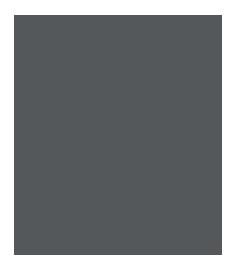
Work will be performed in accordance with the Quality Assurance Project Plan (QAPP), the specific work plan, and applicable SOPs.

8 References

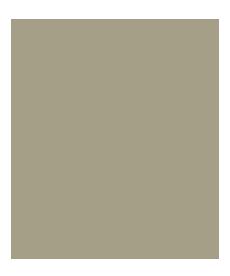
HDR, 2017a. 2017 Uniform Federal Policy-Quality Assurance Project Plan, Environmental Restoration Support at Former Defense Depot Memphis, Tennessee, Revision 0. Prepared for the U.S. Army Corps of Engineers, Mobile District. May 2017.

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





L1 - Analysis of Volatile Organic Analytes by Method 8260





MICROBAC SOP #:	MSV01
PAGE:	1 of 51
REVISION:	25

STANDARD OPERATING PROCEDURES ANALYSIS OF VOLATILE ORGANIC ANALYTES BY METHOD 8260

Issue/Implementation Date: 10 January 2017

Last Review Date: 15 September 2017

Microbac Laboratories, Inc. Ohio Valley Division 158 Starlite Drive Marietta, Ohio 45750

Approved By:

Anthony D. Canter, Volatile Lab Supervisor

Maren M. Beery, Quality Assurance Officer

IIM

Leslie S. Bucina, Managing Director

9-13-17

Date

09115/2017

Date

Date



SECTION

MICROBAC SOP #:	MSV01
PAGE:	2 of 51
REVISION:	25

TABLE OF CONTENTS

<u>PAGE</u>

1.0	Scope and Application	3
2.0	Safety Precautions	4
3.0	Sample Preservation and Storage	5
4.0	Method Performance	6
5.0	Interferences and Corrective Action	6
6.0	Equipment and Supplies	7
7.0	Standards and Reagents	8
8.0	Diagram or Table to Outline Procedures	16
9.0	Sample Preparation	17
10.0	Calibration Procedures	17
11.0	Analytical Procedures	20
12.0	Details of Calculations	24
13.0	Quality Control Requirements	31
14.0	Data Review and Reporting Requirements	35
15.0	Preventive Maintenance	36
16.0	Waste Management and Pollution Control	36
17.0	References	37
	Appendices	38
	Tables	41
	Figures	50



MICROBAC SOP #:	MSV01
PAGE:	3 of 51
REVISION:	25

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). 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.
- **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.



MICROBAC SOP #:	MSV01
PAGE:	4 of 51
REVISION:	25

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 side shields.
- **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



MICROBAC SOP #:	MSV01
PAGE:	5 of 51
REVISION:	25

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 HCI (pH < 2) must be analyzed within 14 days of sample collection. Unpreserved water samples (pH \ge 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 0 to 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 0 to 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 0 to 6°C. Samples exceeding the upper temperature limit are to be flagged CT1.



MICROBAC SOP #:	MSV01
PAGE:	6 of 51
REVISION:	25

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** Other specific QA objectives may be found in the appropriate Statement-of-Work or QAPP.
- **4.7** All new analysts are certified in the methodology by performing a successful initial demonstration of capability (DOC) using spiked control samples and assessing the resultant precision and accuracy data. Thereafter an on-going DOC must be performed annually to retain certification.

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.



MICROBAC SOP #:	MSV01
PAGE:	7 of 51
REVISION:	25

- **5.2** Samples preserved with HCl or sodium bisulfate may result in the loss of 2chloroethylvinylether 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



MICROBAC SOP #:	MSV01
PAGE:	8 of 51
REVISION:	25

- 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.



MICROBAC SOP #:	MSV01
PAGE:	9 of 51
REVISION:	25

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



MICROBAC SOP #:	MSV01
PAGE:	10 of 51
REVISION:	25

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 than 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)
	502.2 Mega Mix with MTBE	2000	500		
VOA Mix 1	502.2 Calibration Mix #1	2000	500	5	200
	Freon 113	2000	500		
VOA Mix 2	Custom Concentrated Ketones #2	2000	500	5	200
	2-Chlorethylvinyl ether (2-CVE)	5000 200			
	1,3-Butadiene	2000	500		
VOA Mix 3	Custom VOC Mix 3	2000	500	5	200
	1-Bromopropane	10000	100		
VOA Mix 4	Acrolein- Acrylonitrile Mix	2000	500	10	100-400
	MA Oxygentates Standard	2000-4000	1000	10	100-400
Vinyl Acetate	Vinyl Acetate	2000	500	5	200

VMS Column

	PRIMARY STANDARD	CONCENTRATION (ugmL)	VOLUME (uL)	FINAL VOLUME (mL methanol)	FINAL CONCENTRATION (ugmL)
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MICROBAC SOP #:	MSV01
PAGE:	11 of 51
REVISION:	25

n					
	502.2 Mega Mix with MTBE	2000	500		
VOA Mix 1	Custom Standard	2000	500	5	200
	(Gases)				
	Custom Concentrated Ketones #2	2000	500	5	200
VOA Mix 2	2- Chlorethylvinyl ether (2-CVE)	5000	200	5	200
	1,3-Butadiene	2000	500		
VMS CCV STD	Acrolein- Acrylonitrile Mix	2000	500	5	200-400
	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
Gases & Auus	VOC Adds Mix	200	1000	10	20
Mega	VOC Mega Mix 1- Bromopropane Intermediate*	<u> </u>	<u> 1000 </u> 20	<u> </u>	<u> 20 </u> 20
1,3-Butadiene	Vinyl acetate	2000	100	10	20
and Vinyl Acetate Mix	1,3-Butadiene	200	1000	10	20
Oxygenates LCS Mix	MA Oxygenates Acrolein	2000-4000 5000	500 200	<u> </u>	<u>100-200</u> 100

1-Bromopropane Intermediate solution is prepared by diluting 0.1g of 99%

1-Bromopropane into 10mL of methanol.



MICROBAC SOP #:	MSV01
PAGE:	12 of 51
REVISION:	25

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			
0200 LCS WIX	Custom Gases	250	800	10	20			
VMS ADDS Mix	Custom VMS LCS ADDS	200-2000	500	5	20-200			
1,3-Butadiene	Vinyl acetate	2000	100	10	20			
and Vinyl Acetate Mix	1,3-Butadiene	200	1000	10	20			
Acrolein Alt	Acrolein	5000	100	5	100			

7.8 50 ug/mL BFB intermediate solution preparation:

PRIMARY STANDARD	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL METHANOL)	FINAL CONCENTRATION (ug/mL)
BFB Intermediate Solution	2000	125	5	50

- 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	WORKING STANDARDS CONCENTRATIONS (ug/								
STANDARD, CONCENTRATION	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	N/A
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



MICROBAC SOP #:	MSV01
PAGE:	13 of 51
REVISION:	25

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			WORKI	NG STANI	DARDS C	ONCEN	FRATION	S (ug/L)		
STANDARD, CONCENTRATION	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	N/A
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



MICROBAC SOP #:	MSV01
PAGE:	14 of 51
REVISION:	25

- 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:

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

Water Analyses

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



MICROBAC SOP #:	MSV01
PAGE:	15 of 51
REVISION:	25

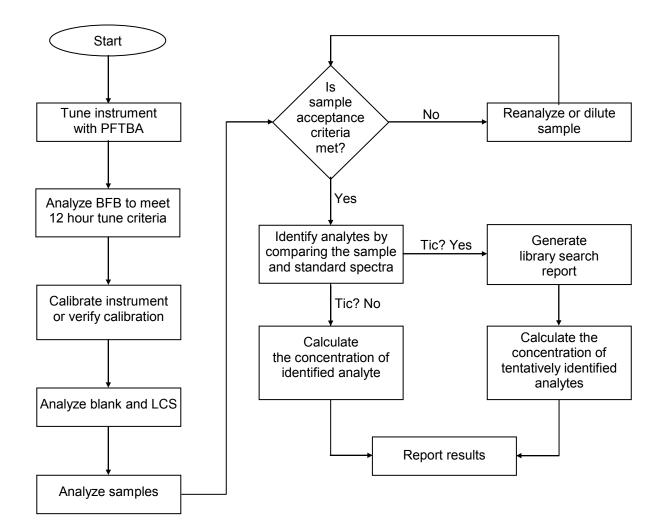
Oxygenates (50-100 ug/mL)	5	5	5	100-200	100-200
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- **7.11** 50ng BFB: Prepared by diluting 10 uL of BFB intermediate solution in 50 mL of DI water then purging 5 mL (else 1 uL of BFB intermediate solution is injected into the GC injection port).
- 7.12 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.



MICROBAC SOP #:	MSV01
PAGE:	16 of 51
REVISION:	25

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES





MICROBAC SOP #:	MSV01
PAGE:	17 of 51
REVISION:	25

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.

The accuracy of the calibration curve is assessed by calculating the percent error (12.7) for each level of the curve. A maximum percent error of 30% (residual test) should be achieved for each level regardless of the calibration model used.

10.6 Five analytes designated as system performance check compounds (SPCC) must meet minimum average response factor criteria (\overline{RRF}) as follows:

COMPOUND	
chloromethane	0.10
1,1-dichloroethane	0.10
bromoform	0.10
chlorobenzene	0.30
1,1,2,2-tetrachloroethane	0.30



MICROBAC SOP #:	MSV01
PAGE:	18 of 51
REVISION:	25

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 \leq 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 \leq 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) $r^2 \ge 0.99$

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 \geq 0.99.

Quadratic Calibration with COD $(r^2) \ge 0.99$



MICROBAC SOP #:	MSV01
PAGE:	19 of 51
REVISION:	25

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 \geq 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

vinyl acetate vinyl chloride 2-chloroethylvinyl ether naphthalene acetone

Additional 8260 Compounds

t-butyl alcohol	paraldehyde
1,4-dioxane	1-bromopropane
propionitrile	isobutyl alcohol
tetrahydrofuran	1-butanol
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 \leq 20 % difference when using average response factor or \leq 20 % drift when using regression fit. Non-CCC's must be \leq 20%difference/drift. Some compounds are historically poor performing analytes and



MICROBAC SOP #:	MSV01
PAGE:	20 of 51
REVISION:	25

may not meet \leq 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.



MICROBAC SOP #:	MSV01
PAGE:	21 of 51
REVISION:	25

- **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: $5.00 \text{ g} (\pm 0.1 \text{ g})$ 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: $5.00 \text{ g} (\pm 0.1 \text{ g})$ 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.



MICROBAC SOP #:	MSV01
PAGE:	22 of 51
REVISION:	25

- **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



MICROBAC SOP #:	MSV01
PAGE:	23 of 51
REVISION:	25

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



MICROBAC SOP #:	MSV01
PAGE:	24 of 51
REVISION:	25

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 \pm 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.

lons present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of coeluting compounds.

lons 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.



MICROBAC SOP #:	MSV01
PAGE:	25 of 51
REVISION:	25

- C_{is} = Concentration of the specific internal standard.
- C_x = Concentration of the surrogate or compound being measured.
- **12.2** Average RRF (\overline{RRF}) :

$$\overline{RRF} = \frac{\sum_{n=1}^{n} RRF}{n}$$

12.3 Standard deviation (s):

$$\mathbf{s} = \sqrt{\frac{\sum \left(x - \overline{x}\right)^2}{n - 1}}$$

12.4 Percent relative standard deviation (%RSD):

$$\% RSD = \left(\frac{s}{\overline{x}}\right) 100$$

where:

$$\overline{x} = \overline{RRF}$$
: $\overline{RRF} = \frac{\sum_{n=1}^{n} RRF}{n}$
s = standard deviation(s): $s = \sqrt{\frac{\sum (x - \overline{x})^2}{n - 1}}$

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

Document Control # 288



MICROBAC SOP #:	MSV01
PAGE:	26 of 51
REVISION:	25

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):

$$\frac{\sum XY - \sum X\sum Y / n}{\sqrt{\left(\sum X^2 - \left(\sum X\right)^2 / n\right)\left(\sum Y^2 - \left(\sum Y\right)^2 / n\right)}}$$

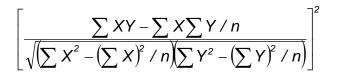
where:



MICROBAC SOP #:	MSV01
PAGE:	27 of 51
REVISION:	25

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):



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:



MICROBAC SOP #:	MSV01
PAGE:	28 of 51
REVISION:	25

$$ug/kg = \frac{(A_x)(I_s)(V_i)(DF)}{(A_{is})(RRF)(V_o)(V_i)(D)}$$

where :

 A_x , I_s , A_{is} , \overline{RRF} , V_{o_i} DF= same as for water W_s = weight of sample extracted in grams

$$V_t$$
 = volume of total extract (mL) = $V_m + \left[\left(W_s \right) \left(\frac{100 - D}{100} \right) \right]$

 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:

 $y = \text{Response ratio} = \text{Response}(x)/\text{Response}(\text{istd}) = R_x/R_{\text{istd}}$ $x = \text{Concentration ratio} = \text{Conc}(x)/\text{Conc}(\text{istd}) = C_x/C_{\text{istd}}$ 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:



MICROBAC SOP #:	MSV01
PAGE:	29 of 51
REVISION:	25

$$x=\frac{b\pm\sqrt{(b^2-4a(c-y))}}{2a}$$

where:

 $y = \text{Response ratio} = \text{Response}(x)/\text{Response}(\text{istd}) = R_x/R_{\text{istd}}$ $x = \text{Concentration ratio} = \text{Conc}(x)/\text{Conc}(\text{istd}) = C_x/C_{\text{istd}}$ 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):



MICROBAC SOP #:	MSV01
PAGE:	30 of 51
REVISION:	25

$$ug/L = \frac{(A_x)(I_s)(DF)}{(A_{is})(\overline{RRF})(V_o)}$$

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:

$$ug/Kg = \frac{(A_x)(I_s)(V_t)(DF)}{(A_{is})(RRF)(V_o)(W_s)(D)}$$

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)



MICROBAC SOP #:	MSV01
PAGE:	31 of 51
REVISION:	25

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



MICROBAC SOP #:	MSV01
PAGE:	32 of 51
REVISION:	25

- **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



MICROBAC SOP #:	MSV01
PAGE:	33 of 51
REVISION:	25

- 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 1/2 the reporting limit/lower limit of guantitation 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 noncompliant 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



MICROBAC SOP #:	MSV01
PAGE:	34 of 51
REVISION:	25

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 reextraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides



MICROBAC SOP #:	MSV01
PAGE:	35 of 51
REVISION:	25

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.



MICROBAC SOP #:	MSV01
PAGE:	36 of 51
REVISION:	25

- 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



MICROBAC SOP #:	MSV01
PAGE:	37 of 51
REVISION:	25

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** U.S. EPA, 40 CFR, Part 136, October 26, 1984
- **17.3** Microbac SOP PAT01 "Methods 5030 and 5035 Purge and Trap for Volatile ` Organics"
- **17.4** Microbac SOP 45 "Method Validation Procedures"
- 17.5 Microbac SOP 41 "Manual Integration of Chromatographic Peaks"
- **17.6** Microbac SOP 33 "Laboratory Waste Management"
- **17.7** Microbac SOP LQAP "Laboratory Quality Assurance Plan"
- **17.8** Microbac SOP GP-TEMP-SSU "Temperature control Systems for Sample Storage Units"



MICROBAC SOP #:	MSV01
PAGE:	38 of 51
REVISION:	25

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	90 57	43	57
diisopropyl ether	45	43, 87	45
vinyl acetate	43	43, 87	43
1,1-dichloroethane	63		63
		65, 83	
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



MICROBAC SOP #:	MSV01
PAGE:	39 of 51
REVISION:	25

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
	98	91	90
toluene	69		69
ethyl methacrylate		41, 99, 86, 114	
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



MICROBAC SOP #:	MSV01
PAGE:	40 of 51
REVISION:	25

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



MICROBAC SOP #:	MSV01
PAGE:	41 of 51
REVISION:	25

Table 1Method 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
2,2-dichlorophopane 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	107-02-0
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
Chioromethane	14-01-3

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



MICROBAC SOP #:	MSV01
PAGE:	42 of 51
REVISION:	25

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	80-123	20	0.25	5	40	200-300
1,2-dichloropropane	78-87-5	80-124	20	0.23	5	20	200-300
1,3,5-trimethylbenzene	108-67-8	80-120	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	0.25	5	20	200-300
1.4-dichlorobenzene	106-46-7	80-120	20	0.2	5	20	200-300
1.4-dioxane	123-91-1	20-160	20	50	100	20	200-300
,	123-91-1	50-150	20	0.5	100	200	200-300
1-bromopropane	71-36-3	50-150	20	0.5 50		20	50-800
1-butanol			-		100		
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



MICROBAC SOP #:	MSV01
PAGE:	43 of 51
REVISION:	25

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
		70-130	20	1.0	10	20	200-300
ethyl methacrylate ethyl-tert-butyl ether	97-63-2 637-92-3	70-130	20	5	10	100	200-300
, , ,	87-68-3	70-130	20	0.25	5	20	200-300
hexachlorobutadiene					-		
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 m+p-xylene **	98-82-8 179601- 23-1	80-122 80-122	20 20	0.25 0.5	5	20 40	200-300 5-800
methacrylonitrile	126-98-7	70-130	20	2.5	5	100	200-300
Methyl acetate	79-20-9	50-190	20	2.5	10	20	200-300
Methyl cyclohexane	108-87-2	80-130	20	1	10	20	200-300
	80-62-6	70-130	20			100	200-300
methyl methacrylate	80-62-6 75-09-2	80-123	20	2.5 0.25	5 5	20	200-300
methylene chloride			20				200-300
methyl-tert-butyl ether	1634-04-4 91-20-3	75-130 59-130		0.5	5	20	
naphthalene			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



MICROBAC SOP #:	MSV01
PAGE:	44 of 51
REVISION:	25

Table 2 (co	ntinued)
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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



MICROBAC SOP #:	MSV01
PAGE:	45 of 51
REVISION:	25

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



MICROBAC SOP #:	MSV01
PAGE:	46 of 51
REVISION:	25

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



MICROBAC SOP #:	MSV01
PAGE:	47 of 51
REVISION:	25

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 CHROMATOG	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					

* Suggested parameters: Adjustments may be made to improve efficiency.



MICROBAC SOP #:	MSV01
PAGE:	48 of 51
REVISION:	25

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

SURROGATE COMPOUND	8260 WATER *	8260 SOIL *	8260 OIL
dibromofluoromethane	86-118	80-120	52-122
Toluene-d ₈	88-110	81-117	35-127
4-bromofluorobenzene	86-115	74-121	26-158
1,2-dichloroethane-d ₄	80-120	80-120	43-128

 Table 6

 Surrogate Standards Recovery Limits

*Denotes one outlier permitted given % R > 10%. Reanalysis required if two or more recovery results are outside acceptance limits.



MICROBAC SOP #:	MSV01
PAGE:	49 of 51
REVISION:	25

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.	$ \leq 30\% \text{ RSD for CCC compounds,} \\ < 15\% \text{ RSD for all target compounds,} \\ \text{if >15\% RSD, then linear regression,} \\ \text{provided } r \geq 0.995 \text{ and } \% \text{ error } \leq \\ 30\%, \text{then quadratic regression,} \\ \text{provided } r^2 \geq 0.990 \\ \text{SPCC minimum } \overline{\text{RRF}} \\ \end{cases} $	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	\leq 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.



MICROBAC SOP #:	MSV01
PAGE:	50 of 51
REVISION:	25

Figure 1

Checklist ID: 6687

Microbac Laboratories Inc.

Data Checklist

Date:	
Analyst:	
Analyst: NA	
Mothod:	
Instrument:	
Curve Workgroup: NA	
Runlog ID:	
nalytical Workgroups;	

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Primary Reviewer.

Secondary Reviewer:

CHECKLIST1 - Modified 03/05/2008 Generated: APR-03-2008 14:06:22





MICROBAC SOP #:	MSV01
PAGE:	51 of 51
REVISION:	25

Figure 2 VOA Preparation/Preservation and Extraction Log

Microbac Laboratories Inc. VOA Preparation/Preservation/Extraction Log

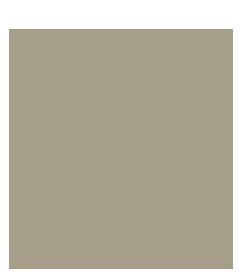
	Reagen	t ID:RGT1000	1								
SAMPLE #	Fraction	Collected	Preserved	PCT-S	Tare Wt	Total WE	Sample Wt	Water	MeOR	Vt	Comments
L08020355-08	A	04/20/08 11:12	06/05/08 09:50	79.86			30.12	5		5	5
L08020355-09	Α.	04/20/08 11:25	06/05/08 09:50	77.08			30.12	5		5	5
L08020355-09	в	04/20/08 11:25	06/05/08 09:51	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	1		25.08		10	15.748916	5
c	omments:	1 - improperl 2 = preserved	y sealed cap out of hold		elferves preserve	ced d with Na		6 = pre	eserve	d by freezi d in field foo But	-

Mic	roba

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L2 - Sample Receiving and Login







MICROBAC SOP #:	LOGIN01
PAGE:	1 of 30
REVISION:	19

STANDARD OPERATING PROCEDURE SAMPLE RECEIVING AND LOGIN

Issue/Implementation Date: 18 February 2015

Last Review Date: 15 September 2017

Microbac Laboratories, Inc. **Ohio Valley Division** 158 Starlite Drive Marietta, Ohio 45750

Approved By:

Chad E. Barnes, Support Services Supervisor

Maren M. Beery, Quality Assurance Officer

Leslie S. Bucina, Managing Director

09-01-17

Date

late Date



SECTION

MICROBAC SOP #:	LOGIN01
PAGE:	2 of 30
REVISION:	19

TABLE OF CONTENTS

<u>PAGE</u>

1.0	Scope and Application	3
2.0	Safety Precautions	3
3.0	Equipment and Supplies	4
4.0	Reagents	5
5.0	Custom Sample Kits	6
6.0	Sample Handling and Custody Procedures	.10
7.0	Overview of Logging Procedures	.15
8.0	Detailed Logging Procedures	.17
9.0	Calibration and Quality Control	.18
10.0	References	.21
	Tables	.22
	Figures	.27



MICROBAC SOP #:	LOGIN01
PAGE:	3 of 30
REVISION:	19

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 Internal Chain of Custody ICOC Infrared Temperature Gun IR gun LIMS Laboratory Information Management System LQAP Laboratory Quality Assurance Plan Safety data sheet SDS Standard Operating Procedure SOP VOA Volatile Organic Analysis Volatile Organic Compounds VOC

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



MICROBAC SOP #:	LOGIN01
PAGE:	4 of 30
REVISION:	19

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, "Laboratory Waste Management".
- **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)



MICROBAC SOP #:	LOGIN01
PAGE:	5 of 30
REVISION:	19

4.0 REAGENTS

- **4.1** 20% Nitric Acid (HNO₃) 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 HNO₃ is used as a preservative, a red label with HNO₃ 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 (H₂SO₄) Baker Instra-analyzed or equivalent; prepared when needed in the conventional lab. 500 mL of concentrated H₂SO₄ 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 H₂SO₄ is used as a preservative, a yellow label with H₂SO₄ 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.



MICROBAC SOP #:	LOGIN01
PAGE:	6 of 30
REVISION:	19

- **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.



MICROBAC SOP #:	LOGIN01
PAGE:	7 of 30
REVISION:	19

- 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% HNO₃ 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 (SO₃) 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.



MICROBAC SOP #:	LOGIN01
PAGE:	8 of 30
REVISION:	19

- 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 H₂SO₄. Other requirements for TOC are as follows: TOC-4 used 4x250 mL plastic with 1:1 H₂SO₄, TOC -14 uses 1x250 mL glass with 1:1 H₂SO₄, TOC-44 uses 4x250 mL glass with 1:1 H₂SO₄.
- 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 ₂ SO ₄ :	40 mL 250 mL 500 mL 1000 mL	1⁄₂ mL = pH < 2 1 mL = pH < 2 2 mL = pH < 2 4 mL = pH < 2
HNO3:	250 mL 500 mL 1000 mL	3 mL = pH < 2 5 mL = pH < 2 10 mL = pH < 2
HCL:	40 mL 1000 mL	3 drops = pH < 2 5 mL = pH < 2
NAOH:	250 mL	3.5 mL = pH > <mark>1</mark> 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



MICROBAC SOP #:	LOGIN01
PAGE:	9 of 30
REVISION:	19

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".



MICROBAC SOP #:	LOGIN01
PAGE:	10 of 30
REVISION:	19

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



MICROBAC SOP #:	LOGIN01
PAGE:	11 of 30
REVISION:	19

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 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



MICROBAC SOP #:	LOGIN01
PAGE:	12 of 30
REVISION:	19

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.



MICROBAC SOP #:	LOGIN01
PAGE:	13 of 30
REVISION:	19

- *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.



MICROBAC SOP #:	LOGIN01
PAGE:	14 of 30
REVISION:	19

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

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



MICROBAC SOP #:	LOGIN01
PAGE:	15 of 30
REVISION:	19

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.

6.10 After Hours Receipt from Clients

When a client cannot drop off sample(s) to the lab during normal business hours, we provide a locked drop box at the shipping/receiving area. The client can contact their Account Manager to notify them that they need to use the drop box; the box will be left unlocked for the client to drop off their cooler. Once they drop off the cooler, they will lock the box. If needed, the cooler will be received later that night or first thing the next morning by the login personnel. If the project is an ongoing event, the client will be provided a key to unlock and lock, on an as needed basis during their project.

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



MICROBAC SOP #:	LOGIN01
PAGE:	16 of 30
REVISION:	19

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.



MICROBAC SOP #:	LOGIN01
PAGE:	17 of 30
REVISION:	19

8.0 DETAILED LOGGING PROCEDURE

- **8.1** Log into the LIMS.
- **8.2** Go to Sample Management; Login; Login ID enter the correct work order (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.



MICROBAC SOP #:	LOGIN01
PAGE:	18 of 30
REVISION:	19

- **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.



MICROBAC SOP #:	LOGIN01
PAGE:	19 of 30
REVISION:	19

- 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 6°C 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.
- 4. When bottle temperature is out for WV samples, NOTE EACH BOTTLE on the form and label each bottle that is out with "Bottle Temp Out" sticker.

The temperature discrepancy still needs noted in the receipt and inspection forms.



MICROBAC SOP #:	LOGIN01
PAGE:	20 of 30
REVISION:	19

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.



MICROBAC SOP #:	LOGIN01
PAGE:	21 of 30
REVISION:	19

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"



MICROBAC SOP #:	LOGIN01
PAGE:	22 of 30
REVISION:	19

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, $\leq 6^{\circ}$ 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, $\leq 6^{\circ}$ C, H ₂ SO 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 ₂ S ₂ O ₃	8 Hours
Coliform, Total	100	Sterile P, G	Cool, < 10° C, Na ₂ S ₂ O 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, $\leq 6^{\circ}$ 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 ₂ S ₂ O ₃	8 Hours
Nitrogen, Ammonia (Distilled/Non-Distilled)	100	P, G	Cool, $\leq 6^{\circ}$ C, H ₂ SO 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, $\leq 6^{\circ}$ C, H ₂ SO ₄ , pH<2	28 Days
Nitrogen, Organic (calc)	100	P, G	Cool, $\leq 6^{\circ}$ C, H ₂ SO ₄ , pH<2	28 Days
Oil and Grease	1000	G	Cool, ≤ 6° C, ĤCl, pH<2	28 Days

P = Polyethylene (preferred when acceptable)
 G = Borosilicate glass with Teflon lined cap
 For more current list of method and preservations, see LIMS tables, Products/Containers



LOGIN01
23 of 30
19

Table1 (continued) Sample Containers, Preservation and Hold Times **CONVENTIONALS - WATER**

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Phenolics, Total	100	Amber, G	Cool, $\leq 6^{\circ}$ C, H ₂ SO ₄ , pH< 2	28 Days
Phosphorus, Total	50	P, G	Cool, $\leq 6^{\circ}$ C, H ₂ SO 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

P = Polyethylene (preferred when acceptable)
 G = Borosilicate glass with Teflon lined cap
 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, $\leq 6^{\circ}$ C, HCl, pH< 2	14 Days
Volatile Aromatics	40 mL	G, Septa Caps	Cool, $\leq 6^{\circ}$ C, HCl, pH< 2	14 Days
Volatile Organics (VOA)	40 mL	G, Septa Caps	Cool, $\leq 6^{\circ}$ 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, $\leq 6^{\circ}$ C, Na ₂ S ₂ O ₃	7 Days

*Provided upon client request when samples contain chlorine.



LOGIN01
24 of 30
19

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	HNO₃, pH< 2	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



MICROBAC SOP #:	LOGIN01
PAGE:	25 of 30
REVISION:	19

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	



MICROBAC SOP #:	LOGIN01
PAGE:	26 of 30
REVISION:	19

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		



MICROBAC SOP #:	LOGIN01
PAGE:	27 of 30
REVISION:	19

Figure 1

Mi	crobac			Lab Project #: 2284.033 Project Name: Lab Contact: Stephanie Mossburg			
	epancies of the shipment con	ditions and the ins	pection records for the	and Inspection samples received and reporte ur receipt policies, except as n			
here were no disi	crepancies. Discrepan	CY .		Resolution			
olers							
Cooler #	Temperature Gun	Temperature	coc#	Airbill #	Temp Required?		
00110685	н	5,0			×		
spection Check	list		Color II.		A11.00		
t/	-		Jestion		Result		
1			ig coolers sealed?		NA		
Z			dy seals intact?		NA		
3			eratures in range of 0-1	69	Yes		
4	310437 244		ce present?		Yes		
5		the second s	ation complete/signed		Yes		
5	Were sample containers intact and match COC?			Yes			
7	Were sample labels intact and match COC?			Yes			
6	Were the correct containers and volumes received?			Yes			
9	Were samples received within EPA hold times?				Yes		
10	Were correct preservatives used? (water only)						
11	Were pH ranges acceptable? (voa's excluded)				NA		
12	Were VOA samples free of headspace (less than 5mm)?			NA			

Microbac Laboratolies + Ohio Valley Division 158 Starlite Drive, Marietta, OH 45750 + T: (740)373-4071 F: (740)373-4835 www.microbac.com

Page 2

L13080127 / Revision: 0 / 8 total pages

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MICROBAC SOP #:	LOGIN01
PAGE:	28 of 30
REVISION:	19

Figure 2

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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,

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MICROBAC SOP #:	LOGIN01
PAGE:	29 of 30
REVISION:	19

Figure 3

Ohio Valley Division

MICROBAC'

Water Short Hold Times

6 Hour	24 Hour	48 Hour	
Fecal / MPN*	CR-6	BOD	
CI-TRL	E	Color	
DO-L	×.	NO3	
pH-L	×.	NO2	
SO3	~	PO4 (orthophosphate)	
	-	Set-5	
81. II.		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			

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LOGIN01
30 of 30
19

Figure 4

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COOLER TEMP >6° C LOG

	Bottle 1	Bottle 2	Bottle 3	Bottle 4	Bottle 5	Bottle 6
SAMPLE ID	°c	°C	°C	°C	٥c	°C
	-					
		-	1			
_						
_		1				
			Exceptions			

nH Lot

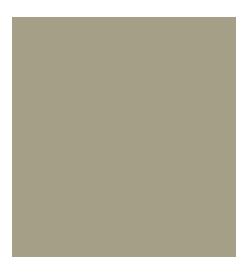
SAMPLE ID	Bottle 1	Bottle 2	Bottle 3	Bottle 4	Bottle 5	Bottle 6
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L3 - Volatile Organic Compounds by GC/MS – SW 846 Method 8260B/C



QA Control Code: A2040038

SOP Name:	Volatile Organic Compounds by GC/MS - SW 846 Method 8260B/C
SOP ID:	M8260B/C-SWGCMSVOA
Revision #:	22
Date Created:	April 9, 2002
Effective Date:	May 5, 2017
Reason for Revision:	Audit findings
Supercedes:	M8260B/C-SWGCMSVOA-21

Approvals:

Analyst

Supervisor

QA/QC Director

Technical Director

Date

Date

Date

Date

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VOLATILE ORGANIC COMPOUNDS BY GC/MS

1. TEST METHOD

1.1 Determination of Volatile Organic Compounds by using SW-846 Method 8260B/C using SW-846 Method 5030B-Purge and Trap for Aqueous Samples and SW-846 Method 5035A – Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples.

2. APPLICABLE MATRICES

2.1 Ground and surface water, wastewater, aqueous sludges, soils and sediments.

3. DETECTION LIMITS

3.1 MDL is verified quarterly.

4. SCOPE AND APPLICATION

- 4.1 This SOP outlines the procedure used to determine volatile organic compounds by Gas Chromatography/Mass Spectrometer (GC/MS). This SOP is used for both aqueous and non-aqueous samples, with method variations described where applicable to the different matrices.
- 4.2 The compounds determined by this method can be found in Table 1.

5. SUMMARY OF TEST METHOD

- 5.1 <u>Water Samples</u>
 - 5.1.1 Helium is bubbled through a 5mL/25mL portion of the sample in a purge chamber at 30 to 40mL/min at ambient temperature.
 - 5.1.2 The purgeables are transferred from the aqueous to the vapor phase and are passed through a sorbent trap.
 - 5.1.3 After purge time is complete, the trap is heated and backflushed with helium to desorb the purgeables onto the gas chromatographic (GC) column.
 - 5.1.4 The GC is temperature programmed to separate the purgeables, which are then detected with a mass spectrometer.
 - 5.1.5 The peaks detected are identified by retention time and characteristic ion patterns.
 - 5.1.6 Quantitation is done using the internal standard technique along with response factors generated by running known amounts of standards.
- 5.2 <u>Soil Samples: Low Level</u>
 - 5.2.1 A small diameter soil core-sampling device is used to collect about 5g of soil sample.
 - 5.2.2 The sample is either extruded into a tared sample container supplied by the laboratory, either containing 5mL organic-free water and magnetic stir bar, or 1g sodium bisulfate in 5mL water with magnetic stir bar, or magnetic stir bar, or the samples may be shipped in EnCore samplers.
 - 5.2.3 If samples are received in EnCore samplers, either analyze the samples within 48 hours or transfer them to tared 40mL glass sample containers and note the weight of the sample and the date and time of transfer.

- 5.2.4 Add 5mL organic free reagent water to soil samples received without the reagent water or sodium bisulfate.
- 5.2.5 Analyze by purge and trap GC/MS, under a heated curve.
- 5.3 Soil Samples: High Level Methanol Preserved
 - 5.3.1 A small diameter soil core-sampling device is used to collect about 5g of soil sample.
 - 5.3.2 The sample is extruded into a tared sample container supplied by the laboratory, containing 10mL of purge and trap grade methanol.
 - 5.3.3 Analyze 100µL of methanol extract in 5mL of organic free reagent water and analyze by purge and trap GC/MS, under a non-heated curve.

6. **DEFINITIONS**

- 6.1 <u>Calibration</u>: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurement. (NELAC)
- 6.2 <u>Internal standards</u>: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
- 6.3 <u>Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample)</u>: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.
- 6.4 <u>Matrix Spike (spiked sample or fortified sample)</u>: A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
- 6.5 <u>Matrix Spike Duplicate (spiked sample or fortified sample duplicate)</u>: A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
- 6.6 <u>Method Blank</u>: A sample of a matrix similar to the batch of associated samples (when available) that is free form the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations the impact the analytical results for sample analyses.
- 6.7 <u>Method Detection Limit</u>: The minimum concentration of a substance (an analyte) that can be measured and reported with 99 % confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
- 6.8 <u>Quantitation Limits</u>: The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user.

- 6.9 <u>Trip Blank</u>: Organic-free reagent water that is place in a 40mL vial and carried through sampling and handling to serve as a check on the contamination of volatiles by diffusion.
- 6.10 <u>Volatile Organic Compound</u>: Any compound containing carbon and hydrogen or containing carbon and hydrogen in combination with any other element which has a vapor pressure of 1.5 psi absolute (77.6 mm Hg) or greater under actual storage conditions.
- 6.11 <u>Verification</u>: Confirmation by examination and provision of evidence that specified requirements have been met.

7. INTERFERENCES

- 7.1 Common interferences with this method include impurities in the purge or carrier gas; leaks within the purge and trap unit or the GC/MS system; and solvent vapors (particularly methylene chloride) within the laboratory.
- 7.2 All plumbing materials used in connection with the purge and trap unit and the GC are stainless steel, copper, or Teflon rather than non- polytetrafluoroethylene (PTFE) or plastic tubing since this type of material may out-gas organic compounds.
- 7.3 Analyze laboratory reagent blanks after each calibration to show that the system is free of contamination.
- 7.4 Contamination by carry-over can occur when a low-level sample is analyzed immediately after a high level sample.
 - In this case, the system must be proven clean with the analysis of a blank, and the low-level sample must be reanalyzed.

8. SAFETY

- 8.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined, therefore treat each chemical compound as a potential health hazard.
- 8.2 Wear appropriate safety clothing and eye protection to minimize the exposure.
- 8.3 Use protective gloves when handling corrosive chemicals.
- 8.4 Read Material Safety Data Sheets (MSDS) for the chemicals used in the laboratory for the identity of the ingredients, the physical and chemical characteristics of the substance, the physical hazards, safe handling and safety precautions.

9. EQUIPMENT AND SUPPLIES

- 9.1 <u>Sample containers</u>
 - 9.1.1 40mL glass VOA vials with screw cap, Greenwood Catalog#340C1251443, or equivalent.
- 9.2 <u>Syringes</u>
 - 9.2.1 5mL glass gas-tight with shut-off valve SGE Catalog #008760 or equivalent.
 - 9.2.2 10μL (Hamilton Catalog #80000), 25μL (Hamilton Catalog #80200), 50μL (Hamilton Catalog #80900), 100μL (Hamilton Catalog #81000) and

1mL (Hamilton Catalog #81330) or equivalent glass gas-tight microsyringes.

9.3 <u>Volumetric flask</u>

9.3.1 Class "A" glassware only. 5mL, 10mL, 50mL and 100mL sizes used to prepare stock standards.

9.4 <u>Balances</u>

9.4.1 Top loading balance (Mettler PE300 or equivalent) capable of reading to ± 0.01 g.

9.5 <u>pH paper</u>

- 9.5.1 pH paper EMD Cat # EM9580 or equivalent.
- 9.6 Purge and Trap System
 - 9.6.1 See Table 5, 6 and 7
 - 9.6.2 The desorber is capable of rapidly heating the trap as required by this method. The temperature program begins at the purge temperature, continues to the desorb temperature, and ends with the bake temperature.
 - 9.6.3 Purging chambers are designed to accept a 5mL/25mL sample size with a water column at least 3 cm deep.
 - The purge gas flows through the sample in finely divided bubbles.

9.7 <u>Gas Chromatograph</u>

- 9.7.1 GCs used for analysis are Hewlett Packard 5890s or equivalent.
- 9.7.2 Different GC columns are used based on analytical method and target compound separation.
- 9.7.3 For instrument specifications, see Table 8, 9 and 10.
- 9.8 <u>Mass Spectrometer</u>
 - 9.8.1 Hewlett Packard 5971/5972 mass selective detectors or equivalent are used for this procedure. See Table 8.
- 9.9 Data Systems
 - 9.9.1 Hewlett Packard MSD Chemstation Software is used to view, evaluate, quantitate and print the data.
 - 9.9.2 Hewlett Packard MSD Chemstation Software Version: MSD Chemstation E.02.02.1431, MSD Chemstation C.00.07 MSD Chemstation G1701BA Version B.01.00, MSD Chemstation D.01.00 MSD Chemstation G1034 Version C.01.05 MSD Chemstation D.03.00.611
 - 9.9.3 Mass spectral library from HP Analytical, NIST02 MS Spectral Database is used in tentative identification of unknown peaks.
 - 9.9.4 Store all GC/MS data on magnetic media for five years, so that it may be retrieved as needed once the hard disk has been cleared.

10. REAGENTS AND STANDARDS

- 10.1 Reagents
 - 10.1.1 DI Water analyte free, generated by boiling de-ionized water and transferring the hot water to a clean glass jar for cooling before use.

- 10.1.2 Methanol purge and trap grade. Used in the preparation of stock standards, and for extraction of soils. JT Baker Catalog #9077-02 or equivalent.
- 10.1.3 p-BromoFluoroBenzene (BFB) *Supplier subject to change
- 10.1.4 Trip Blank: Prepare Trip Blank with 5mL analyte-free water in 40mL vials, acidified by 1:1 HCl to pH < 2. Label the trip blank vial with the initial of the preparer and the date and time that the trip blank was prepared.
- 10.2 Standards
 - 10.2.1 Prepare fresh standards as needed, store them in glass vials with Teflon faced septa. Replace after 6 months. For standard preparation see table 11
 - 10.2.2 The initial verification standards are purchased from a second source or a different lot number from the same supplier is used.

11. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

- 11.1 <u>Water Samples</u>
 - 11.1.1 Sample containers used for this method are glass bottles with Teflon faced septa or Teflon faced lid-liners.
 - 11.1.2 Collect at least two vials for each sample to allow for possible re-runs or dilutions.
 - 11.1.3 Collect a third volatile vial to ensure the sample is properly preserved.
 - After analysis, record in the instrument logs the pH of the sample.
 - 11.1.4 Collect extra sample if site specific matrix spike and matrix spike duplicate (MS/MSD) are required.

Note: Care should be taken when sampling such that no air bubbles or headspace is present in the sample containers.

- 11.1.5 Preserve water samples to be analyzed for aromatics with 1:1HCl to pH<2.
- 11.1.6 Samples are iced at 2-6°C upon sampling and delivered to the laboratory.
- 11.1.7 Samples are stored at 2-6°C from the time of receipt until analysis.
- 11.1.8 Analyze preserved samples within 14 days from sampling and unpreserved samples within 7 days from sampling.
- 11.2 <u>Soil Samples: High Level Closed-System vials (preserved with Methanol)</u>
 - 11.2.1 Sample vials are provided by the laboratory containing 10mL of Methanol.
 - 11.2.2 Weigh bottles before they leave the laboratory.
 - 11.2.3 Add sample using a special device that will deliver approximately 5g of sample directly into the vial.
 - 11.2.4 Seal vial immediately, ice at 2-6°C and deliver to the lab.
 - 11.2.5 Upon receipt, weigh samples and record weight for use in final sample calculations.
 - 11.2.6 Store samples at 2-6°C until analysis.
 - 11.2.7 Analyze 100uL of the methanol extract in 5mL organic-free water or equivalent, within 14 days from sampling.

Note: A separate container is required for percent moisture determination.

11.3 Soil Samples: Low Level using EnCore Samplers

- 11.3.1 The laboratory provides EnCore samplers for sample collection.
- 11.3.2 Collect at least 3 EnCore samples.
- 11.3.3 Ice samples at 2-6°C or freeze at -7 to -15°C and deliver to laboratory.

Note: A separate container (4oz or 8oz jar) is required for percent moisture determination purposes.

- 11.3.4 Upon receipt, the whole EnCore kit sample (about 5.0g) is transferred into 40ml vial within 48 hours.
- 11.3.5 Record the date and time of sample transfer.
- 11.3.6 Seal vial immediately, and either analyze immediately with 5mL organic-free water within 48 hours or freeze at -7 to -15°C, within 48 hours from sampling, for analysis with 5mL organic-free water within 14 days from sampling.
- 11.4 <u>Soil Samples: Low Level Closed-System vials (no chemical preservation)</u>
 - 11.4.1 Collect about 5g soil sample in 40mL labeled, tared vial with stir bar.
 - 11.4.2 Collect at least 3 vials for analysis, and another vial for determination of percent solids.
 - 11.4.3 Ice samples at 2-6°C or freeze at -7 to -15°C and deliver to the laboratory.
 - 11.4.4 Upon receipt, weigh samples and record weight for use in final sample calculations.
 - 11.4.5 Store samples at 2-6°C or freeze at -7 to -15°C and analyze with 5mL organic-free water within 48 hours or freeze at -7 to -15°C, within 48 hours from sampling, for analysis with 5mL organic-free water within 14 days from sampling.
- 11.5 <u>Soil Samples: Low Level Closed-System vials (no chemical preservation) with</u> <u>organic-free water</u>
 - 11.5.1 Collect about 5g soil sample in 40mL labeled, tared vial with 5mL organic-free water and stir bar.
 - 11.5.2 Collect at least 3 vials for analysis, and another vial for determination of percent solids.
 - 11.5.3 Ice samples at 2-6°C or freeze at -7 to -15°C and deliver to the laboratory.
 - 11.5.4 Upon receipt, weigh samples and record weight for use in final sample calculations.
 - 11.5.5 Store samples at 2-6°C or freeze at -7 to -15°C and analyze with 5mL organic-free water within 48 hours or freeze at -7 to -15°C, within 48 hours from sampling, for analysis within 14 days from sampling.
- 11.6 Soil Samples: Low Level Closed-System vials preserved with sodium bisulfate
 - 11.6.1 Collect about 5g soil sample in 40mL labeled, tared vial with 1g sodium bisulfate in 5mL organic-free water and stir bar.
 - 11.6.2 Collect at least 3 vials for analysis, and another vial for determination of percent solids.
 - 11.6.3 Ice samples at 2-6°C and deliver to the laboratory.

- 11.6.4 Upon receipt, weigh samples and record weight for use in final sample calculations.
- 11.6.5 Store samples at 2-6°C for analysis within 14 days from sampling.

Note: This preservation technique may result in destruction or creation of certain volatile organic compounds. Use this preservation technique only if Vinyl Chloride, Trichloroethene, Styrene, 2-Chloroethyl vinyl ether, Trichlorofluoromethane, cis-1,3-Dichloropropene, trans-1,3-Dichloropropene and Acetone are not contaminants of concern or the soil does not contain carbonaceous material.

- 11.7 <u>Soil Samples: Low Level</u>
 - 11.7.1 Samples are collected in 4oz or 8oz jars with no headspace and iced at 2- 6° C.
 - 11.7.2 Collect a separate jar for percent solids determination.
 - 11.7.3 Samples are stored at 2-6°C.
 - 11.7.4 If samples are analyzed within 48 hours of sampling, analyze about 5g well-mixed sample with 5mL organic-free water in a 40mL glass vial. Sample received in 4 oz jar can be preserved within 48 hours into Terracores. Preserved samples then can be analyzed within 14 days.
 - 11.7.5 If samples cannot be analyzed within 48 hours from sampling, preserve by adding about 5g well mixed sample to 10mL methanol in a 40mL glass vial. This preserved sample must be analyzed within 14days of sampling. Analyze 100uL of the methanol extract with 5mL organic-free water.

Note: This technique may be used for waste characterization, unknown or oily wastes, where chemical reaction with freezing or preservative is not known. The sample must be mixed very quickly with a spatula or equivalent device and added to 5mL organic-free water to minimize the loss of volatile organic compounds.

Any vial that is frozen must be laid on its side to prevent breakage, and thawed before analysis.

12. QUALITY CONTROL

- 12.1 BFB MS Tuning Check Compound
 - 12.1.1 Analyze every 12 hours.
- 12.2 Initial Calibration
 - 12.2.1 Analyze a minimum of five concentration levels, for e.g.: 1, 5, 20, 50, 100, 150, 200µg/L. (Concentration levels are subject to change based on instrument sensitivity and/or saturation, and project requirements, certain ketones and other compounds are added at an elevated concentration).
 - 12.2.2 Assure that relative response factors (RRFs) and % Relative Standard Deviation (%RSD) criteria are met.
 - 12.2.3 A new initial calibration is required when continuing calibrations do not pass required criteria. A new initial calibration is required after 31 days.

12.2.4 Set the retention time window using the midpoint standard of the curve when ICAL is performed.

12.3 <u>Continuing Calibration</u>

- 12.3.1 Analyze a calibration check solution from the primary every 12 hours immediately after the BFB.
- 12.3.2 Solution is used to verify instrument performance as compared to the initial calibration.
- 12.3.3 Assure that RRFs and % Difference (%D) criteria are met.
- 12.3.4 Retention for CCC, Samples and QC is updated using mid-point of ICAL. Retention is not updated using CCV check samples.

12.4 Method Blanks

- 12.4.1 Prepare specifically for each matrix type.
- 12.4.2 Analyze immediately after the calibration standards each day to ensure that the system is free from carry-over or any other interferences.

12.5 <u>Surrogates (S)</u>

- 12.5.1 Monitor and report for all blanks, samples, and spikes.
- 12.5.2 Assure that recoveries are within limits.
- 12.6 <u>Matrix Spike/Matrix Spike Duplicate and Blank Spike</u>
 - 12.6.1 Choose a representative sample to be used for MS/MSD.
 - 12.6.2 MS/MSD is required for each matrix type. For water samples, MS/MSD is analyzed only if the client provides extra sample volume. Otherwise, a Blank Spike and Blank Spike Duplicate are analyzed.
 - 12.6.3 MS/MSD and LCS are required for every group of samples run as a batch or every 20 samples.
 - 12.6.4 Calculate % Recovery and Relative Percent Difference (RPD).
- 12.7 Internal Standards (IS)
 - 12.7.1 Monitor the integrated area and the retention time of the quant ion of the IS for all standards, blanks, samples and spikes.
- 12.8 Accuracy and Precision
 - 12.8.1 Each analyst must perform an initial, one time demonstration of accuracy and precision. Documentation must be delivered to the QA officer for inclusion in personnel folder.
 - 12.8.2 Prepare four aliquots of LCS sample from a source other than that used for calibration.
 - 12.8.3 Analyze these four aliquots under the same conditions used for sample analysis.
 - 12.8.4 IDOC must be performed every time there is a significant change in the method, personnel, instrument type, or sample matrix.
 - 12.8.5 All of the IDOCs are kept in the employee's training files.
- 12.9 <u>Method Detection Limits</u>
 - 12.9.1 Determine the MDLs by analyzing seven replicate standards each containing analytes at a concentration of $1 / 5 \mu g/L$.
 - 12.9.2 After analysis, down load the data to a personal computer and use standardized MDL templates to perform the statistical calculations in excel.

- 12.9.3 Calculate the MDL by determining the standard deviation of the values and multiplying by the "t" value.
- 12.9.4 The calculated MDL must be below the quantitation limits for the method. If they are not, the data is reviewed again for possible sources of error and the procedure will be repeated.
- 12.9.5 Perform an MDL study initially for all normally targeted compounds or when conditions change.
- 12.9.6 Perform an MDL study for extra targeted compounds as required.
- 12.9.7 Perform an MDL study on one instrument for each type of test being performed. (Low Soil, High Soil, Water 5 mL and 25 mL purge).
- 12.10 Manual Integration
 - 12.10.1 At times, manual integration will be necessary due to incomplete or incorrect integration by the automated analytical system.
 - 12.10.2 Manual integration cannot be used to satisfy Quality Control Criteria.
 - 12.10.3 Do not include baseline background noise; include only the area between where the beginning and end of the peak intersects with the baseline.
 - 12.10.4 Any time a compound is integrated in the calibration standard it must then be consistently integrated in the samples per professional judgment.
 - 12.10.5 When a manual integration is performed, the hardcopy of the quantitation report will flag the compound with an "m".
 - 12.10.6 Print the before and after manual integration chromatograms with the raw data.
- 12.11 Limit of Detection (LOD)
 - 12.11.1 Establish LOD by spiking a quality system matrix at approximately 1-4X detection limit.
 - 12.11.2 LOD is specific to each combination of analyte, matrix, method (including sample preparation) and instrument configuration.
 - 12.11.3 LOD must be verified quarterly.
 - 12.11.4 LOD must be verified on each instrument used, and every time the method is modified.
- 12.12 Limit of Quantitation (LOQ)
 - 12.12.1 LOQ must be greater than the LOD.
 - 12.12.2 LOQ must be verified quarterly for each quality system matrix, method and analyte, by analyzing QC sample containing the analytes of concern in each quality system matrix 1-2X the claimed LOQ.
 - 12.12.3 LOQ must be performed if the method is modified.
- 12.13 Initial Calibration Verification (ICV)
 - 12.13.1 Analyze a second source initial calibration verification standard at mid level concentration immediately following the initial calibration curve.

13. CALIBRATION AND STANDARDIZATION

- 13.1 <u>GC/MS Tuning and Performance Check</u>
 - 13.1.1 Analyze the BFB solution every 12 hours to verify acceptable instrument performance.
 - 13.1.2 Reture the MS and reanalyze the BFB if the spectrum does not meet criteria.

- 13.1.3 Tune the mass axis and abundance scales such that the analysis of the instrument performance check solution (BFB) meets the criteria outlined in Table 2.
- 13.1.4 Once an acceptable BFB has been acquired, instrumental conditions must remain the same throughout the calibration and sample analyses.
- 13.1.5 Prior to the analysis of Initial calibration standards, tune the GC/MS system using perfluorotributylamine (PFTBA).
- 13.2 Initial Calibration
 - 13.2.1 After tuning criteria have been met, analyze an initial calibration consisting of a minimum of five calibration standards; e.g. at the following concentration levels: 1, 5, 20, 50, 100, 150 and 200µg/L (The standard concentrations may be subject to change based on instrument sensitivity and/or saturation, and project requirements. Ketones and certain other compounds are added at elevated concentrations).
 - 13.2.2 Analyze an initial calibration verification standard from a second source.
 - Note: The lowest standard analyzed must be equal to the reporting limit.
 - 13.2.3 Tabulate the area response of the characteristic ions against the concentration for each target analyte and internal standards using MS Chemstation software.
 - 13.2.4 The RRF is calculated as follows:
 - $RRF = \underline{A_{\underline{s}} \times C_{\underline{is}}}{A_{\underline{is}} \times C_{\underline{s}}}$

Where

- A_s = Peak area of the analyte or surrogate
 - A_{is} = Peak area of the internal standard
 - C_s = Concentration of the analyte or surrogate
 - C_{is}= Concentration of the internal standard
- 13.2.5 Calculate the %RSD for all target analytes from the initial calibration.

 $%RSD = Standard Deviation of RRF \times 100$

Mean of RRF

Where: mean of $RRF = \underline{sum of RRF}$

n = number of calibration standards used

- 13.2.6 The %RSD should be $\leq 15\%$ for each target analyte for Method 8260B and $\leq 20\%$ for each analyte for Method 8260C.
 - 13.2.6.1 System performance check compounds (SPCCs) (For Method 8260B): Minimum Average RRF for Chloromethane, 1,1-Dichloroethane and Bromoform must be 0.10; Minimum Average RRF for Chlorobenzene and 1,1,2,2-Tetrachloroethane must be 0.30.
 - 13.2.6.2 Calibration Check Compounds (CCCs) (For Method 8260B): The RSD for each individual CCC must be \leq 30%. The CCC include 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, Vinyl Chloride.
 - 13.2.6.3 1,4-Dioxane minimum RRF requirement is 0.05 and <50% RSD.

- 13.2.6.4 For DOD, Each analyte must meet one of the three options, Option 1: RSD for each analyte $\leq 15\%$, Option 2: linear least squares regression for each analyte: $r2 \geq 0.99$, Option 3: nonlinear least squares regression (quadratic) for each analyte: $r2 \geq$ 0.99.
- 13.2.7 When the %RSD of all target analytes meet criteria, the curve is assumed to be constant over the calibration range, and the average response factor is to be used for quantitation.
- 13.2.8 When the client requests extra target compounds, a curve for these compounds will be deemed acceptable only when a $\pm 30\%$ RSD is achieved between the five initial responses factors.
- 13.2.9 When the %RSD exceeds criteria, perform a linear regression (five-point curve) or quadratic regression (six-point curve) of the instrument response versus the concentration of the standards. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression will produce the slope and intercept terms for a linear equation in the form

y = ax + b,

Where

y = instrument response (peak area or height)

- a = slope of the line(also called the coefficient of x)
- $\mathbf{x} =$ concentration of the calibration standard
- b = intercept
- 13.2.9.1 The use of linear regression may not be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards.
- 13.2.9.2 The regression calculation will generate a correlation coefficient(r).
- 13.2.9.3 In order to be used for quantitative purposes, the correlation coefficient must be greater than or equal to 0.990.
- 13.2.9.4 Inspect the curve to determine if the linearity fits all the standards.
- 13.2.9.5 If the criteria cannot be met, recalibrate the instrument again or report the failures in the case narrative and/or non-conformance sheet.
- 13.2.10 Establish the retention time window position for each analyte and surrogate, once per initial calibration, at the midpoint standard of the initial calibration curve.
- 13.2.11 The relative retention time (RRT) is established by calculating the ratio of retention time of analyte over retention time of its associated internal standard.
- 13.2.12 The RRT of each target analyte must be within ± 0.06 RRT units. If criteria are not met, then correct the problem by performing instrument maintenance, and then rerun the initial calibration curve.
- 13.2.13 If the height of the valley between two isomer peaks < 25% of the sum of the two peak heights, then the isomers are reported as individual

compounds. Otherwise, structural isomers are identified as isomeric pairs. E.g. m/p-Xylenes.

- 13.3 <u>Continuing Calibration</u>
 - 13.3.1 Analyze a BFB. Make sure it meets criteria listed in Table 2.
 - 13.3.2 Analyze a continuing calibration check standard and compare it to the mean RRF of the initial curve rather than running an entire initial calibration curve every 12 hours.
 - 13.3.3 Calculate %D for all target analytes.

$$\%D = \frac{RRF_{C} - RRF_{I}}{RRF_{I}} \times 100$$

Where $RRF_C = Relative Response factor from continuing calibration$ $RRF_I = Mean Relative Response factor from initial calibration$

- 13.3.4 If continuing calibration passes criteria listed in Section 18.3, proceed with analysis of blanks and samples.
- 13.4 <u>Method Blank and Blank Spike</u>
 - 13.4.1 Prepare specifically for each matrix type.
 - 13.4.2 Analyze immediately after the calibration standards each day.
 - 13.4.3 If the method blank passes criteria listed in Section 18.4 and Blank Spike passes criteria in Section 18.6, proceed with analysis of samples.
- 13.5 Initial Calibration Verification
 - 13.5.1 Analyze second source ICV immediately after the initial calibration standards.

14. **PROCEDURE**

- 14.1 Allow all standards to warm to ambient temperature prior to use.
- 14.2 Rinse all syringes to be used with purge and trap quality methanol.

Note: Analyze samples using a 12-hour sequence.

• The 12-hour period begins with the injection time of the BFB.

Convention for Data File Naming

• Subdirectories are named according to the department name, than instrument name, month, date, and lastly the file number E.g. VA091702

WhereDepartment= VOAInstrument= AMonth= SeptemberDate $= 17^{th}$ Year= 2002

• Data File is named as: department name – instrument name – sequentially. *E.g. VA000001, VA000002 etc.*

14.3 BFB Tuning

- 14.3.1 Add 2µl of 25µg/mL BFB solution to 5mL/25mL reagent water and purge.
- 14.3.2 Use the same conditions for the BFB as for all blanks, standards, samples and spikes.
- 14.3.3 Analyze the BFB as follows:

- Click on the instrument icon.
- Edit sequence to run BFB
- Click on OK
- Click on run sequence
- Wait for instrument to complete the run
- 14.3.4 Use the MSChemStation software to acquire the spectrum of BFB in the following manner:
 - Integrate m/z 95 (the major ion of BFB) to find the max scan or apex of the peak.
 - Average three scans; the max scan and the scans immediately before and after the max.

Note: Background subtract, must be a scan chosen before the elution of the BFB peak but no more than 20 scans from the beginning of the BFB peak.

- 14.3.5 Check the resulting spectrum; it must meet the ion abundance criteria outlined in Table 2.
- 14.4 Initial Calibration
 - 14.4.1 After tuning criteria have been met, initially calibrate the GC/MS system at a minimum of five concentration levels. See Table 12 for water working standard preparation and Table 13 for soil working standard preparation.

Note: Calibration standards for water matrix are made in 40ml vial and for soil matrix in 5ml syringe.

- Aqueous samples and high level soils are purged at ambient temperature, and low-level soils are purged at 40° C.
- Therefore, calibrations for waters and high level soils must use an unheated purge, while calibrations for low level soils require a heated purge at 40°C.
 - 14.4.1.1 Analyze all standards, blanks, and samples under the following instrumental conditions:
 - Click on the instrument icon.
 - Click on Edit sequence to run the curve
 - Click on OK
 - Click on run sequence
 - Wait for instrument to complete the run

Note: The GC column separates the analytes that are then detected by the mass spectrometer.

14.4.2 Acquire data for each of the five calibration points.

• Compare the data using a METHOD FILE set up for the target, internal standard, and surrogate compounds, containing expected retention times, and ion ratios for each analyte.

- A quant ion and one or two secondary ions have been chosen (Table 3) for each analyte and make up a characteristic ratio used to identify each compound.
- The quant ion for each compound is integrated and these areas are used to generate RFs.
- 14.4.3 Create a calibration file inside the METHOD from the data points run for the initial curve.
 - The METHOD shows a RF for each analyte at each concentration level.
 - The average RF, the relative retention time (each analyte's distance from the internal standard), and the Relative Standard Deviation (RSD) are calculated.
- 14.4.4 Once a valid initial curve is run and evaluated, run ICV and then proceed with the analysis of blanks, spikes and samples if there is time remaining in the 12-hour period.
 - Update the average response factors from the curve into the METHOD and they will be used for quantitation for all blanks and samples that follow.
 - If there is no time remaining, begin a new 12-hour sequence with the analysis of a BFB.
 - If the BFB passes criteria, analyze a continuing calibration check standard.
- 14.5 Continuing Calibration
 - 14.5.1 Analyze a BFB.
 - 14.5.2 If the BFB passes criteria, analyze a continuing calibration check standard.
 - 14.5.3 If the continuing calibration meets criteria, proceed with the analysis of blanks and samples.
 - 14.5.4 If continuing calibration does not meet criteria (Section 18.3), analysis must stop. See section 19.3.
 - 14.5.5 A continuing calibration must be performed every twelve hours. Monitor internal standard areas and retention times for the continuing calibration verification.
 - The extracted ion current profile (area of the quantitation ion) must not change by more than a factor of 2 in either direction from the midpoint of the initial calibration.
 - The retention time for any internal standard must not change by more than 30 seconds.
 - 14.5.6 Should either of these two items be out of limits, the GC/MS system must be inspected for potential problems and corrections made as needed.
- 14.6 Method Blank and Blank Spike
 - 14.6.1 Analyze a method blank immediately following either the initial or continuing calibration of the GC/MS system, and prior to analyzing any samples.
 - 14.6.2 For Method Blank preparation, see Table 4.
 - 14.6.3 Purge the water blank and methanol soil blank at ambient temperature and the soil blank at heated purge.

14.6.4 Analyze the method blank after the calibration standards to ensure that the system is free from carryover or any other interferences that may be present.

• Note: No analytes may be present in the blank above the RL with the following exceptions: Methylene Chloride and Acetone are allowed to be present at a level of 2x RL. These compounds are routinely found in the air in the laboratory. Identification of these compounds in a sample at or above the RL have to be flagged with a B on the result page for the sample and a discussion in the case narrative needs to be included about the positive identification of these compounds in the sample.

- 14.6.5 The method blank must meet the same QC requirements as the samples for that particular matrix type.
 - Surrogate recovery limits and internal standard area criteria must be met for a blank to be valid.
- 14.6.6 If the blank does not meet criteria, the system must be checked for problems and action may need to be taken.
 - The system may need to be baked out to remove residue from previous samples. Heat oven to 220°C for one hour and bake the trap. Increase the temperature of the transfer line.
 - A new blank must be run and criteria met before analysis of samples can begin.
- 14.7 Sample Analysis

Note: Samples may only be analyzed once the tune, calibration, and blank have all met criteria except in cases where samples must be loaded on the instrument overnight, in which case, the QC and calibration samples are checked after analysis.

- Before loading the sample, rinse the 5mL syringe with reagent water 3 times.
- Allow all samples to warm to ambient temperature before loading.

14.7.1 Water Samples prepared manually

- 40ml sample vial is prepared by adding surrogates and internal standards as described in Table 4.
- The vial is loaded on Autosampler.
- Autosampler takes 5ml/25ml in to the sparge tube.
- Determine the pH of each water sample and record it on the Analysis Run log page.
- Test the pH by dipping the pH paper into the sample vial after analysis is complete.
- Record the pH of each sample.
- 14.7.2 Water Samples loaded on the Autosampler
 - Load the vial onto the ARCON auto-sampler where the robotic mechanisms move the sample through steps that include:

- collection of 25mL/5ml of water,
- add surrogate or internal standard as described in Table 4
- 11minute purge
- 14.7.3 High Level Soil Analysis of Methanol preserved samples
 - Add 100μ L of the methanol extract to a 40mL vial containing 40mL of reagent water.
 - Add internal standards and surrogates as described in Table 4.
- 14.7.4 Low-level Closed-System Soil Analyses for samples containing Sodium Bisulfate preservative or organic-free water
 - Load the vial onto the ARCON auto-sampler where the robotic mechanisms move the sample through steps that include:
 - addition of internal standards and surrogates as described in Table 4
 - heating for 1.5 minutes at 40°C
 - stirring the sample and maintaining 40°C during the 11minute purge time.
- 14.7.5 Low-level Soil Analysis for samples without preservative or organic-free water
 - using a 5mL syringe, add 5mL of organic-free reagent water with addition of surrogate and internal standards as described in Table 4
 - Load vial to Arcon Autosampler
- 14.7.6 Analyze the sample as follows:
 - Click on the instrument icon
 - Click on Edit sequence, add samples to sequence
 - Click on OK
 - Click on run sequence
 - Wait for instrument to be ready

Note: The auto-sampler unit goes through the same sequence for all samples, blanks, and standards.

- *Purge the sample with helium for 11 minutes.*
- Heat low level soils to $40^{\circ}C$ during this purge time, water and high level samples are purged at room temperature.

• The sample is desorbed while rapidly heating the trap and back-flushed with helium.

• The trap is then baked to remove any residue remaining on the trap.

• The trap is allowed to cool down to room temperature, and is then ready to accept the next sample.

Note: Any analyte that exceeds the calibration range requires a dilution.

14.7.7 Sample Dilutions

- If any target compound exceeds the initial calibration range in a sample, the sample must be diluted.
- The dilution factor must get the largest analyte peak in the upper half of the initial calibration range.

• All dilutions must meet the same QC requirements as non-diluted samples.

14.7.7.1 Water samples:

- For water samples requiring a 10x dilution, take 1mL aliquot sample with a gas tight 5mL syringe and add it to 9 mL reagent water in a Class A 10 mL volumetric flask.
- Invert the flask three times before adding contents to a 5mL gastight syringe.
- Add surrogate and internal as described in Table 4
- Further dilutions may be made in a similar manner depending upon the level of dilution required.
- 14.7.7.2 Low Level Soils:
 - For low level soil samples, the smallest amount of sample allowed to be weighed is 0.1 g.
 - Any sample requiring a more dilute analysis must be treated as a high level soil and extracted with methanol.
- 14.7.7.3 High Level Soils:
 - 14.7.7.3.1 For high level soils, dilutions are done by injecting less amount of methanol extract into the 5mL syringe. Example: inject 50uL for a 2x dilution.

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- 14.8 Matrix Spike/Matrix Spike Duplicate and Blank Spike
 - 14.8.1 With each group of samples analyzed as a batch, analyze a blank spike, matrix spike and matrix spike duplicate.
 - 14.8.2 The purpose of these matrix spikes is to determine whether the sample matrix contributes to the analytical results.
 - 14.8.3 Spike a representative sample with the target compounds.
 - 14.8.4 Calculate the % recovery and relative % difference (RPD) between the recoveries and ensure that they meet the criteria for the MS/MSD.
 - 14.8.4.1 Calculate the % recovery for the Blank Spike. 14.8.5 To calculate Spike recoveries:

.8.3	To calculate Spike recoveries:	
	SSR-SR	

	SA
Where:	SSR = spiked sample result
	SR = sample result (for MS/MSD calculation only)

- SA = spike added
- 14.8.6 Prepare water and low level soil matrix spikes and blank spike as described in Table 4
- 14.8.7 For high level soil matrix spikes, add spiking solution/internal standard/surrogate as described in Table 4 to 5g soil.
- 14.8.8 Extract the matrix spike and blank spike sample and analyze as any other high level sample.
- 14.8.9 Field or trip blanks may <u>not</u> be used for MS/MSD purposes.
- 14.8.10 One MS/MSD and Blank Spike is required for every group of samples run as a batch or at least one set of spikes per 20 samples and if MS/MSD is not given for water samples run blank spike and blank spike duplicate.
- 14.9 Analytical Sequence (Subject to change)

Initial Analytical	Continuous Analytical Run
Run	
• BFB0501	• BFB0502
VSTD001 ppb	• VSTD0502
VSTD005 ppb	• VBLK02
VSTD020 ppb	• Samples
VSTD050 ppb	• LCS
VSTD100 ppb	• MS
VSTD200 ppb	• MSD
• ICV	
VBLK01	
Samples	
• LCS	
• MS	
• MSD	

14.10 Manual Integration

Note: At times manual integration will be necessary due to incomplete or incorrect integration by the automated analytical system. This normally occurs when there is matrix interference, baseline noise or compound co-elution.

Manual integration cannot be used in order to solely satisfy Quality Control Criteria. It should also not be used as a substitute for corrective action on the chromatographic system. All manual integrations must be noted in the case narrative.

- 14.10.1 Integrate the area of the quantitation ion of the compound of interest.
- 14.10.2 Do not include baseline background noise, and include only the area between where the beginning and end of the peak intersects with the baseline.
- 14.10.3 Integrate the compound in the sample any time it is integrated in the calibration standard using professional judgment.
- 14.10.4 Flag the compound with an "m" in the hardcopy (quantitation report) when a manual integration is performed.
- 14.10.5 Print out the EICP for all compounds that have been manually integrated. Print out the spectrum of the manually integrated compound before and after the manual integration is done.
- 14.11 Data Interpretation
 - Maintain all GC and mass spectral data generated with each run of the instrument within a data file.
 - Store data files on the computer hard drive, and archive on the server for retrieval as needed once the hard drive has been cleared.

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- For quantitation, send data files through MSChemstation Software, where the computer compares known information about target compounds to what is present in each data file.
 - Information contained in the Method File used by the program includes: The relative retention time of each analyte The ion to be used for quantitation and one or two secondary ions, which are characteristic to each compound (Table 3). The response factor for each analyte to be used in determining the concentration.
- 14.11.1 Procedure

Naming Methods: Method prefix, instrument name, matrix, month, date, e.g., 82BS0104.M

- 14.11.1.1 Sequence log pages are maintained electronically for each instrument
- 14.11.1.2 Click the MSChemstation icon on the processing PC.
- 14.11.1.3 Load the method by using the pull down menu top left choice and click on select method.
- 14.11.1.4 Load the first BFB Data File from the first instrument log using the pull down menu top left choice and click on select data file.
- 14.11.1.5 Find the BFB peak on the chromatogram and click on the max scan (max ion 95).
 - Note the scan number.
- 14.11.1.6 Determine where the scan to the left and the scan to the right are located by clicking slightly to the right and left of the max scan noting the scan numbers.
- 14.11.1.7 Drag the cursor from the max scan -1 to the max scan +1.
 - Click on a background scan directly to the left of the BFB peak and click on subtract in the pull down menu called Tuner.
- 14.11.1.8 Click on "evaluate BFB".

Note: If all ion ratios pass, save the information in a file.

• The Autofind options under the Tuner pull down menu does the same thing as steps 14.11.1.6 – 14.11.1.10.

- 14.11.1.9 Click on Save BFB to Forms File under the Tuner pull down menu.
- 14.11.1.10 Click on Print BFB under the Tuner menu.
 - The criterion is listed in Table 2.
- 14.11.1.11 Load the midpoint file from the initial calibration.
- 14.11.1.12 Click on quantitate to screen
- 14.11.1.13 Click on clear all calibration responses
- 14.11.1.14 Click on calibrate
 - Add new level

- Enter standard level and 50 for internal standard concentration.
- 14.11.1.15 Load the next initial calibration data file.
 - Repeat steps 14.11.1.12 14.11.1.15
 - Do this for all five initial calibration points (5, 20, 50, 100, and 200µg/L).
- 14.11.1.16 Print out the initial calibration using the pull down menu, click on response factors to printer.
- 14.11.1.17 Carefully review all information on the printout.
 - Look for isomeric pairs that separate chromatographically and have the same retention time and response factors (ethylbenzene, o-xylene & m/p-xylene).
 - Verify that all compounds are picked up. Check to see if the initial calibration meets criteria.
- 14.11.1.18 Qarea using the pull down menu, each point that needs editing and repeat step 14.11.1.15 choosing recalibrate.
- 14.11.1.19 Load the second BFB.
- 14.11.1.20 Pass it by repeating steps 14.11.1.5 14.11.1.9.
- 14.11.1.21 Load the check standard data file.
 - Send to quant using the pull down menu.
 - Click on View Results on screen and verify that all of the compounds are being picked up by the program correctly. If not, Qarea using pull down menu.
- 14.11.1.22 Retention time for CCV, samples and QC is evaluated using the mid-point of ICAL. Retention time is not updated using the CCV check samples.
- 14.11.1.23 Verify that Quantitate using Initial Calibration is clicked on.
- 14.11.1.24 Load next data file (blank), quantitate it and review in qarea, checking surrogate recoveries, correct integration of peaks, internal standard area recoveries and any necessary dilutions of target compounds.
- 14.11.1.25 Repeat step 14.11.1.24 for each blank, sample and spike that is associated with the SDG maintaining the order of steps 14.11.1.20 14.11.1.26 when you get to the next BFB. See Section 14.11.2 for Data Interpretation.
- 14.11.1.26 Send each blank and sample to the tentative identified program using the software pull down menus. Use information from the summary discussion to review the non-target data.
- 14.11.1.27 Print out each run, standards and spikes in medium format (quant report and chromatogram), blanks and samples in full format (quant report + Chromatogram + spectra).
- 14.11.1.28 Put the reports in data file order with the BFB report first. Put the instrument logs with each set of reports.
 - Data is now ready for **EISC forms**.
- 14.11.2 Data Interpretation for MS Chemstation Software

11 722	
	14.11.2.1 Examine all spectra for all possible "hits" or matches made to target compounds from printed out file by an analyst trained in the interpretation of mass spectra by doing the following:
	14.11.2.2 Generate a reference spectrum for each analyte by running known standards (QREF from pull down menu).
	14.11.2.3 Compare this reference to the spectrum of the peak found in the sample.
	 14.11.2.4 Compare the criteria required for positive identification of an analyte as follows: The analyte in the sample must elute at the same relative retention time as in the daily calibration standard (±0.06 RRT)
	units).
	• All ions present in the reference spectrum >10% of the largest
	ion must be found in the sample spectrum.
	• The ratio of the ions found in the sample must agree within
	$\pm 20\%$ of the ions found in the reference spectrum.
	• Ions >10% in the sample spectrum but not found in the reference spectrum must be accounted for.
	• Quantitative analysis is done once a target compound is identified by the internal standard method using the equations below. The relative response factor from the initial calibration standard is used to calculate the concentration of the sample.
	14.11.2.5 Send all samples and blanks through a library search program in
	an effort to identify up to 30 non-target compounds, upon client's request.
	14.11.2.6 Do <u>not</u> report the following compounds:
	• Compounds less than 10% of the nearest internal standard area,
	• Compounds which elute earlier than 30 seconds before the first target compound or three minutes after the last purgeable compound,
	Carbon dioxide, and
	• Semi volatile target compounds.

- 14.11.2.7 The computer software provides a mass spectral library for comparison to unknown compounds found in samples. Criteria for making tentative identifications are:
 - Ions >10% of the largest ion in the reference spectrum must be present in the sample spectrum.
 - The relative intensities of major ions should agree within $\pm 20\%$.
 - Molecular ions present in the reference spectrum must be present in the sample spectrum.
 - Ions present in the sample spectrum, but not the reference spectrum should be reviewed for possible

background contamination or presence of co-eluting compounds.

- Ions present in the reference but not the sample should be verified by performing manual background subtraction to remove interferences.
- If after review, the analyst is at a loss to identify the compound use the following method:
 - If the computers match probability is 85% or greater report that compound.
 - If the computer match probability is <85%, try to classify the compound and give it a name like "unknown chlorinated hydrocarbon" if it can be determined.
- 14.11.2.8 Do the quantitation of tentatively identified compounds based on comparison of the total ion area of an unknown peak to the total ion area of the nearest internal standard:
 - Do not identify peaks that have an area <10% of the nearest internal standard.
 - Since no calibrations are run for these unknown peaks, use response factor of 1 to calculate concentrations.
- 14.11.2.9 Identify 15 of the largest alkane peaks if they are in the sample.
 - Also provide the library search information for each peak.

14.12 Documentation Requirements

14.12.1 Assure that GC and GC/MS Instrument log contains the following:

- CHEMTECH sample ID
- pH of water sample
- Dilution details
- All standards, samples, blanks, etc., run on the instrument in the order they were analyzed
- Date and time of injection of each sample and standard
- Computer data file number
- Analyst signature
- Supervisor signature
- 14.12.2 Label all chromatograms as follows:
 - CHEMTECH and/or client sample number
 - Volume/weight injected
 - Date and time of injection
 - GC column ID
 - GC Instrument ID
 - Identified compound names
- 14.12.3 The following quant reports and chromatograms and data system printouts must be included in the data package:
 - All standards and blanks from initial and continuing calibrations
 - All samples, blanks, blank spikes and MS/MSD
- 14.13 Instrument Maintenance

14.13.1 See Maintenance P255 SOP

- 14.14 % Moisture
 - 14.14.1 All soil results are reported on a dry weight basis. The % moisture is determined for all of the samples in the laboratory by the metals department.
- 14.15 Record in the logbook if there are any instrument errors.
 - Rerun the samples.

Note: Errors include

- Leaked samples
- Electric shutdown

15. CALCULATIONS

15.2

15.1 Water Calculation in ug/L

 $(A_x)(I_s)$ (Df) (A_{is}) (RRF) (V_0) Where $A_x =$ Area for the compound to be measured A_{is} = Area for the specific internal standard I_s = Amount of internal standard added in nanograms (ng) RRF = Relative response factor of the initial calibration curve standard. $V_0 =$ Volume of water purged in milliliters (mL) Df = Dilution factor. Low Level Soil Calculation in ug/Kg dry weight basis $(A_x)(I_s)(Df)$ (A_{is}) (RRF)(W_s)(D) Where A_x = Area for the compound to be measured A_{is} = Area for the specific internal standard I_s = Amount of internal standard added in nanograms (ng) RRF = Relative response factor of the initial calibration curve standard.

- Df = Dilution factor
- W_s = Weight of sample

D=100 - %moisture

100

15.3 High Level Soil Calculation in ug/Kg dry weight basis

<u>(A x)(I s) (Vt) 1000 (Df)</u>
(A_{is}) (RRF) $(V_a)(W_s)(D)$
A_x = Area for the compound to be measured

Where

 A_{is} = Area for the specific internal standard

 I_s = Amount of internal standard added in nanograms (ng)

RRF = Relative response factor of the initial calibration standard.

 V_t = Total volume of methanol extract in milliliters (mL), (usually 10 mL)

 $V_a =$ Volume of aliquot in microliters (uL) (usually 100 uL)

Df = Dilution factor

 W_s = Weight of sample D = <u>100 - %moisture</u> 100

Note: If there are interferences to the quant ion caused by either high background or co-eluting compounds with similar ions, use a secondary ion for quantitation. A list of the target analytes and their primary and secondary ions is found in Table 3.

16. METHOD PERFORMANCE

- 16.1 Analysis is performed in accordance with the method. All quality control and quality assurance procedures are followed. Refer to P203-IDOC,MDL SOP for further information.
- 16.2 Each analyst will make a one-time demonstration of the ability to generate acceptable accuracy and precision with this method. Refer to P203-IDOC, MDL SOP for further information.

17. POLLUTION PREVENTION

- 17.1 Use only the amounts of chemicals required. Do <u>not</u> make large quantities of solutions.
- 17.2 Use hood when working with solvents.
- 17.3 Keep the area clean and clutter free in the extractions lab and around the instruments in order to avoid any mishaps.
- 17.4 Trap exhaust from vacuum pumps.
- 17.5 Keep chemicals away from drains.
- 17.6 Properly collect and dispose of waste according to Chemtech's Waste Disposal SOP.
- 17.7 Laboratory is properly equipped with spill cleanup equipment and laboratory personnel trained. Depending upon the size and type of spill, it may be handled by the individual or department creating the spill or by specially trained personnel.
- 17.8 Small spills may occur routinely and shall be handled by the individual person or department creating the spill. Spill kits are stored in a blue basket or blue cover bin located in each laboratory and chemical storage area. The spill kits can handle water based, solvent and mercury spills. Specially trained personnel handle larger spills, which may pose a threat to health or environment involves a large volume not easily contained.
- 17.9 A detailed description of the procedure for handling a spill or accident is covered in the CHEMTECH Emergency and Contingency Plan.
- 17.10 The Safety Coordinator is responsible for implementing the Chemical Hygiene and the CHEMTECH Emergency and Contingency Plans. It is the responsibility of various company personnel to assist in implementing the different aspects of the Plan. These include: Laboratory Coordinator, Technical Director, Operations Manager, Department Managers and Supervisors.

18. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QC

18.1 BFB-MS Tuning Check Compounds

- 18.1.1 Spectrum produced must meet the criteria outlined in Table 2.
- 18.2 <u>Initial Calibration</u>
 - 18.2.1 All Criteria in section 13.2 must be satisfied.
- 18.3 <u>Continuing Calibration</u>
 - 18.3.1 The %D for each analytes & surrogates must be ≤20% for opening Continuous Calibration for 8260B/C and DOD. For DOD, End Continuous Calibration % D for each analytes and surrogates must be ≤50%.
 - 18.3.2 The SPCC and CCC criteria must be met for Method 8260B.
 - 18.3.3 1,4-Dioxane must meet 0.05 minimum RRF and < 50% D.
 - 18.3.3 If the analyte is failing biased high, with no positive hits in the samples analyzed under this calibration check sample for that analyte, then no further corrective action is taken. The failure is documented in the case narrative/ non-conformance.
- 18.4 <u>Method Blank</u>
 - 18.4.1 No analyte should be present in the blank at a concentration greater than the reporting limit except for Acetone and Methylene Chloride, which can be present up to 2X the reporting limit.
 - 18.4.2 If the analyte is present greater than the above criteria, all associated sample results must be flagged with the B qualifier.
 - 18.4.3 For DoD work No analyte must be detected at >1/2RL and >1/10 the amount measured in any sample or >1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes should be detected at > RL.
- 18.5 <u>Surrogates</u>
 - 18.5.1 Surrogate recovery limits must be within the limits specified for each matrix.
- 18.6 MS/MSD and Blank Spike
 - 18.6.1 The %recovery for all analytes must be within control limits.
 - 18.6.2 2-Chloroethylvinyl ether recovery may not meet criteria for water MS/MSD due to acidification of the sample for preservation. Mention this in the case narrative/non-conformance.
- 18.7 Internal Standard
 - 18.7.1 Monitor all samples, blanks, and spikes for retention time shift and fluctuation of extracted ion areas.
 - 18.7.2 Make sure that the GC retention time is within ± 30 seconds of the corresponding internal standard in the midpoint standard of the initial calibration.
 - 18.7.3 Verify that the areas of the internal standard do not change by more than a factor of 2 (-50% to +100%) from the areas in the midpoint standard of the initial calibration. On days when ICAL is not performed, the daily initial CCV can be used.
 - 18.7.4 Monitor all continuing calibration verification standards for retention time shift and fluctuation of extracted ion areas.
 - 18.7.5 Verify that the retention time is within ± 30 seconds of the corresponding internal standard in of the initial calibration.

18.7.6 Verify that the areas of the internal standard do not change by more than a factor of 2 (-50% to +100%) from the areas of the corresponding internal standard in the midpoint standard of the initial calibration. On days when ICAL is not performed, the daily initial CCV can be used.

18.8 Initial Calibration Verification

- 18.8.1 The ICV standard recoveries must be within the 70-130% range. Up to 10% of the compounds may be allowed to fail marginally.
- 18.8.2 For DoD work, all project analytes must be within $\pm 20\%$ of true value.

18.9 <u>Limit of Detection</u>

18.9.1 All analytes spiked should be positively identified.

18.10 Limit of Quantitation

18.10.1 Analysis must meet the acceptance criteria for the laboratory control sample.

19. CORRECTIVE ACTION FOR OUT-OF-CONTROL DATA

- 19.1 BFB-MS Tuning Check Compounds
 - 19.1.1 Rerun the BFB tune.
 - 19.1.2 If it still fails, re-tune the instrument and run again. If it still fails, clean the source.
- 19.2 <u>Initial Calibration</u>
 - 19.2.1 After the system performance check has met the criteria, CCCs are used to check the validity of the initial calibration.
 - 19.2.2 If the QC criterion is not met for any CCC, take a corrective action prior to sample analysis.
 - 19.2.3 If the problem cannot be corrected, generate a new calibration or report the failures in the case narrative and/or non-conformance sheet.

19.3 <u>Continuing Calibration</u>

- 19.3.1 If the criteria for continuing calibration are not met, rerun the continuing calibration.
- 19.3.2 If the continuing calibration fails again, acquire a new initial calibration or report the failures in the case narrative and/or non-conformance sheet.

19.4 <u>Method Blank</u>

- 19.4.1 Rerun the method blank if it fails the first time.
- 19.4.2 If it fails second time, evaluate the system and contact the department supervisor.
- 19.4.3 For DoD work Reprocess the failing blank with the associated samples in a subsequent preparation batch, except when the sample analysis results in a non-detect.
- 19.5 <u>Surrogates</u>
 - 19.5.1 Should any injection fail to meet the required limits, reanalyze the sample.
 - 19.5.2 If the second injection is acceptable, report only the second set of data.
 - 19.5.3 If the second injection also fails, report both sets of data.
 - 19.5.4 In the case of high level soils, first reanalyze the original methanol extract.
 - 19.5.5 If this fails, re-extract the sample, then analyze the new extract.
- 19.6 <u>Laboratory Control Sample</u>

19.6.1 If recovery of the LCS is outside the control limits, re-analyze the LCS.

- 19.6.2 If the recovery is above the control limits, and the affected compound is not detected above the LOQ in any associated client sample, the data may be reported with a "Q" flag applied to the compound and the failure documented in the case narrative/ non-conformance.
- 19.6.3 If the recovery is below the control limits, or the affected compound is detected above the LOQ in any associated client sample, the LCS and affected samples must be re-extracted.
 - 19.6.3.1 If the samples cannot be re-extracted, the results must be reported with a "Q" flag, and the failure documented in the case narrative/ non-conformance.
 - 19.6.3.2 If the data will be reported associated with the failed LCS, for DOD projects, the client must be informed of the failure and consulted for corrective actions.
- 19.7 <u>MS/MSD</u>
 - 19.7.1 No corrective action is required if limits are exceeded for MS/MSD analysis but the blank spike meets the criteria. However if more than 50% of the recoveries or 50% of the %RPD's are out, find the cause of this and reanalyze one or both of the spikes.
- 19.8 Internal Standards
 - 19.8.1 If any sample fails to meet criteria, re-analyze the sample.
 - 19.8.2 If the reanalysis is within limits, then report only the second set of data.
 - 19.8.3 If the re-analysis also fails, report both sets of data.
 - 19.8.4 If the continuing calibration verification standard fails criteria, a new initial calibration needs to be performed.
- 19.9 <u>Limit of Detection</u>
 - 19.9.1 If LOD verification fails, then repeat the detection limit determination and LOD verification at a higher concentration and set the LOD at the higher concentration.
- 19.10 Limit of Quantitation
 - 19.10.1 Reevaluate the LOD and the LOQ.
- 19.11 Initial calibration verification (ICV)

19.11.1 If criteria are not met, reanalyze a new initial calibration curve.

20. Contingencies for handling out-of-control or unacceptable data

- 20.1 Following are the result qualifiers used for out-of-control and unacceptable data:
 - U: Indicates the compound was analyzed but not detected.
 - J: Indicates an estimated value, the result reported is below the initial calibrations lowest point.
 - **B**: Indicates the analytes were found in the blank as well as the sample.
 - E: Indicates the analyte concentrate exceeds the calibrated range of the GC instrument.

• **D**: Indicates all compounds identified in an analysis at a secondary dilution factor.

- N: Indicates presumptive evidence of a compound. This is used for all non-target results where identification is made.
- Q: Indicates a QC (LCS) failure associated with the compound

- 20.2 Issue a corrective action form any time there is a deviation from the SOP or the client requirements are not met.
- 20.3 If a sample or extract is damaged, broken, or spilled, contact the project manager and issue a corrective action.
- 20.4 For more details regarding corrective action procedure, please refer to Corrective Action Report SOP.
- 20.5 For **DOD** work- use DOD QSM flagging criteria.

21. WASTE MANAGEMENT

21.1 Keep samples for 30 days after analysis and dispose them off according to the procedures explained in the SOP for waste disposal.

22. **REFERENCES**

- 22.1 USEPA Test Methods for Evaluating Solid Wastes, SW-846, Method 5030B-Purge and Trap for Aqueous Samples, Revision 2, December 1996.
- 22.2 USEPA Test Methods for Evaluating Solid Wastes, SW-846, Method 5030C-Purge and Trap for Aqueous Samples, Revision 3, May 2003.
- 22.3 USEPA Test Methods for Evaluating Solid Wastes, SW-846, Method 5035A Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples. Revision 1, July 2002.
- 22.4 USEPA Test Methods for Evaluating Solid Wastes, SW-846, Method 8000B Determinative Chromatographic Separations. Revision 2, December 1996
- 22.5 USEPA Test Methods for Evaluating Solid Wastes, SW-846, Method 8260B Volatile Organic Compounds by GC/MS, Revision 2, December 1996.
- 22.6 USEPA Test Methods for Evaluating Solid Wastes, SW-846, Method 8260C Volatile Organic Compounds by GC/MS, Revision 3, August 2006.
- 22.7 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013 & Version 5.1 January 2017.

23. LIST OF TABLES/ATTACHMENTS

- 23.1 Table 1: Target Compound List
- 23.2 Table 2: BFB Tuning Criteria
- 23.3 Table 3: Characteristic Ions for Volatile Target Compounds
- 23.4 Table 4: QC/Sample Preparation
- 23.5 Table 5: Purging Conditions
- 23.6 Table 6 & 7: Purge & Trap system
- 23.7 Table 8: Instrument Specifications
- 23.8 Table 9: Instrument Temperature and Flow Conditions
- 23.9 Table 10: Instrument Temperature Conditions (or equivalent)
- 23.11 Table 11: Standards and Solutions (or equivalent)
- 23.12 Table 12: Water working standard preparation
- 23.13 Table 13: Soil working standard preparation

IAKGET COMPOUND LIST				
COMPOUND NAME	COMPOUND NAME			
1,1,1,2-Tetrachloroethane	Carbon disulfide			
1,1,1-Trichloroethane	Carbon Tetrachloride			
1,1,2,2-Tetrachloroethane	Chlorobenzene			
1,1,2-Trichloroethane	Chloroethane			
1,1,2-Trichlorotrifluoroethane	Chloroform			
1,1-Dichloroethane	Chloromethane			
1,1-Dichloroethene	cis-1,2-Dichloroethene			
1,1-Dichloropropene	cis-1,3-Dichloropropene			
1,2,3-Trichlorobenzene	Cyclohexane			
1,2,3-Trichloropropane	Dibromochloromethane			
1,2,4-Trichlorobenzene	Hexachloroethane			
1,2,4-Trimethylbenzene	Dibromomethane			
1,2-Dibromo-3-Chloropropane	Dichlorodifluoromethane			
1,2-Dibromoethane	Ethyl Benzene			
1,2-Dichlorobenzene	Hexachlorobutadiene			
1,2-Dichloroethane	Isopropylbenzene			
1,2-Dichloropropane	m/p-Xylenes			
1,3,5-Trimethylbenzene	Methyl Acetate			
1,3-Dichlorobenzene	Methyl tert-butyl Ether			
1,3-Dichloropropane	Methylcyclohexane			
1,4-Dichlorobenzene	Methylene Chloride			
2,2-Dichloropropane	Naphthalene			
2-Butanone	n-Butylbenzene			
2-Chloroethyl vinyl ether	N-propylbenzene			
2-Chlorotoluene	o-Xylene			
2-Hexanone	p-Isopropyltoluene			
Diethyl ether	Sec-butylbenzene			
4-Chlorotoluene	Styrene			
4-Methyl-2-Pentanone	t-1,3-Dichloropropene			
Acetone	Tert butyl alcohol			
Acrolein	tert-Butylbenzene			
Acrylonitrile	Tetrachloroethene			
Benzene	Toluene			
Bromobenzene	trans-1,2-Dichloroethene			
Bromochloromethane	Trichloroethene			
Bromodichloromethane	Trichlorofluoromethane			
Bromoform	Vinyl Acetate			
Bromomethane	Vinyl chloride			
Allyl chloride	Ethyl acetate			
Ethyl methacrylate	Isobutyl alcohol			
Methacrylonitrile	1,4-Dioxane			

TABLE 1TARGET COMPOUND LIST

TABLE 2BFB TUNING CRITERIA

Mass	Ion Abundance Criteria		
50	15.0-40.0 percent of mass 95		
75	30.0-60.0 percent of mass 95		
95	Base peak, 100 percent relative abundance		
96	5.0-9.0 percent of mass 95		
173	Lass than 2.0 percent of mass 174		
174	Greater than 50.0 percent of mass 95		
175	5.0-9.0 percent of mass 174		
176	95.0-101.0 percent of mass 174		
177	5.0-9.0 percent of mass 176		

TABLE 3

CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS

Analyte	Primary Ion*	Secondary Ion(s)	Internal Standard for Quantitation	
Dichlorodifluoromethane	85	87	IS1	
Chloromethane	50	52	IS1	
Vinyl chloride	62	64	IS1	
Bromomethane	94	96	IS1	
Chloroethane	64	66	IS1	
Trichlorofluoromethane	151	101,153	IS1	
1,1-Dichloroethene	96	61, 63	IS1	
Carbon disulfide	76	78	IS1	
Methylene Chloride	84	49, 86	IS1	
Acetone	58	43	IS1	
t-Butyl alcohol	59	74	IS1	
trans-1,2-Dichloroethene	96	61, 98	IS1	
Acrolein	56	55,58	IS1	
Acrylonitrile	53	40,39	IS1	
t-Butyl methyl ether	73	57	IS1	
1,1-Dichloroethane	63	65, 83	IS1	
2-Butanone	72	43	IS1	
2,2-Dichloropropane	77	97	IS1	
cis-1,2-Dichloroethene	96	61, 98	IS1	
Bromochloromethane	128	49,130	IS1	
Chloroform	83	85	IS1	
1,1,1-Trichloroethane	97	99, 61	IS1	
Carbon tetrachloride	117	119	IS2	
1,1-Dichloropropene	75	110,77	IS2	
Benzene	78	76,77	IS2	
1,2-Dichloroethane	62	98	IS2	
Trichloroethene	95	97, 130, 132	IS2	
1,2-Dichloropropane	63	112	IS2	
Bromodichloromethane	83	85, 127	IS2	
Dibromomethane	174	95,174	IS2	
cis-1,3-Dichloropropene	75	77, 39	IS2	
Vinyl Acetate	43	86	IS2	
trans-1,3-	75	77, 39	IS2	
Dichloropropene				
1,1,2-Trichloroethane	83	97, 85	IS3	

Methyl cyclohexane

CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS Analyte Primary Secondary Internal Standard						
Analyte	I I IIIIai y Ion*	Ion(s)	Quantitation			
2-Chloroethyl vinyl ether	63	65,106	IS3			
1,3-Dichloropropane	76	78	IS3			
Dibromochloromethane	129	127	IS3			
Bromoform	173	175, 254	IS3			
4-Methyl-2-pentanone	100	43, 85	IS3			
Toluene	92	91	IS3			
Tetrachloroethene	164	129, 131, 166	IS3			
Isopropylbenzene	101	120	IS3			
1,1,2,2-Tetrachloroethane	83	131, 85	IS3			
2-Hexanone	43	58, 57, 100	IS3			
1,2-Dibromoethane	107	109,188	IS3			
Chlorobenzene	112	77, 114	IS3			
1,1,1,2-Tetrachloroethane	131	133,119	IS3			
Ethylbenzene	91	106	IS3			
o- Xylene	106	91	IS3			
m+p- Xylene	106	91	IS3			
Styrene	100	78	IS3			
Bromobenzene	156	77,158	IS4			
1,2,3-Trichloropropane	75	77	IS4			
n-Propylbenzene	91	120	IS4			
2-Chlorotoluene	91	126	IS4			
1,3,5-Trimethylbenzene	105	120	IS4			
4-Chlorotoluene	91	126	IS4			
tert-Butylbenzene	119	91,134	IS4			
1,2,4-Trimethylbenzene	105	120	IS4			
sec-Butylbenzene	105	134	IS4			
p-Isopropyltoluene	119	134,91	IS4			
1,3-Dichlorobenzene	146	111,148	IS4			
1,4-Dichlorobenzene	146	111,148	IS4			
n-Butylbenzene	91	92	IS4			
1,2-Dichlorobenzene	146	111,148	IS4			
1,2-Dibromo-3-Chloropropane	75	155,157	IS4			
1,2,4-Trichlorobenzene	180	182,145	IS4			
Hexachlorobutadiene	225	223,227	IS4			
Naphthalene	128		IS4			
1,2,3-Trichlorobenzene	180	182,145	IS4			
Cyclohexane	56	69, 84	IS1			
Methyl acetate	43	74	IS1			
	02	50.00	102			

83

59, 98

IS2

CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS							
Analyte	Primary	Secondary	Internal Standard for				
	Ion*	Ion(s)	Quantitation				
Trichlorotrifluoroethane	101	103	IS1				
Diethyl ether	74	45	IS1				
Hexachloroethane	117	201	IS4				
Allyl chloride	41	39, 76	IS1				
Ethyl acetate	43	61, 70	IS1				
Ethyl methacrylate	69	41, 39	IS2				
Isobutyl alcohol	43	41, 42	IS2				
Methacrylonitrile	41	39, 67	IS2				
1,4-Dioxane	88	43, 58	IS2				
Surrogate Com	pounds (Syster	n Monitoring Co	mpounds)				
Dibromofluoromethane 113 IS							
1,2-Dichloroethane-d4	65	102	IS2				
Toluene-d8	98	70, 100	IS3				
4-Bromofluorobenzene	95	174, 176	IS4				
Internal Standards							
Pentafluorobenzene (IS 1)) 168		IS1				
1,4-Difluorobenzene (IS 2)	114	68, 88	IS2				
Chlorobenzene-d5 (IS 3)	117	82, 119	IS3				
1,4-Dichlorobenzene-d4 (IS 4)	152	115, 150	IS4				

TABLE 3 CHARACTERISTIC IONS FOR VOLATILE TARGET COMPOUNDS

*The primary ion should be used unless interferences are present, in which case, a secondary ion may be used.

**m/z 43 is used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

QC/Samples	Matrix	Internal Std (ul) 50ppm	Surrogate (ul) 50ppm	MeOH Added (ul)	Final volume (ml)
50 ppb CCC	Water	40.0	40.0	NA	40mL
20 ppb CCC	Water	40.0	40.0	NA	40mL
Method Blank	Water	40.0	40.0	NA	40mL
High Level Soil Blank	Water	800.0	800.0	100	40mL
Blank Spike/MS/MSD	Water	40.0	40.0	NA	40mL
Sample	Water	40.0	40.0	NA	40mL
50 ppb CCC	Soil	5.0	5.0	NA	5mL
20 ppb CCC	Soil	5.0	5.0	NA	5mL
Method Blank	Soil	5.0	5.0	NA	5mL
Blank Spike/MS/MSD	Soil	5.0	5.0	NA	5mL
Sample	Soil	5.0	5.0	NA	5mL

 TABLE 4

 QC/Sample Preparation (concentrations are subject to change)

Note: Follow the above table if surrogate and internal standard solutions are added manually. If surrogate and internal standard solutions are added by the auto sampler, then the same stock solution is used to add these solutions using the auto sampler loop. The same technique and amount of solution is added to all calibration standards and samples following an initial calibration curve.

TABLE 5Purging Conditions (subject to change)

Instrument Name	Purge Flow	Purge Temp.	Purge Time	Dry Purge Time	Desorb Temp.
MSVOAD	40mL/min	35°C	11 Min.	3 Min	250°C
MSVOAF	40 mL/min	35°C	11 Min.	3 Min	190°C
MSVOAH	40 mL/min	35°C	11 Min.	1 Min.	190°C
MSVOAK	40 mL/min	35°C	11 Min.	0 Min.	190°C
MSVOAI	40 mL/min	35°C	11 Min.	0.5Min.	190°C

Instrument Name	Auto-sampler	Concentrator	Sample Heater
MSVOAD	OI-4552 Arcon	Tekmar 2000	Yes and Stirrer
MSVOAF	OI-4552 Arcon	OI 4660 Eclipse	Yes and Stirrer
MSVOAH	Arcon Dynatech	OI 4660 Eclipse	
MSVOA K	OI 4552 Arcon	OI 4560	Yes and Stirrer
MSVOA I	EST Arcon	OI 4660 Eclipse	Yes and Stirrer

TABLE 6Purge & Trap System (subject to change)

TABLE 7Purge & Trap system (subject to change)

Instrument Name	Desorb Time	Bake Temp.	Bake Time.	2016 Line and Valve Temp.
MSVOAD	6 Min.	260°C	10 Min.	110°C
MSVOAF	2 Min.	210°C	6 Min.	110°C
MSVOAH	2 Min.	210°C	6 Min.	110°C
MSVOAK	3 Min.	210°C	5 Min.	110°C
MSVOAI	2 Min.	210°C	5 Min.	110°C

Trap = Vocarb 3000 from Supelco Catalog # 21066-U and OI#10 Trap or equivalent

TABLE 8
Instrument Specifications (subject to change)

Instrument Name	Column	Supplier	Catalog #	Model of GC	Model of MS
MSVOAD	RTX-VMS 20M x	Restek		HP5890	HP5971
	0.18mm ID x 1um		49914		
	film thickness				
MSVOAF	RTX-VMS 20M x	Restek		HP5890	HP5972
	0.18mm ID x 1um		49914		
	film thickness				
MSVOAH	RTX-VMS 20M x	Restek		HP5890	HP5971
	0.18mm ID x 1um		49914		
	film thickness				
MSVOAK	RTX-VMS 20M x	Restek		HP5890	HP5972
	0.18mm ID x 1um		49914		
	film thickness				
MSVOAI	RTX-VMS 60M x	Restek		HP5890	HP5971
	0.25mm ID x		19916		
	1.4um film		17710		
	thickness				

TABLE 9 Instrument Temperature and Flow Conditions (or equivalent) (subject to change)

Instrument Name	Injector Temperature	Detector B Temperature	Carrier Flow
	-	Mass Spectrometer	
MSVOAD	220°C	280°C	30mL/Minute
MSVOAF	220°C	260°C	30mL/Minute
MSVOAH	220°C	280°C	30mL/Minute
MSVOAK	220°C	280°C	30mL/Minute
MSVOAI	220°C	280°C	30mL/Minute

TABLE 10
Instrument Temperature Conditions (or equivalent) (subject to change)

Instrument Name	Initial Temperature	Initial Hold	Temperature Ramp	Final Temperature	Final Hold
MSVOAD	50°C	4 Minutes	18 °C /Minute Ramp A =	100°C Final A =	0 Minute Final A =
MSVOAF	42°C	1 Minutes	$25 ^{\circ}C$ /Minute 10 $^{\circ}C$ /Minute Ramp A =	$\frac{210 \text{ °C}}{100 \text{ °C}}$ Final A =	5.0 Minutes 0 Minute Final A =
MSVOAH	40°C	2 Minutes	$25 ^{\circ}C /Minute$ $10 ^{\circ}C /Minute$ $Ramp A =$ $25 ^{\circ}C /Minute$	$ 220 ^{\circ}C \\ 100^{\circ}C \\ Final A = \\ 220 ^{\circ}C $	3.0 Minutes 2 Minutes Final A = 2.20
MSVOAK	42°C	1 Minutes	$10 ^{\circ}C /Minute$ Ramp A = $25 ^{\circ}C /Minute$	100°C Final A = 220 °C	Minutes 0 Minute Final A = 3.0 Minutes
MSVOAI	42°C	1 Minutes	$\frac{10 \text{ °C /Minute}}{\text{Ramp A} =}$ $\frac{25 \text{ °C /Minute}}{\text{Minute}}$	$100^{\circ}C$ Final A = 220 °C	0 Minute Final A = 7.0 Minutes

TABLE 11Standards and Solutions (or equivalent)

Standard Name	Supplier	Catalog Number	Concentration of stock	Preparation Details	Final Concentration of working solution
8260 Internal Standard	Restek	555581	25,000 ug/mL	20uL into 10mL Volumetric QS DI water	50ug/mL
Arcon 8260 Internal Standard	Restek	555581	25,000 ug/mL	100uL into 10mL Volumetric QS DI water	250ug/mL
Arcon 8260 Surrogate Standard	Restek	555582	25,000 ug/mL	100uL into 100mL Volumetric QS DI water	250ug/mL
8260 Surrogate Standard	Restek	555582	25,000 ug/mL	20uL into 100mL Volumetric QS DI water	50ug/mL
8260 Calibration Working STD Acrolein only	Absolute	91980	5000ug/mL	4.0mL into 25mL volumetric QS DI water	160ug/mL
8260 Calibration working STD Bromochloromethane only	Restek	30225	2,000 ug/mL	4.0mL in 50mL Volumetric QS DI water	160.0ug/mL
BFB	Restek	30067	2500ug/mL	250ul into 25mL volumetric QS DI water	25ug/mL
8260 Calibration working Stock Standard 160ppm	Restek	555408 555406 555407 30006 30489 30042 30499 30225 556166 30470	8000ug/mL 2000ug/mL 10,000ug/mL 5,000ug/mL 2000ug/mL 2000ug/mL 2000ug/mL 2000- 40,000ug/mL 50,000ug/mL	1000uL 800uL 800uL 1600uL 800uL 800uL 800uL 800uL 800uL 160uL in 10mL Volumetric QS DI water	160ug/mL for most components
8260 Calibration working Stock Standard 100ppm			160ug/mL	625uL 160ug/mL Stock solution in 325mL DI water	100ppm
8260 Calibration working Stock Standard 20ppm			160ug/mL	125uL 160ug/mL Stock solution in 875mL DI water	20ppm
8260 Calibration working Stock Standard 10ppm			160ug/mL	62.5uL 160ug/mL Stock solution in 937.5mL DI water	10ppm

Water Working Standard Level	Stock Solution Std. Concentration (ppm)	Volume used(ul)	Surrogate (ul) 50ppm	Internal std (ul) 250ppm	Final volume (ml)
1 ppb	10ppm	4.0uL	0.8ul	8.0ul	40mL Vial
5 ppb	20ppm	10.0uL	4.0u1	8.0u1	40mL Vial
10 ppb	20ppm	20.0uL	8.0ul	8.0u1	40mL Vial
20 ppb	160ppm	5.0uL	16.0ul	8.0u1	40mL Vial
50 ppb	160ppm	12.5uL	40.0ul	8.0u1	40mL Vial
100 ppb	160ppm	25.0uL	80.0ul	8.0u1	40mL Vial
150 ppb	160ppm	37.5uL	120.0ul	8.0u1	40mL Vial
200 ppb	160ppm	50.0uL	160.0ul	8.0ul	40mL Vial
50 ppb ICV	160ppm	12.5uL	40.0ul	8.0ul	40mL Vial

TABLE 12Water Working Standard Preparation

TABLE 13Soil Working Standard Preparation

Soil Working Standard Level	Stock Solution Std. Concentration	Volume used from stock	Surrogate (ul) 50ppm	Internal std (ul)	Final volume (ml)
	(ppm)	(ul)		50ppm	
5 ppb	10ppm	2.5ul	0.5ul	5ul	5mL Vial
10 ppb	10ppm	5.0ul	1.0ul	5ul	5mL Vial
20 ppb	20ppm	5.0ul	2.0ul	5ul	5mL Vial
50 ppb	100ppm	2.5ul	5.0ul	5ul	5mL Vial
75 ppb	100ppm	3.75ul	7.5ul	5ul	5mL Vial
100 ppb	100ppm	5.0ul	10.0ul	5ul	5mL Vial
200 ppb	100ppm	10.0ul	20.0ul	5ul	5mL Vial
50 ppb ICV	100ppm	2.5ul	5.0ul	5ul	5mL Vial

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READ RECEIPT

Employee Name: _____

Department:

_____M8260B/C-SWGCMSVOA_____

Method or Document Read (Include Title, Number, Revision, as applicable)

Employee Statement: I have read and understand the information in the above mentioned method or document.

Employee Signature

Supervisory Statement: I have reviewed this document or method with the employee.

Supervisory Signature

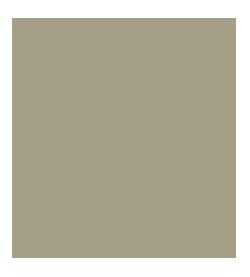
Note: This receipt is to be returned to the Quality Assurance Department for incorporation into employee training record files. If you have questions or would like to review your train record files, please see QA.

Date

Date



L4 - Sample Preparation for Toxicity Characteristics Leachate Procedure





SAMPLE PREPARATION FOR TOXICITY CHARACTERISTICS LEACHATE PROCEDURE

1. Test Method

1.1 Sample preparation for toxicity characteristics leachate procedure by Method SW 846-1311.

2. Applicable Matrices

- 2.1 Liquid, solid and multiphase waste
- 3. Method Detection Limit
 - 3.1 NA

4. Scope and Application

- 4.1 This method determines the mobility of organic and inorganic analytes present in liquid, solid and multiphase wastes.
- 4.2 Total analysis of a sample that demonstrates individual analytes not present or substantially below the regulatory level need to be determined by TCLP.
- 4.3 If the analysis for any TCLP extract exceeds the regulatory level, it may not be necessary to analyze the remaining fractions.
- 4.4 If the analysis of the extract from a bottle extractor shows analyte levels greater than the regulatory level, the ZHE extraction may not be necessary.

5. Summary

- 5.1 For liquid wastes, those containing less than 0.5% dry solid material, the waste after filtration through a 0.6 to 0.8-micron glass fiber filter, is defined as the TCLP extract.
- 5.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. A special extractor vessel is used when testing for volatile analytes. Following extraction the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8-micron glass fiber filter.
- 5.3 For multiphase samples the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together, if compatible. If not compatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

6. **Definitions**

6.1 <u>Analyst</u>: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

- 6.2 <u>Batch</u>: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents.
 - 6.2.1 <u>Preparation Batch</u>: is composed of one to 20 environmental samples of the same matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours.
 - 6.2.2 <u>Analytical Batch</u>: is composed of prepared environmental samples (extracts, digestates or concentrates), which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
- 6.3 <u>Blank</u>: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis the blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.
- 6.4 <u>Corrective Action</u>: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.
- 6.5 <u>Duplicate Analyses</u>: The analysis or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
- 6.6 <u>Holding Times (Maximum Allowable Holding Times)</u>: The maximum times that samples may be held prior to analysis and still be considered valid or not compromised.
- 6.7 <u>Method Blank</u>: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations tat impact the analytical results for sample analyses.
- 6.8 <u>Preservation</u>: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.
- 6.9 <u>Pure Reagent Water</u>: Water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method.
- 6.10 <u>Standard</u>: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies.
- 6.11 <u>Standard Operating Procedures (SOPs)</u>: A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive task.
- 6.12 <u>Test Method</u>: An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP.

7. Interferences

7.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

8. Safety

- 8.1 Wear appropriate safety clothing and eye protection.
- 8.2 Use protective gloves when handling corrosive chemicals.
- 8.3 Always use safety carts when transporting large bottles of chemicals.
- 8.4 Read material safety data sheet (MSDS) for the chemicals used in the laboratory for the identity of the ingredients, the physical and chemical characteristics of the substance, the physical hazards, and safe handling and safety precautions.

9. Equipment and Supplies

- 9.1 Rotary agitator, 30 ± 2 rpm, for 2 & 3
- 9.2 Zero-headspace Extraction Vessels (ZHE), internal volume of 500-600mL, for volatile.
- 9.3 Bottle Extraction Vessels, plastic for metals, borosilicate glass for semivolatile organic
- 9.4 Pressure Filter, (to 50 psi), stainless steel
- 9.5 Filters for 2 and 4, borosilicate glass fiber, containing no binder materials, pore sizes 0.6 to 0.8 um.

Extraction Fluid # 2

- 9.6 Gas tight syringe to collect extracts form ZHE
- 9.7 Mettler balance AE100
- 9.8 Beakers- 500 ml or Erlenmeyer Flask

10. Reagents and Standards

- 10.1 $1.0 \text{ N HNO}_3 = 64 \text{ ml concentrated HNO}_3 / 1000 \text{ ml}$
- 10.2 1.0 N HCl = 83 ml concentrated HCl / 1000 ml
- 10.3 1.0 N NaOH = 40.0 g NaOH pellets / 1000 ml

Extraction Fluid # 1

500 ml DI water	500 ml DI water
5.7 ml Glacial Acetic Acid	5.7 ml Glacial Acetic Acid
64.3 ml 1.0 N NaOH solution	Dilute to 1000 ml
Dilute to 1 liter	(pH should be 2.88 +/- 0.05)
(pH should be 4.93 +/-0.05)	

11. Sample Collection, Shipment, and Storage

11.1 Refrigerate at 4^oC until extraction. Do not add preservatives prior to extraction. If organic are to be analyzed for, use glass containers with teflon lined septa. Preserve extracts according to the guidance given in the individual; analytical methods. The general chemistry department will immediately acidify extracts for metallic aanalytes with nitric acid to pH <2 unless precipitation occurs.</p>

SAMPLE MAXIMUM HOLDING TIMES (days) For Non-CLP Methods:

Analysis Type	From Field Collection to 1311 Extraction	From 1311 Extraction to Preparative Extraction	From Preparative Extraction to Determinative Analysis
Volatile Soil	<mark>14</mark>	NA	<mark>14</mark>
Volatile Water	<mark>14</mark>	<mark>NA</mark>	<mark>14</mark>
<mark>Semi-Volatile</mark> Soil	<mark>14</mark>	7	<mark>40</mark>
<mark>Semi-Volatile</mark> Water	<mark>14</mark>	<mark>7</mark>	<mark>40</mark>
Pesticide Soil	<mark>14</mark>	<mark>7</mark>	<mark>40</mark>
Pesticide Water	<mark>14</mark>	<mark>7</mark>	<mark>40</mark>
Mercury Soil	<mark>28</mark>	<mark>NA</mark>	<mark>28</mark>
Mercury Water	<mark>28</mark>	NA	<mark>28</mark>
Cyanide Soil	<mark>14</mark>	NA	<mark>14</mark>
Cyanide Water	<mark>14</mark>	NA	<mark>14</mark>
Metals Soil (except Mercury)	180	NA	180
Metals Water (except Mercury)	<mark>180</mark>	NA	<mark>180</mark>

SAMPLE MAXIMUM HOLDING TIMES (days) For ISM/SOM CLP methods:

Analysis Type	From VTSR to 1311 Extraction	From 1311 Extraction to Preparative Extraction	From Preparative Extraction to Determinative Analysis
Volatile Soil	<mark>10</mark>	<mark>NA</mark>	<mark>7</mark>
Volatile Water	<mark>5</mark>	<mark>NA</mark>	<mark>7</mark>
Semi-Volatile	<mark>10</mark>	<mark>7</mark>	<mark>40</mark>
<mark>Soil</mark>			
<mark>Semi-Volatile</mark> Water	<mark>5</mark>	7	<mark>40</mark>
Pesticide Soil	<mark>10</mark>	<mark>7</mark>	<mark>40</mark>
Pesticide Water	<mark>5</mark>	<mark>7</mark>	<mark>40</mark>
Mercury Soil	<mark>26</mark>	<mark>26</mark>	<mark>26</mark>
Mercury Water	<mark>26</mark>	<mark>26</mark>	<mark>26</mark>
Cyanide Soil	<mark>12</mark>	<mark>12</mark>	<mark>12</mark>
Cyanide Water	<mark>12</mark>	<mark>12</mark>	<mark>12</mark>

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SOP ID: M1311-TCLP Revision #10

QA Control # A2040044

Effective Date: June 23, 2017 Page 5 of 14

Metals Soil	<mark>180</mark>	<mark>180</mark>	<mark>180</mark>	
(except Mercury)				
Metals Water	<mark>180</mark>	<mark>180</mark>	<mark>180</mark>	
(except Mercury)				
VTSR= Validated Time of Sample Receipt				

validated Time of Sample Receipt

12. **Quality Control**

Laboratory Reagent Blank 12.1

12.1.1 Analyze a minimum of one blank (using the same extraction fluid as used for the samples) for every 20 extractions that have been conducted in an extraction vessel.

14.3.2 Rotate ZHE extractor every time which is used for Blank Extraction.

12.2 Spike Sample

- 12.2.1 Perform a matrix spike for every waste type. A minimum of one matrix spike must be analyzed for each batch.
- 12.2.2 Matrix spikes are added after filtration of TCLP extract and before preservation. Matrix spikes are not added prior to TCLP extraction of the sample.

13. **Calibration and Standardization**

13.1 NA

14. Procedure

- 14.1 Procedure for all tests other than Volatile
 - 14.1.1 Room temperature must be constant $(21-25^{\circ}C)$. Record the temperature in the temperature log twice a day. If the temperature is outside control limits contact the supervisor.
 - 14.1.2 If the sample is a soil or other solid with no free liquid, proceed to section 14.2
 - 14.1.3 If the sample is a liquid, or has a phase which appears to be fluid, proceed to section 14.3.
 - 14.1.4 Determine whether or not the waste requires particle size reduction. Any particles, which are too large to pass through a 9.5 mm sieve, should be crushed to a small enough size to pass through the sieve.
 - 14.1.5 Take a 5.0 gram subsample of the waste and place it into a 500 ml beaker or Erlenmeyer flask. Add 96.5-ml DI water to the beaker, cover with a watch glass and stir for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is less than 5.0 use extraction fluid #1. If the pH is greater than 5.0, add 3.5 ml 1.0 N HCl, mix briefly and heat to 50° C.
 - 14.1.6 Hold at 50° C for ten minutes, cool to room temperature, and record the pH. If the pH is less than 5.0, use extraction fluid #1; if the pH is greater than 5.0, use extraction fluid # 2. (pHs should be taken using multirange pH paper since only a less than or greater than 5.0 result needs to be

measured. The probe may be damaged from the type of samples routinely encountered in TCLP analysis.)

- 14.1.7 Weigh a 100.0 gram aliquot of sample into a 2000 ml plastic jar and combine it with 2000 ml of the appropriate extraction fluid. If sufficient sample is not available, weigh as much as is available and combine the sample with 20X the amount of extraction fluid. If semivolatiles organic are to be determined a glass jar must be used, but a plastic jar is suitable for metals only.
- 14.1.8 Place the sample into the rotary agitator and rotate for 18 +/- 2 hours. Be sure to counterbalance the agitator when odd numbers of samples are extracted.
- 14.1.9 When the rotation period is completed, remove the sample from the agitator and allow it to settle. Check and record the pH. Samples in which the solids do not easily separate may be centrifuged. Do not use prefilters to aid in filtration. Only sufficient sample to support the analysis needs to be filtered (500 mls). In cases where the filtrate may need to be combined with a previously separated phase, filter the entire sample.
- 14.1.10After insertion into the filtration device, rinse all the filters with 1000 ml 1.0 N HNO3 followed by two 1000-ml volumes of DI water. Filter the extract or prefiltered extract through a 0.8u glass fiber filter. The filtrate can now be transferred to sample bottles appropriate for the required analysis. The general chemistry department will immediately acidify extracts for metallic analytes with nitric acid to pH <2 unless precipitation occurs. Store samples at 4^{0} C until the time of analysis.
- 14.2 Assemble the pressure filtration device and place a pre-weighed filter on the support screen. Record the weight of the filter. Rinse the filter with 1000 ml 1.0 N HNO₃ followed by two 1000 ml volumes of DI water.
 - 14.2.1 Weigh out a 100-gram subsample of waste and add it to the filtration device. Apply pressure gradually and increase to 10 psi until air or pressurizing gas moves through the filter. If this point is reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. NOTE: Instantaneous application of high pressure can degrade the filter or when the liquid flow has ceased at 50 psi for a period of 2 minutes, stop the filtration. If the sample fails to yield any filtrate during the pressure filtration procedure, treat it as 100% solid and proceed as described in Section A.

NOTE: Some wastes will obviously contain some materials that appear to be liquid, i.e.-oily wastes. If after filtration the material does not filter it is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

14.2.2 Weigh any filtered liquid (filtrate) and record the weight.

14.2.3 Determine the weight of the solid phase of the waste by subtracting the weight of the filtrate from the weight of the original sample. Calculate and record the percent solids using the formula:

- 14.2.4 If the percent solids are less than 0.5% then the filtrate is the sample extract. More sample may be filtered if necessary.
- 14.2.5 If the percent solids are greater than 0.5% remove the solid phase along with the filter and dry at 100 +/- 200C to a constant weight. Determine the % dry solids using the formula:

% Dry = (wt. of dry waste & filter) - (tared wt. of filter) X 100Solids initial wt. of waste

- 14.2.6 If the percent dry solids is less than 0.5% then the filtrate is the extract as described previously.
- 14.2.7 If the percent dry solids is greater than 0.5% then filter another sample of waste, retaining both the solids and the filtrate. Using the percent solids result from the initial filtration, calculate the volume of extraction fluid needed using the formula:

Wt. of ext. = 20 X percent solids X wt. of waste filtered Fluid 100

- 14.2.8 Treatment of the solid portion of waste from this point on is the same as that which is described in Section A. It may be necessary to filter two portions of the waste to determine the type of extraction fluid to use and to perform the actual extraction. The test to determine the type of extraction fluid to use may need to be modified (scaled down) if only a small amount of solid materials is available.
- 14.2.9 It should be noted that the filtered solids along with the entire filter are added to the extractor.
- 14.2.10After obtaining the final filtered extract, it may be combined with the initial sample filtrate if physically compatible. If the two phases are not compatible they should be analyzed separately and the results combined mathematically using the formula:

Final analyte concentration = (V1) (C1) + (V2) (C2)V1 + V2

- V1 = Volume of the first phase
- C1 = Concentration of analyte in the first phase
- V2 = Volume of the second phase

C2 = Concentration of the analyte in the second phase

- 14.3 Procedure for Volatile
 - 14.3.1 Room temperature must be constant (21-25^oC). Record the temperature in the temperature log twice a day. If the temperature is outside control limits contact the supervisor.
 - 14.3.3 If the percent solids or percent dry solids is <0.5% the filtrate is defined as the TCLP extract. Store in VOA vials and refrigerate until analysis.
 - 14.3.4 Determine whether or not particle size reduction is required as per Section 14.1.
 - 14.3.5 Weigh 25 gram of sample into Zero-Headspace Extractor (ZHE). Apply gentle pressure to 10 psi to force any liquid phase through the filter and into a tared collection container. Gradually increase the pressure in 10-psi increments to a maximum 50 psi, continuing to collect any liquid expelled. Reweigh the collection container. Store the filtrate at 4⁰C under minimal headspace conditions.
 - 14.3.6 Calculate and add to the ZHE the required amount of extraction fluid #1 (EF#1):

Weight (g) EF #1 = 20 (25-g. of filtrate)

- 14.3.7 Expel all air from the ZHE and pressurize to 5 to 10 psi. Place it in the rotary agitator and rotate for 18 +/- 2 hrs.
- 14.3.8 Express the aqueous leachate through the ZHE filter and collect. This filtrate plus the original filtrate (14.25) are collectively defined as the TCLP extract. If miscible, they are combined and analyzed. If immiscible, they are analyzed separately and the results are combined mathematically as in Section 14.2.
- 14.3.9 Rotate ZHE extractor every time which is used for Blank Extraction.

15. Calculations

15.1 Calculate results as per the specific method.

16. Method Performance

16.1 NA

17. Pollution Prevention

- 17.1 Use amount of chemicals as required. Do not make large quantities of solutions.
- 17.2 Use the hood when working with strong chemicals or fumes.
- 17.3 Keep the work area clean and clutter free to avoid any mishaps.

18. Data Assessment and Criteria for QC

- 18.1 Laboratory Reagent Blank
 - 18.1.1 The value of blank must be <MDL

- 18.2 Spike Samples
 - 18.2.1 The control limits are 75-125% recovery.

19. Corrective Actions for Out-of-Control Data

19.1 Laboratory Reagent Blank

- 19.1.1 If the blank is outside the limit, verify that there is no contamination.
- 19.1.2 Use fresh clean glassware.
- 19.1.3 Verify that the laboratory water is of good quality.
- 19.1.4 Prepare fresh reagents and standard if necessary.
- 19.2 <u>Spike sample</u>: If spike sample is outside control limits:
 - 19.2.1 Try a dilution (eliminate interference)
 - 19.2.2 Check technique (pipetting, homogeneity)
 - 19.2.3 If spike still fails contact supervisor, technical director for assistance.

20. Contingencies for Handling Out-of-Control and Unacceptable Data

- 20.1 When all the above mentioned (Section 19) corrective measures have been taken and data remain outside the QA criteria set forth above, immediately contact your supervisor.
- 20.2 Document the situation clearly in your laboratory notebook and place a copy of the information in the case narrative of the final data report.
- 20.3 The supervisor must contact the QA/QC Director, Laboratory Manager, and Technical Director and notify them of the situation.
- 20.4 A corrective action plan must be developed in order to solve the problem.

21. Waste Management

21.1 Keep sample for 180 days after analysis and dispose of them according to the procedures explained in the SOP for waste disposal.

22. References

22.1 Test Method for Evaluating Solid Wastes, SW 846, 3 rd Edition, Method 1311, Revision 0, July 1992 - Toxicity Characteristics Leaching Procedure Federal Register, Volume 57, No. 227, 55114-55117.

23. Tables, appendix, attachments

23.1 Extraction Log

Effective Date: June 23, 2017 Page 10 of 14

Appendix

CHEMITECH TCLP EXTRACTION LOC SOP ID: N 1311-17129-08 Matrix :	P) Bate Extra Revia	action Date: En06.04-15 00+06-05-15 action Time: In <u>5:00 NM 001-10</u> 30 AM
Weigh By: <u>JP</u> Extraction By: <u>JP</u> Chemical Used		15(-3) nec checks Lot Number
TOLP-FLUID-1	F C	WP40517
HCL-TGLP, IN	-	WP38575
HN03-TCL5,1N		WP38574

Prep Pos :

KD Bath Temperature: $\underline{NP^+c}$ Received Date: $\underline{O(c \cdot OS^+1S)}$ Delivered Date: $\underline{O(c \cdot OS^+1S)}$

NAG Envap Temperature:____ MEXT Received By: MET Delivered By: 7.P

65

prev: 20 por on 06-01-15

Analysis Gróyb : _____ IP, UG Extraction Group : 2P

Effective Date: June 23, 2017 Page 11 of 14

Appendix

CHEMTE		P	repBatch II	D: PB03010	5		
Analytical Method:	131)	Extraction Date: $\emptyset \in \mathcal{O}h = 15$		Concentration Date:			
Sample Number	Sample Weight (g)	Volume Extraction Fluid #1 (mL)	Multiphasic	Phase Miscible	Phases Combined	Final Leacha PH	te m[-
G2518-01	n/n-	NA	NA	NO	NA	7.0).0
62502-01	100.03	2000	WA	N-1-	NA	5.5	1.5
BLANK	-	2000		J.	d.	4.41	1.0

- OBLAND USED AN METER IN WE PHI
- -> Mutaix spikes use asses after fitration and before preservation.
- -> USED MY Stripps:.

(2) 0.0- 3.0- W1431 (2) 3.0 - 55. W1434 (2) 6.0- 8.0. W1436

15115 MB (15115

Extracts relinguished on the same date as received.

Appendix

CHEMTECH 284 Sheffield Street, Mountainside, New Jersey 07092 Phone - 908 789 8900 Fax : 908 789 8922

		TCLP Solid	d Determina	ition		1
Analyst <u>J7</u>				Supervisor Rev	iew:	5
Preparation Date:	06-04-15	Preparation Time:	41.45 B	m		
Initial Room Tempe	rature:	SIGC	Final Room	Temperature:	2200	
Sample Number	Sample Weight	Eilter Weight (g)	Filtrate (mL)	Filter - Solid (After 100°C)	% solids	% Dry Solids
G2502-01	NA	NA	NA	NO	100	NA
G2518-01	NO	NA	NA	NB	205	Nor

Appendix

CHEIMTE	284 Sneffield S	treet, Mountainside,	New Jersey C7092	2 Phone - 908 789 5	900 Fax : 908 789	8922
		TCLP Flui	d Determina	tion		1
Analyst: <u>ZP</u> Reparation Date:	06-04-15	Preparation Tin	ne: <u>ի</u> ւկ	Supervisor F 5 P M	leview:	A
Initial Room Tempe	rature:	21°C	Final Room 1	femperature:	220	C
Sample Number	Sample Weight	Volume DI Water (mL)	PH after 5 min stir	PH after 10 min stir	Extraction Fluid 1 or	pH Extraction
G2502-01	5.02	91.5	8.6	4.0	₩)	3.91
G2518-01	NA	NA	NA	WA-	No	NA

(1) 8.2 . 9.8 . W1438

Effective Date: June 23, 2017 Page 14 of 14

CHEMTECH 284 Sheffield Street, Mountainside, NJ 07092 (908) 789-8900

READ RECEIPT

Employee Name:

Department:

M1311-TCLP

Method or Document Read (Include Title, Number, Revision, as applicable)

Employee Statement:

I have read and understood the information in the above mentioned method or document.

Date

Supervisory Statement: I have reviewed this document or method with the employee.

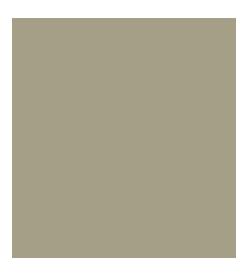
Supervisor Signature

Date

Note: This receipt is to be returned to the Quality Assurance/Quality Control Department for incorporation into employee training record files. If you have questions or would like to review your train record files, please see QA/QC Director.



L5 - Sample Receipt, Sample Acceptance Policy, Sample Login, Sample Identification, and Sample Storage



QA Control Code: A2040128

SOP Name:	SAMPLE RECEIPT, SAMPLE ACCEPTANCE POLICY, SAMPLE LOGIN, SAMPLE IDENTIFICATION, & SAMPLE STORAGE
SOP ID:	P250-Log-in Procedure
Revision #:	21
Date Created:	April 9, 2002
Effective Date:	July 27, 2017
Reason for Revision:	Annual Review
Supercedes:	P250-Log-in Procedure-20

Approvals:

Analyst

Supervisor

QA/QC Director

Technical Director

"The technical information contained herein is to be considered confidential and proprietary and is not to be disclosed, copied, or otherwise made available to other parties without the express written consent of Chemtech

Date

Date

Date

Date

SAMPLE MANAGEMENT

SAMPLE RECEIPT, SAMPLE ACCEPTANCE POLICY, SAMPLE LOG-IN, SAMPLE IDENTIFICATION, & SAMPLE STORAGE

1. Scope and Application

- 1.1 Procedures for sample handling, receipt, acceptance, login, and storage are defined in this SOP.
- 1.2 Defining sample acceptance and handling practices is critical to ensure the validity of the laboratory data.

2. Purpose and Responsibility

- 2.1 This SOP defines the procedures that Sample Management staff must utilize whenever receiving and logging in samples.
- 2.2 The SOP instructs the staff to use checklists to ensure sample integrity and provides the resources to electronically track samples through the laboratory.

3. Definitions

- 3.1 <u>Document Control Officer (DCO)</u>: The person who is to contact the people at the SMO or region when there are questions about the SDG.
- 3.2 <u>Sample Management Office (SMO)</u>: Federal contractor run department responsible for making sure that the contract laboratory receives the sample correctly. SMO also handles scheduling the work.
- 3.3 <u>Sample Delivery Group (SDG)</u>: A set of samples that can come in all at once or over a maximum of seven days for the same site.
 - There are to be no more than twenty samples per SDG.
 - If more than twenty samples come in for the same project, then the set is split up so that no more than twenty samples are grouped together.
- 3.4 <u>Sample Management Technician</u>: The individual responsible for correctly receiving and logging samples in for the laboratory.
- 3.5 <u>Chain of Custody (COC)</u>: An unbroken trail of accountability that ensures the physical security of samples & extracts, and includes the signatures of all who handle the samples.
- 3.6 <u>Laboratory Chronicle</u>: A form that must be completed with every set of samples that is received. This form documents the condition in which the samples were received.
- 3.7 <u>Preservation</u>: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.
- 3.8 <u>Pure Reagent Water</u>: Water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method.
- 3.9 <u>Record of Communication</u>: A form that documents the communication between the client and laboratory personnel.
- 3.10 <u>Standard Operating Procedures (SOPs)</u>: A written document which details the method of an operating, analysis or action whose techniques and procedures are

thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive task.

- 3.11 <u>Test Method</u>: An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP.
- 3.12 <u>Trip Blank</u>: Organic-free reagent water that is place in a 40ml vial and carried through sampling and handling to serve as a check on the contamination of volatiles by diffusion.
- 3.13 <u>Validation</u>: The process of substantiating specified performance criteria.

4. Procedure

- 4.1 <u>Sample Acceptance Policy</u>
 - 4.1.1 Sample storage locations are unique. Upon receipt of sample coolers at the laboratory, examine the coolers for damage or broken custody seals. Note the condition of the seals and cooler(s) on the Chain of Custody and follow Section 4.2.1. Send the COC to the Project Manager and obtain the Login Guidance (See Appendix K). Samples are logged into separate SDGs based on the analyses to be done.. If custody seals and coolers are intact, begin the sample acceptance procedure. If they are not intact, immediately notify the appropriate Laboratory Project Manager by email and request that they contact the client.
 - 4.1.2 After the Test Detail, Lab Chronicle and labels are received, remove the samples from the shipping containers under the hood, and set them up on the bench according to the Field Chain of Custody (COC). See SOP P204-COC for information regarding field COCs'. See Appendix B for a copy of the Field COC. Make sure that all sample labels are legible, written in indelible ink, and water- resistant.
 - 4.1.3 Check the COC/Test Detail and ensure that all sample containers listed on the COC are present. Any deviation from the number of sample containers, and/or sample damage or contamination must be listed on the Laboratory Chronicle. Inform the Laboratory Project Manager of the nonconformances by raising the ticket.
 - 4.1.4 Examine the field (COC) for completeness and correctness. Use the Sample Acceptance Checklist, Appendix A, as a guideline whenever accepting samples. Check the following:
 - 4.1.4.1 Check the client name and address, and make sure that it is legible.
 - 4.1.4.2 Ensure that the client has clearly written the project sampling location and identification, and that the client has indicated the name of the firm that has collected the samples. Verify the sample collector's name. Check that the billing information is also completed. If any information is missing, request the information from the client, if the client is present. If not, make a note on the Laboratory Chronicle.

- 4.1.4.3 Now check the sample information. Check the individual sample identification, the sample location, date and time of sample collection, matrix, number of containers, analysis request, and indication that the proper preservatives were used. See Appendix C for a listing of the correct preservative for each test performed. Check for any special remarks concerning the samples. List any non-conformances on the Laboratory Chronicle.
- 4.1.4.4 Pay close attention to the sample holding time and sample volume requirements. See Appendix C for a list of test holding times and sample volumes. If any sample holding times are less than 3 days, notify the appropriate supervisor of the situation. If holding times have been exceeded or the client did not provide sufficient volume, immediately inform the appropriate Laboratory Project Manager by raising a ticket and list the non-conformant samples on the laboratory chronicle. The Laboratory Project Manager will contact the client and give instructions to proceed with the acceptance or the Project Manager will indicate whether you should ship the non-conformant samples back to the client.
- 4.2 <u>Sample Receipt</u>
 - 4.2.1 Check the cooler for the presence of a temperature blank. If the cooler has a temperature blank, check the temperature and record. If a temperature blank is not provided, check the temperature of the samples utilizing the Wahl Infrared Thermometer-Model DHS 100X or equivalent. Aim the gun at the sample and press the measure temperature button. Record the cooler temperature on the COC and the laboratory chronicle. See Appendix C for proper sample preservation.
 - 4.2.1.1 All samples that require thermal preservation shall be considered acceptable if the arrival temperature is 2-6°C of the required temperature.

Note: Refer to Appendix C for the required preservation.

- 4.2.1.2 Samples that are brought to the laboratory soon after collection may not meet this criterion. These samples are acceptable if there is evidence (the samples are packed in a cooler with ice) that the sample collection personnel have initiated the chilling process.
- 4.2.1.3 Copy of the COC sent to PM to make log-in guidance via e-mail.
- 4.2.1.4 The Laboratory Project Manager and the Client should resolve all discrepancies between the AR & TRP and the COC prior to sample login.
- 4.2.1.5 Make sure to keep the samples cool at 4° C.

- 4.2.1.6 Proceed to Sample Login if there are no discrepancies and/or sample non-conformances. Sign the COC and enter the date and time of receipt.
- 4.2.1.7 If there are discrepancies and/or non-conformances, proceed to Sample Login once the client has given us clarifications and/or instructions to proceed.
 - 4.2.1.7.1 Once all discrepancies and/or non-conformances have been resolved, login the samples according to the Record of Communication given to you by the Laboratory Project Manager. See Appendix F for a copy of the Record of Communication.
 - 4.2.1.7.2 If the client does not wish to proceed with the analysis of the non-conformant samples, place the appropriate samples in the disposal area or ship back to the client as per their instructions.
- 4.2.1.8 Sign the field COC and enter the date and time of receipt.
- 4.2.2 Take the pH of any samples requiring a preservative using the narrow range pH paper.
 - 4.2.2.5 Collect a portion of the sample in a small plastic cup.
 - 4.2.2.6 Dip the pH paper in the sample.
 - 4.2.2.7 Record the pH of the sample by fraction on the Laboratory Chronicle.
 - 4.2.2.8 Discard the sample use following the waste disposal guidelines in P205-Waste disposal SOP.
 - 4.2.2.9 Notify the client of any pH discrepancy.
 - 4.2.2.10 If the pH does not comply with the preservative requirement, adjust the pH of the sample to the proper pH and note the pH and the pH adjustment on the Laboratory Chronicle. Use Appendix C as a guide for the appropriate preservative to use.

Note: Aqueous samples for metals analysis may be collected and shipped without acid preservation. However, acid must be added at least 24 hrs. before analysis to dissolve any metals that adsorb to the container walls.

- 4.2.3 Check the residual chlorine for all samples that have preservation dependent upon residual chlorine levels, see Appendix C. Samples containing residual chlorine have a preservative added at the time of sampling.
 - Determine if sample has residual chlorine using the HACH Test Kit.
 - Add contents of one pillow to a portion of the sample.
 - If the sample develops a pink/pinkish color, sample has residual chlorine. Follow direction in Appendix C.

Note: Ascorbic acid will be used for Cyanide preservation only if the sampling site is known to be positive for residual chlorine.

- 4.2.4 Verify that the date and time on the sample container matches the sample date and time on the COC/Test Detail.
- 4.2.5 Verify that the client sample identification listed on the label of the sample container matches the client sample identification on the COC exactly.
- 4.2.6 If the sample identifications or sample dates and times do not match, indicate this on the Laboratory Chronicle and notify the Laboratory Project Manager by raising a ticket.
- 4.2.7 SM/PM ticket is raised if there is any issue with the shipment or login guidance.

Note: <u>For North Carolina Work</u>: When aqueous samples received from North Carolina for the Pesticide (By method 8081A/B or EPA 608) or PCB (EPA 608) analysis, Total Residual Chlorine needs to be logged in to find out the presence of Total Residual Chlorine before samples are given to extraction department.

- 4.3 <u>Sample Log- In Procedure</u>
 - 4.3.1 The Login Module contains mainly 3 sections:
 - 4.3.1.1 <u>Project Management</u>: This section shows the number of orders created by project management department and provides the link for detailed view of the order and login report.
 - 4.3.1.1.1 New Order
 - 4.3.1.1.1.1 The Project Manager creates a new order.
 - 4.3.1.1.2 Information for Priority, PO#, Customer ID, Customer Contacts, Billing Contacts, Project ID, EDD Type, Data Package Type and Special Comments are added. Appropriate protocol has been selected from options Regular, DoD, NJDKQP as per clients request.
 - 4.3.1.1.1.3 The Project Manager selects Test Groups and Tests as per the Chain of Custody requirements or pre-defined Statement of Work.
 - 4.3.1.1.1.4 Add the required method, associated bottle type and preservation as per the Chain of Custody.
 - 4.3.1.1.2 Edit Order
 - 4.3.1.1.2.1 Follow same steps as in Section 4.3.1.1.1, to check and edit login. This step allows Project Manager to change, modify or delete any inserted data.
 - 4.3.1.1.3 <u>Copy Order</u>
 - 4.3.1.1.3.1 Provide Order from which all details are copied to the new Order.

4.3.1.1.3.2 Change login as per new requirement if required.

- 4.3.1.2 <u>Sample Management</u>: After New Project is created by Project Management, Sample Management enters the remaining information for login. This section shows the number of orders created by sample management department and provides the link for detailed view of the order and login report.
 - 4.3.1.2.1 Login
 - 4.3.1.2.1.1 Enter sample information per Matrix for each sample Lab Sample ID, Sequence, Customer Sample ID, Sample Date, Sample Date, Sample Time, Receive Date, Receive Time, No. of Container, Comment.

4.3.1.2.1.2 Select Test Group or Test for each sample. Compare log in test with Chain of Custody to make sure that all requested test has been logged.

- 4.3.1.2.1.3 Click on Submit button to approve selected data.
- 4.3.1.2.1.4 Click on Summary button to review login summary. View and edit data, if required.
- 4.3.1.2.1.5 Once all correct information is entered, click on Proceed for Signoff.
- 4.3.1.2.1.6 Status for this Order ID changes to Signoff Pending.
- 4.3.1.2.1.7 Click on sample number for updating sample priority, Test Group and Test.
- 4.3.1.2.2 Verification of Shipment
 - 4.3.1.2.2.1 Enter storage location for each test, or all tests.
 - 4.3.1.2.2.2 Enter information on Method of Shipping and Shipping charges.
 - 4.3.1.2.2.3 Click on submit.
 - 4.3.1.2.2.4 View and edit Bottle Type and Preservative for each test.
 - 4.3.1.2.2.5 Click on Update.
 - 4.3.1.2.2.6 Answer questions for Sample receipt.
 - 4.3.1.2.2.7 Click on Update.
 - 4.3.1.2.2.8 View and edit any login information entered.
 - 4.3.1.2.2.9 Click on Update and Transfer data.
- 4.3.1.3 <u>Other</u>:
 - 4.3.1.3.1 <u>Invoice</u>
 - 4.3.1.3.1.1 Invoice is created by accounting personnel.
 - 4.3.1.3.2 Labels

- 4.3.1.3.2.1 After data transfer, Enter Order ID to print labels for each sample bottle from Sample Master in Sample Management.
- 4.3.1.3.2.2 Print laboratory chronicle or Question report or Shipment Verification Detail form and Test Detail from intranet program to perform all sample checks (See Appendix L and M).
- 4.3.1.3.2.3 Project is signed off, reviewed, scanned and saved on the server by sample management.
- 4.3.1.3.2.4 If any problems are observed while labeling the samples, communicate to PM to follow up with the client by raising a ticket.

4.3.1.3.3 <u>Report</u>

- 4.3.1.3.3.1 Login Report: Enter Order ID and Click on View Report button.
- 4.3.1.3.3.2 Test Detail/Laboratory Chronicle: Enter Order ID and select Checkbox as per requirement (Sample Test Report or Question Report).
- 4.3.1.3.3.3 Click on View Report button.
- 4.3.1.3.4 Login Log Additional Samples
 - 4.3.1.3.4.1 Select Order ID.
 - 4.3.1.3.4.2 Samples already logged will be displayed with the test details.
 - 4.3.1.3.4.3 Enter additional samples and click on Submit button.
- 4.3.1.3.5 <u>Project Track</u>: This section shows the tickets which are under New Login status, Signoff Pending, and Signoff but not Supervised projects, and provides the link for detail data. This section redirects to Project Track Module to reply a ticket or Review and Supervised functionality.
- 4.3.2 Take folder and label the folder with the order number (with proper project code number), client name, and fax and hardcopy due dates.
- 4.3.3 Place a copy of the login report in the appropriately colored project folder. Make sure and add the original COC, Laboratory Chronicle and the Record of Communication (if one was necessary) to the folder.
- 4.3.4 Add third party carrier information (i.e. Federal Express or UPS receipts) to the project folder. Add the pickup and delivery tickets to the project folder if Chemtech was the sample carrier.
- 4.3.5 Give the completed project folder to the Laboratory Project Manager. (The Laboratory Project Manager must review each hard copy login summary against the COC for comparability and completeness. He/she must correct any errors, sign and date the login sheet).
- 4.4 <u>Sample Storage & Security</u>

- 4.4.1 <u>Sample Custodian</u>: See Appendix I for list of authorized sample custodians.
 - 4.4.1.1 Store the samples in the assigned refrigerator and according to conditions specified in methods or preservative protocols until the samples are requested.
 - 4.4.1.1.1 Check refrigerator temperature daily and record.
 - 4.4.1.1.2 Thermal preservation must be kept ±2°C of specified preservation temperature.
 - 4.4.1.1.3 Samples that require a 4°C thermal preservation have an acceptable preservation range of just above freezing for water to 6°C.
 - 4.4.1.1.4 Do not store volatile samples with any other samples. Place the volatile samples in the refrigerators designated for volatiles only.
 - 4.4.1.1.5 Store any suspected highly contaminated volatile samples in the refrigerator designated for suspected highly contaminated volatile samples.
 - 4.4.1.1.6 Make sure that all volatile refrigerators have storage blanks.
 - 4.4.1.2 Keep the samples locked at all times and release the samples only when samples are requested via the Sample Request Form (work list) by the analyst. See Appendix G for a copy of the Sample Request Form.
 - 4.4.1.3 Obtain the samples listed on the Sample Request Form and sign the appropriate sections of the Sample Retrieval & Return Logbook. See Appendix H for a copy of a logbook page.
 - 4.4.1.4 Ask the analyst, extraction or digestion technician receiving the samples to sign the Sample Retrieval & Return Logbook to acknowledge they have received the samples.
 - 4.4.1.5 The analyst will inform the Sample Custodian when the samples can be returned to storage. All samples must be returned to storage at the by the end of the day. Make sure that the analyst or extraction technician signs the appropriate sample return section of the Sample Retrieval & Return Logbook. See SOP P204-COC-01 for a detailed description of internal chain of custody procedures.
 - 4.4.1.6 Tracking Extracts and Digestates:
 - 4.4.1.6.1 When the extractions or digestions are complete, the extraction or digestion technician will have the appropriate analyst sign the extraction or digestion log that the extracts/digestates have been received.
 - 4.4.1.6.2 When analysts have completed work with the extracts or digestates, sign the extract or digestate log book and place them in refrigerators designated for extracts or digestates.

- 4.4.1.7 Retain the samples for a one-month period after the report is submitted to the client. The sample custodian in consultation with Sample Management Supervisor decides the samples to be disposed after 30 days.
- 4.5 <u>Sample Receiving (EPA PE CLP Samples)</u>
 - 4.5.1 Upon receipt of sample coolers at the laboratory, examine the coolers for damage or broken custody seal. Note the condition of the seal(s) and cooler(s) on the TR (Traffic Report)/COC (Chain of Custody) and follow Section 4.2.1. Note the temperature on the TR. Sign the field TR and enter the date and time of receipt. Send the TR to the project manager.
 - 4.5.2 Follow log-in procedure as per Section 4.3.
 - 4.5.3 Once the log-in procedure is complete, print the DC-1 Form and labels.
 - 4.5.4 Record in the 'Remarks' column of the DC-1 form if samples were received on ice.
 - 4.5.5 Take DC-1 form (Appendix J), TR and labels to the place where the samples are lined up. Check all items on the checklist and label all containers. Store them at the assigned location.
 - 4.5.6 Notify SMO if there are any discrepancies between the TR and the samples received (circle on DC-1 Form with '*'). Contact SMO if any of the items are missing, e.g. temperature bottle, custody seal, sample tag, air bill, etc. or if there is any sample breakage or MS/MSD is not assigned, etc. Wait for the Record of Resolution and make changes to log-in if required and notify the laboratory. Record cooler temperature in Remark #9.
 - 4.5.7 For aqueous Proficiency testing samples, log each bottle individually and assign separate lab ID #.
- 4.6 <u>Procedure for relinquishing samples to client</u>
 - 4.6.1 Sample management supervisor or sample management personal will have to verify that "Relinquished by & Date" field is properly filled out before shipping samples back to client.
 - 4.6.2 All signed documents needs to be scanned and saved on server.
 - 4.6.3 Any sample which needs to be shipped with ice must be shipped between Monday to Thursday unless there is a specific instruction from project manager to ship it on Friday, Saturday or Sunday.
 - 4.6.4 Sample cooler must have custody seal and cooler must have tape around it so cooler cannot be tampered.

5. Disposal of Samples

- 5.1 Samples are retained for 1 month after report submission to clients unless otherwise directed by client.
- 5.2 Dispose all non-CLP samples according to the Waste Disposal Plan (See Chemtech Chemical Waste Disposal Guidelines SOP P205-Chemical Waste Disposal-02.

5.3 Unused CLP sample volume and used sample containers are kept for 60 days after report submission. After 180 days, the samples are properly discarded utilizing the appropriate waste disposal procedure SOP P205-Chemical Waste Disposal-02.

6. Health and Safety

- 6.1 The toxicity and carcinogenity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure of these compounds should be minimized.
- 6.2 Always wear safety glasses for eye protection when working with these reagents.
- 6.3 Use protective gloves when handling the chemicals.

7. Corrective Action Procedure

- 7.1 A corrective action report is issued whenever there is damage to a sample (broken or lost) where data quality may be affected. This process is explained in detail in the Corrective Action Procedure SOP P202-CAR-02.
- 7.2 Issue a corrective action report whenever there is a deviation from the SOP.

8. Personnel Qualifications

- 8.1 All employees must undergo laboratory health and safety training and ethics training and must read the laboratory QA Manual and all SOPs related to their area of work.
- 8.2 A signed and dated statement from each technical employee that they have read, understood, and are using the latest version of the laboratory QA Manual and SOPs, and that they have read, acknowledged and understood their personal, ethical and legal responsibilities, is maintained in their training file.
- 8.3 The Technical Director certifies and documents that all technical employees have the appropriate education and/or technical background to perform the tasks for which the employee is responsible.

9. References

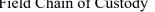
- 9.1 USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, Office of Emergency and Remedial Response. OSWER 9240.1-05A-P, PB99-963506, EPA540/R-99/008, October 1999.
- 9.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013 & Version 5.1 January 2017.

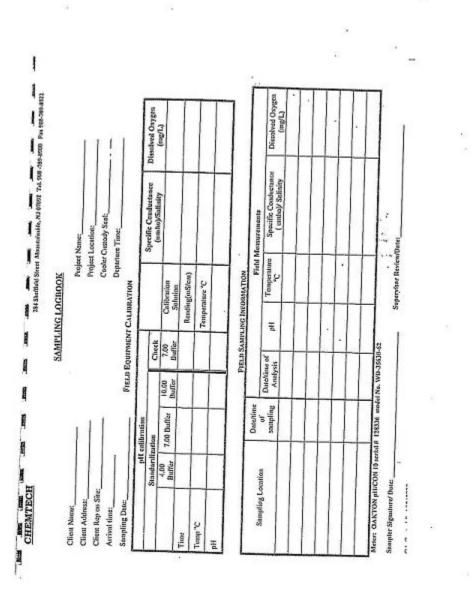
10. Appendices

- 10.1 Appendix B Field Chain of Custody
- 10.2 Appendix C Sample Holding Times, Preservatives, and Volume
- 10.3 Appendix D Analytical Request and Test Review Forms
- 10.4 Appendix E Rush Approval Form
- 10.5 Appendix F Record of Communication
- 10.6 Appendix G Sample Request Form
- 10.7 Appendix H Sample Retrieval and Return Logbook Page

- 10.8 Appendix I Authorized Sample Custodians
- 10.9 Appendix J DC-1 form
- 10.10 Appendix K Log-in guidance
- 10.11 Appendix L SM Login Sample Test Detail
- 10.12 Appendix M SM Login Question Report

<u>APPENDIX B</u> Field Chain of Custody





APPENDIX C

Water Sampling and Holding Time Information

Parameter	EPA Method	Standard Method and/or SW 846 Method	Preservation	Container	Holding Time	Minimum Volume
Turbidity	180.1	2130B	Cool, 4 deg C	P or G	48 Hrs	100 mL
Nitrate	300		Cool, 4 deg C	P or G	48 Hrs	250 mL
Nitrate-Nitrite	300		Cool to 4 deg C, Conc. H_2SO_4 to pH<2	P or G	28 Days	250 mL
Fluoride	300	4500 F-C	Cool, 4 deg C	P or G	28 Days	300 mL
Cyanide		4500-CN C&E	Cool, 4 deg C 50%NaOH pH>12	P or G	14 Days	500 mL
Sulfate	300	4500-SO4 E	Cool, 4 deg C	P or G	28 Days	50 mL
Total Dissolved Solids		2540C	Cool, 4 deg C	P or G	7 Days	100 mL
Calcium	200.7		1:1 HNO ₃ to pH<2	P or G	6 Months	100 mL
Calcium- Hardness	200.7		1:1 HNO ₃ to pH<2	P or G	6 Months	100 mL
Alkalinity		2320B	Cool, 4 deg C	P or G	14 Days	100 mL
Bromide	300		None	P or G	28 Days	250 mL
Chloride	300	4500-CL C	Cool, 4 deg C	P or G	28 Days	100 mL
Chlorite	300		1mL EDA to 1L Cool, 4 deg C	P or G	14 Days 10 mins	250 mL
Color		2120B	Cool, 4 deg C	P or G	24 Hrs	100 mL
Foaming Agents (MBAS)		5540C	Cool, 4 deg C	P or G	48 Hrs	250 mL
Odor		2150B	Cool, 4 deg C	G only	24 Hrs	200 mL
Conductivity	120.1	2510B, 9050A	Cool, 4 deg C	P or G	28 Days	100 mL
Silica	200.7		Cool, 4 deg C	P only	7 Days	50 mL
Ortho Phosphate	300	4500 P-E	Cool, 4 deg C	P or G	48 Hrs	50 mL
Chlorine, Residual Disinfectant		4500CI-G	None	P or G	15 minutes	200 mL
pH, Hydrogen ion		4500-H-B	None	P or G	15 minutes	25 mL
Temperature		2550B	None	P or G	15 minutes	1000 mL
Volatiles (Regulated)	524.2		Cool, 4 deg C 1:1 HCl to pH<2	G, screw cap Teflon faced silicone septum	14 Days	60-120 mL

CHEMTECH SOP ID: P250-Log-in Procedure Revision #21 QA Control Code: A2040128

Effective Date: July 27, 2017 Page 14 of 32

Parameter	EPA Method	Standard Method and/or SW 846 Method ASTM D1067-92	Preservation	Container P or G	Holding Time	Minimum Volume 100 mL
Acidity as CaCO ₃			ý 5		14 Days	
Alkalinity as CaCO ₃		2320B	Cool, 4 deg C	P or G	14 Days	100 mL
Ammonia		4500-NH3 H	Cool, 4 deg C, Conc. H_2SO_4 to pH<2	P or G	28 Days	400 mL
Biochemical Oxygen Demand		5210B	Cool, 4 deg C	P or G	24 Hrs.	1000 mL
Carbonaceous BOD		5210B	Cool, 4 deg C	P or G	24 Hrs.	1000 mL
Cyanide		9012A	Cool 4 deg C, 50% NaOH to pH>12 0.6 g ascorbic acid if residual chlorine present	P or G	Sulfide absent: 14 Days (Sulfide Present 24 Hrs.)	500 mL
Cyanide, Amenable		4500-CN C,G	Cool 4 deg C, 50% NaOH to pH>12 0.6 g ascorbic acid if residual chlorine present	P or G	Sulfide absent: 14 Days (Sulfide Present 24 Hrs.)	500 mL
Acid Soluble & Insoluble Sulfide		9030B	2N Zn Acetate, 6N NaOH to pH > 9, Cool, 4 deg C	P or G	7 Days	8 oz.
Total Hardness	200.7		HNO₃ to pH<2	P or G	6 Months	100 mL
Total Kjeldahl Nitrogen		4500-N OrgBorC	Cool, 4 deg C Conc. H_2SO_4 to pH<2	P or G	28 Days	500 mL
Oil & Grease		1664A	Cool 4 deg C, 1:1 HCL or conc. H ₂ SO ₄ to pH<2	G	28 Days	1000 mL
Orthophosphate	300	4500-P E	Filter immediately, Cool 4 deg C	P or G	48 Hrs.	50 mL
Phenols	420.1	9065	Cool 4 deg C, Conc. H_2SO_4 to pH<2	G	28 Days	500 mL
Total Phosphorus	365.3		Cool 4 deg C, Conc. H_2SO_4 to pH<2	G	28 Days	50 mL
Total-Residue (TS)		2540 B	Cool, 4 deg C	P or G	7 Days	100 mL
Residue-filtered (TDS)		2540 C	Cool, 4 deg C	P or G	7 Days	100 mL
Residue-non- filtered (TSS)		2540 D	Cool, 4 deg C	P or G	7 Days	100 mL

CHEMTECH SOP ID: P250-Log-in Procedure Revision #21 QA Control Code: A2040128

Effective Date: July 27, 2017 Page 15 of 32

Parameter Residue-	EPA Method	Standard Method and/or SW 846 Method 2540 F	Preservation	Container P or G	Holding Time 48 Hrs.	Minimum Volume 1000 mL
Settleable (SS)						
Residue-Volatile	160.4		Cool, 4 deg C	P or G	7 Days	100 mL
Salinity		2520 C	Cool, 4 deg C	G	28 Days	100 mL
Specific Conductance	120.1	2510B, 9050A	Cool, 4 deg C	P or G	28 Days	100 mL
Sulfate	300	4500-SO4 E	Cool, 4 deg C	P or G	28 Days	50ml
Sulfide		9034	Cool 4 deg C, add 2N Zinc Acetate + 6N NaOH to pH>9	P or G	7 Days	50 mL
Sulfite (SO3)		4500-SO3 B	Fix cooled samples (<50°C) immediately by adding 1mL EDTA soln./100mL sample	G, Bottle and Top	15 minutes	50 mL
Temperature		2550 B	None Required	G, Bottle and Top	15 minutes	1000 mL
Metals	200.7		1:1 HNO ₃ to pH<2	G	6 Months	100 mL
Mercury		7470A	Cool, 4 deg C	P or G	28 Days	8 oz.
Organochlorine Pesticides/PCB	608	8081A/N,8082/ 8082A	Cool, 4 deg C 0.008% Na ₂ S ₂ O ₃ if residual chlorine present, Adjust to pH 5-9 with 10N NaOH or 1:1 H2SO4	G, Amber Teflon- lined screw cap	7 days until extraction 40 days after extraction	1000 mL
Volatile Organics	624	8260B/C	Cool, 4 deg C 4 drops 10% Na ₂ S ₂ O ₃ if residual chlorine present 1:1 HCl to pH <2	Teflon-faced	7 days without HCl 14 days with HCl	40 mL
Semi volatile Organics	625	8270C/D	Cool, 4 deg C 0.008% Na ₂ S ₂ O ₃ if residual chlorine present	G, Amber Teflon- lined screw cap	7 days until extraction 40 days after extraction	1000 mL
DRO		8015B	Cool, 4 deg C 0.008% Na ₂ S ₂ O ₃ if residual chlorine present	G, Amber Teflon- lined screw cap	7 days until extraction 40 days after extraction	1000 mL
COD		SM5220D	Cool, 4 deg C H2SO4 to pH<2	Р	28 Days	1000 mL
TOC		SW9060	Cool, 4 deg C HCl or H2SO4 to	Р	28 days	1000 mL
Horbisida		Lloyd Kahn	pH<2	C Ambar	14 days	11
Herbicide		SW8151	Cool, 4 deg C	G, Amber	7 days until extraction 40 days after extraction	1 L

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SOP ID: P250-Log-in P	rocedure
Revision #21	QA Control Code: A2040128

Effective Date: July 27, 2017 Page 16 of 32

Parameter	EPA Method	Standard Method and/or SW 846 Method	Preservation	Container	Holding Time	Minimum Volume
GRO		8015B	Cool, 4 deg C 0.008% Na ₂ S ₂ O ₃ if residual chlorine present 1:1 HCl to pH <2	G, Vial screw cap with center hole Teflon-faced silicone septum	7 days without HCl 14 days with HCl	40 mL
Gases		3810	Cool, 4 deg C 0.008% Na ₂ S ₂ O ₃ if residual chlorine present 1:1 HCl to pH <2	G, Vial screw cap with center hole Teflon-faced silicone septum	7 days without HCl 14 days with HCl	40 mL
HPLC (Explosive)		8330A/B	Cool, 4 deg C	G, Amber Teflon- lined screw cap	7 days until extraction 40 days after extraction	1000mL
Hexavalent Chromium		3500 Cr D	Cool, 4 deg C	Р	24 Hrs.	100mL
Ferrous Iron		HACH 8146	Cool, 4 deg C	Amber G	24 Hrs.	250mL
RSK 175		RSK 175	Cool, 4 deg C 1:1 H2SO4 or HCl to ph<2	G, Vial screw cap with center hole Teflon-faced silicone septum	14 days	40 mL
Formaldehyde		HACH 8110	Cool, 4 deg C	Р	Analyze Immediately within 48hrs	1000mL
Ferrous Iron		HACH 8146 SM3500	Cool, 4 deg C	Р	Analyze Immediately within 48hrs	1000mL
Chemical Warfare Agents		8270-modified	Cool, 4 deg C	G, Amber Teflon- lined screw cap	7 days until extraction 40 days after extraction	1000mL
Glycols		Chemtech SOP	Cool, 4 deg C	G	28 days	100mL
Perchlorate	314.0			P or G	28 days	500mL

Container Key: P = Plastic G =Glass

G =Glass DW= Drinking Water

Soil/Hazardous Waste Sampling and Holding Time Information

Parameter	EPA Method	Standard Method and/or SW 846 Method	Preservation	Container	Holding Time	Minimum Volume
Ignitability		1010	None	P or G	None	8 oz.
Ignitability of Solids		1030	None	P or G	None	8 oz.
Corrosivity pH Waste>20% water		9040B	Cool, 4 deg C	Р	15 minutes	4 oz.
Corrosivity Toward Steel		1110	Cool, 4 deg C	Р	14 Days	4 oz.
Reactivity Cyanide		SW-846 7.3.3.2	Cool, 4 deg C	Р	14 Days	8 oz.
Reactivity Sulfide		SW-846 7.3.4.2	Cool, 4 deg C	Р	14 Days	8 oz.
TCLP Volatile Organics		1311	Cool, 4 deg C	G	14 Days to TCLP extraction, 14 days to analysis	4 oz.
TCLP Metals		1311	Cool, 4 deg C	G	180 Days to TCLP extraction, 180 days to analysis	16 oz
TCLP Mercury		1311	Cool, 4 deg C	G	28 Days to TCLP extraction, 28 days to analysis	16 oz
TCLP Semi volatiles		1311	Cool, 4 deg C	G	14 Days to TCLP extraction, 7 days to extraction, 40 days to analysis	16 oz
TCLP Pesticides and Herbicides		1311	Cool, 4 deg C	G	14 Days to TCLP extraction, 7 days to extraction, 40 days to analysis	16 oz
PH		9040B, 9041A, 9045C	Cool, 4 deg C	Р	15 minutes	4 oz.

CHEMTECH SOP ID: P250-Log-in Procedure Revision #21 QA Control Code: A2040128

Effective Date: July 27, 2017 Page 18 of 32

Parameter	EPA Method	Standard Method and/or SW 846 Method	Preservation	Container	Holding Time	Minimum Volume
Temperature		2550 B		Р	15 minutes	4 oz.
Metals		6010B/C	Cool, 4 deg C	P or G	6 Months	8 oz.
Mercury		7471A	Cool, 4 deg C	Cool, 4 deg C P or G 28 Da		8 oz.
Organochlorine Pesticides		8081A/B	Cool, 4 deg C	P or G	14 Days for extraction, 40 days to analysis	8 oz.
PCB's		8082/8082A	Cool, 4 deg C			8 oz.
Chlorinated Herbicides		8151A	Cool, 4 deg C	G, wide mouth, Teflon liner	14 Days	8 oz.
Volatile Organics		8260B/C	Cool, 4 deg C	G, wide mouth, Teflon liner	14 Days	4 oz.
Semi volatile Organics		8270C/D	Cool, 4 deg C	Amber Glass	14 Days to extraction, 40 days to analysis	8 oz.
Total Cyanide		9012A	Cool, 4 deg C	P or G	14 Days	8 oz.
Amenable Cyanide		9010B	Cool, 4 deg C	P or G	14 Days	8 oz.
Acid Soluble & Insoluble Sulfide		9030B	Cool, 4 deg C No Headspace	P or G	7 Days	8 oz.
Extractable Sulfide		9031	Cool, 4 deg C Fill solid surface with 2N Zinc Acetate until moistened, 4 drops 2N Zinc Acetate/100mL sample, 50%NaOH to pH>9	P or G	7 Days	8 oz.
Sulfate		9038, 9056	Cool, 4 deg C	P or G	28 Days	8 oz.
pH, Soil and Waste		9045C	Cool, 4 deg C	G	15 minutes	8 oz.

Parameter	EPA Method	Standard Method and/or SW 846 Method	Preservation	Container	Holding Time	Minimum Volume
Phenol		9065	Cool 4 deg C	G	28 Days	8 oz.
Oil & Grease (Sludge, Sludge- Hem)		9071B	Cool 4 deg C	G	28 Days	8 oz.
Paint Filter Liquids Test		9095	Cool, 4 deg C	P or G		8 oz.
Nitrate		9056	Cool, 4 deg C	P or G	48 Hrs	8 oz.
Bromide		9056	Cool, 4 deg C	P or G	28 Days	8 oz.
Chloride		9056	None	P or G	28 Days	8 oz.
Fluoride		9056, 9214			28 Days	8 oz.
Cation- Exchange Capacity	ange acity			8 oz.		
DRO		8015B	Cool, 4 deg C	Amber Glass	14 Days to extraction, 40 days to analysis	8 oz.
GRO		8015B	Cool, 4 deg C	G, wide mouth, Teflon liner	14 Days	4 oz.
Gases		3810	Cool, 4 deg C	Amber Glass	14 Days	8 oz.
Hexavalent Chromium		3060, 7196A	Cool, 4 deg C	P	30 Days to extraction, 7 days to analysis	4 oz.
Explosives		8330A/B	Cool, 4 deg C	Amber Glass		
TOC		SW9060 Lloyd Kahn	Cool, 4 deg C	G	28 Days 14 Days	8 oz.
Herbicide		SW8151	Cool, 4 deg C	G	14 Days to extraction, 40 days to analysis	4 oz.
Formaldehyde		HACH 8110	Cool, 4 deg C	G	Analyze Immediately within 48hrs	4 oz.
Ferrous Iron		HACH 8146 SM3500	Cool, 4 deg C	G	Analyze Immediately within 48hrs	4 oz.
Chemical Warfare Agents		8270-modified	Cool, 4 deg C	G	14 days until extraction 40 days after extraction	8 oz.

CHEMTECH SOP ID: P250-Log-in Procedure Revision #21 QA Control Code: A2040128

Effective Date: July 27, 2017 Page 20 of 32

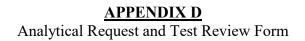
Grain Size ASTM D422	P or G		4 oz.
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CLP Sampling and Holding Time Information

CLI Samping and Holding Third Information					
Parameter	EPA Method	Preservation	Container	Holding Time	Minimum Volume
METALS (aqueous)	ILM05.3 ILM05.4 ISM01.2	HNO_3 to pH<2, Cool 4deg C	Р	180 Days from VTSR	1000ml
CYANIDE (aqueous)	ILM05.3 ILM05.4 ISM01.2	NaOH to pH>12, Cool 4deg C	Р	12 Days from VTSR	1000ml
MERCURY (aqueous)	ILM05.3 ILM05.4 ISM01.2	HNO ₃ to pH<2, Cool 4deg C	Р	26 Days from VTSR	1000ml
VOLATILE ORGANICS (aqueous)	OLM04.3, SOM01.X	HCL pH < 2, Cool 4deg C	G	10 Days from VTSR with preservative, 7 Days from VTSR without preservative	40ml
SEMI- VOLATILE ORGANICS (aqueous)	OMLO4.3, SOM01.X	Cool 4deg C	G	5 Days from VTSR for extraction 40 Days after extraction	1000ml
PESTICIDES (aqueous)	OLM04.3, SOM01.X	Cool 4deg C	G	5 Days from VTSR for extraction 40 Days after extraction	1000ml
PCBs (aqueous)	OLM04.3, SOM01.X	Cool 4deg C	G	5 Days from VTSR for extraction 40 Days after extraction	1000ml
METALS (solid/soils)	ILM05.3 ILM05.4 ISM01.2	Cool 4deg C	G	180 Days from VTSR	8 oz
*CYANIDE	ILM05.3 ILM05.4 ISM01.2	Cool 4deg C	G	12 Days from VTSR	8 oz
MERCURY (solid/soils)	ILM05.3 ILM05.4 ISM01.2	Cool 4deg C	G	26 Days from VTSR	8 oz
VOLATILE ORGANICS (solid/soils)	OLM04.3, SOM01.X	Cool 4deg C	G	10 Days from VTSR	4 oz
SEMI- VOLATILE ORGANICS (solid/soils)	OLM04.3, SOM01.X	Cool 4deg C	G	10 Days from VTSR for extraction 40 Days after extraction	8 oz
PESTICIDES (solid/soils)	OLM04.3, SOM01.X	Cool 4deg C	G	10 Days from VTSR for extraction 40 Days after extraction	8 oz
PCBs (solid/soils)	OLM04.3, SOM01.X	Cool 4deg C	G	10 Days from VTSR for extraction 40 Days after extraction	8 oz

*When chlorine is present ascorbic acid is used to remove the interference (0.6 g ascorbic acid) Note: Unpreserved soil samples must be refrigerated at a temperature of $-7 \deg C$ ($\pm 2 \deg C$)

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Order Date Tima 1	1/25/2009 2:20128 PM		Deliverables				
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Contact; Dolinery Juba Time /							
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Sri Dottio Yuno	Preservation		And			- 66	
2 Liter Plastic	Cani 4 dep C	-	E	- Sent	MUCHA	Method	2
Snu mi Plestic	Conc H2SO4 to pH 2 3		1 SAM 1	9005 155	Water Water	SM5210 B SM2540 D	-
			F	TICN	Water	N 00\$MS	
8 oz Glass	NONE	ч	2 SAM			-	
			2	Field pH	Weter	8 H-0055WS	
Custody Seal NO	Extra Lables NO	Chain	Chain Of Custody .	NO I	No. of Coolors	0	2
Shipping Comment :	2 day sampling oversion of later them 2 octors, set up on 4/7/03, os far the potter, thean, no later than 2 octors, pedap on A/8/09 no later 1 later 2 clock	then 2 other 9 no loter U	A, set up on 4/7 son 2 clock	1/03. 08/2ar the h	attar,Altan, mo later		
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APPENDIX E

Rush Approval Form

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CHEMTECH

Expedite Turnaround Approved

TA ID: Scheduled: Created By: Comments:

Project ID: Client: Approved By:

Test Manis Groups Manuy Manis ICP-RCRA

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TestGroup METALS RCRA METALS RCRA

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Matrix

Solid Solid Solid Taibarbund Quantity 2 Bus.Days 3 2 Bus.Days 1 2 Bus.Days 1 2 Bus.Days 1

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APPENDIX F

Internal Record of Communication Login Change Form

Order Number:	Today's Date:				
X					
Client:	Sample Date:				
Project:	Form Initiated by:				
Client Contact:	Project Manager:				
General Comments/Special Instructions:					

Call Initiated by \Box Client \Box Chemtech

Login Changes

SAMPLE NUMBER	Add Test	Delete Test	Change TAT

Signature _____ I

Date _____

Effective Date: July 27, 2017 Page 25 of 32

APPENDIX G Sample Request Form

WORKLIST(Hardcopy Internal Chain)

/orkList Name : G2420E		WorkList ID : 77565			Date : 6/3/2015 1:16:51 PM				
Due Date	Matrix	Sample	Test	Preservative	Customer	Storage Location	Customer Sample	Collect Date	Method
06/03/2015	Water	G2420-35	Diesel Range Organics	Cool 4 deg C	UNIO02	G51	FB-5-27-15	05/27/2015	8015B
06/10/2015	Water	G2420-35	EPH	1:1 HCl to pH <	UNIO02	G51	FB-5-27-15	05/27/2015	NJEPH
06/03/2015	Water	G2420-35	Pesticide-TCL	Cool 4 deg C	UNI002	G51	FB-5-27-15	05/27/2015	8081B

Date/Time	06/03/15	
Received by:	fin	
Relinquished by:	(?	

Date/Time	0.6/03/15
Received by:	<u>Q</u>
Relinquished by:	fi

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Page 1 of 1

<u>APPENDIX H</u> Sample Retrieval and Return Logbook Page

DEP-077 Rev. 3/04

New Jersey Department of Environmental Protection

Page 1 of 1

Internal Chain of Custody

Instructions: Use 1 form for each 20 samples of aliquot

Printed Name

Printed Name

Printed Name

Signature

Signature

Signature

		Laboratory Person Breaking Fig	d Seal on Sample Shuttle & Accepting	Responsibility for Sample			
oratory: Chemtech Loc			ation: 284 Sheffield Street, Mountainside,NJ 7092. Title: Sample,Custodian Date Broken 6/2/2015 Military Time Seal Broken: 13:50:00 Analytical Parameter/FractiofD-15				
San	nple No.	Aliquot/Extract No.	Sample No.	Aliquot/Extract No.			
G2477	32477-01 IA1						
G2477	-02	SV1					
G2477	-03	QA1					
			a na sa				
Date	Time	Relinquished By	Received By	Purpose of Change of Custody			
d2)15		signature (Auche -	Signature South				
Ø	16:	Printed Name SWEAAL WOHT	Printed Norman unchasta	To voc les			
		Signature	Signature				
		Printed Name	Printed Name				
	1.400000	Signature	Signature				
		Printed Name	Printed Name				
		Signature	Signature				
		Printed Name	Printed Name				
		Signature	Signature				

Printed Name Printed Name Distribution: White - Original (Sent With Report) Yellow - Contractor Archive Pink - Sample Custodian - Interim Copy

Printed Name

Printed Name

Printed Name

Signature

Signature

Signature

APPENDIX I

Authorized Sample Custodians

PERSONS AUTHORIZED AS SAMPLE CUSTODIAN

NJ: SNEHAL MEHTA

DESIGNATED SAMPLE CUSTODIAN

APPENDIX J DC-1 form

Lab Name CH	EMTECH CONSULT	SAMPLE LO ING GROUP	G-IN SH	EET				Page_of
Received By ((Print Name)				Log-	in Dat	æ	
Received By ((Signature)							
Case Number	·	Sample Deli	verv Gr	roup No.	MC0C85	i i	MOD.	REF.NO
Remarks:					Corres	pondi	ng	
 Centroly, Scal(s) 	Present/Absent*							Remarks: Condition of
2. Castady Seal Nos.	htset/Booken	EPA Sample #	Aqueo Sampl pH	e	nple Tag #	Assigi Laba		Sample shipment, etc.
 Tooffis Reports/Chain Of Castedy Records 	Present/Alwent*							
4. Airbill	Aistell/Sticker Present/Alwent*							
5. Airbill Nn.								
6. Sumple Tage Sample Tag #	Present/Absent* Listed/Not Listed On TR/ Chain-of-Costedy							
7. Sample Condition	Intuct/Broken*/Leoking	<u> </u>	-	-				++
8. Cooler Tempenture Indicator Botto	Present/Absaut ^a							
9. Cooler Temperature			-	_				+
 Does information on costudy preceds, traffic reports, and sample tage 	YanNor							
 Date Received at Lab Time Received. 								
Sampl	e Transfer							
Fraction	Fraction							
Area #	Area #							
By	Ву							
On	On							
	O and attach recor	, d of resolutio	n					
Reviewed By				Logbook				
Date				Logbook	Page			

FORM DC-1

ISM01.3

APPENDIX K Login Guidance



<u>APPENDIX L</u> SM Login Sample Test Detail

SM Login Order Sample Test Details

DrderID	E2409	Project1D	Kelly AFB
CustomerID SHAW22 Customer Name CB&I	Project Manager	reginald	
	- Minist	Sales Executive	Franco
	Order Date	06/07/2013	
EDD Type	Envirodata 2010	DataPackage Type	Level 3

Comment : Next DOD requirements: Run samples without dilution to meet limits, Level III data patkage equivalent to ND Reduced. Lab must be notified to meet requirements of newer versions of methods required for bacocc, 6270D, 90518, 8052A, 6020A, 6010C, 9010C, Need HC + EDD Enviro Data EDD and ERPIMS EDD. Mail White copy of COC with report to Houston Office Send Errino Data EDD and ERPIMS EDD to Jennifer hoang@shalvgrp.com, Kurt.Schmidt@shalvgrp.com, and Carlston.Edmunds@shalvgrp.com

Lab Sample ID	Matrix	CustomerSa	mple	Collect D	wte Colle	octTime	Receive Date	a Bott	le Qty
E2489-01	Water	55043MW009-13	060	06/06/2013	09;55	00	\$/2/2013-10-00	0 00	6
Test		5 TestGroup	Meth	boi	PrepMethod	Pres	ervative	BottleType	Pr
Netals Group	64		50 2	D.	1LM05.4	1:1	HN03 to pH ≈ Z	500 ml Plasti	c 10
SVOCMS Gro	ups		827	b-Madified	3510	Casi	4 deg G	I Liter Ambe	r 10
VOCMS Grau	фВ		62, Ó	D-Law	N64	13.8	ICI to pH < 2	40 ml.Vials	10
F2489-02	Water	LF017MW005-13	760	06/06/2013	13 (67	00	6/7/2013 10:11	5 OC	5
Test		TestGroup	Meth	bon	PrepMethod	Pres	ervative	BottleType	Pr
Netals Group	24		50 Z	D	ILM05.4	111	HN03 to pH < 2	500 ml Plasti	c 10
SVOCMS Gro	up5		827	0-Madified	3510	Cuol	4 deg C	1 Liter Ambe	r 10
VOCMS Grou	рВ		826	D-Law	NA	1:1 8	ici to pH < 2	40 ml vials	10
E2489-03	Water.	LF014MW026-13	060	06/06/2013	12:41	:00	6/7/2013 10:11	x oc	ą.
Test		TestGroup	Meth	bod	PrepMethod	Pres	ervative	BattleType	Pr
VOCMS Grau	рВ		526	0-Lake	NA	1:38	ICI to pH < 2	40 ml Vials	10
E2489-04	Water	7601-130505		06/06/2013	05:45	:00	\$/7/2013-10-11	a: tác	e.
Test		TestGroup	Meth	od	PrepMethod	Pres	ervative	BottleType	Pr
VOCMS Grau	фВ		82,6	D-Low	164	138	ici III рн « 2	40 mLVBIs	10
E2439-05	Water	_F001M\Y002-13	060	06/06/2013	40(37	00	6/7/2013 10:11	2 00	6
Test		TestGroup	Meth	boi	PrepMethod	Pres	ervative	BottleType	Pr
Metals Group	24		502	0	ILM05.4	1:1	HNO3 to pH < 2	500 ml Plasti	c 10
SVOCHS GR	mb2		B2,7	D-Modified	35 10	Cool	4 deg C	1 Liter Ambe	10
VOCMS Grou	рВ		B2.6	0°Low	NA	1:1 8	ICI to pH < 2	40 mi Vals	10
E2489-06	Whiter:	LFD01MW003-13	060/	06/06/2013	11:14	1:00	6/7/2013 10/11	x.00	B
Test		TestGroup	Meth	bod	PrepMethod	Pres	ervative	BottleType	Pr

<u>APPENDIX M</u> SM Login Question Report

	Shipment Verification Detail			
19		ProjectID	Kelly AFB	

OrderID E2489	ProjectID K	elly AFB	
CustomerID SHAW22	Project Manager re	ginald	
Customer Name CB81	Sales Executive Fr	ránco	
Customer Contact Jennifer Hoang	Order Date 0	6/07/2013	
Are samples submitted with a chain of custody?	Yes	No	NA
Are the number of samples the same as stated on the chain of custody?	Yes	No	NA
Are bottle caps tight and securely in place?	Yes	No	NA
Were all containers infact when received?	Yes	No	NA
Were samples submitted in an ice chest?	Yes	No	NA
Were samples received cold?	Yes	No	NA
Were samples within the holding time for the requested test(s)?	Yes	No	NA
Is the volume of sample submitted sufficient for the requested test(s)?	Yes	No	NA
Are all samples for volatile organic analyses free of headspace?	Yes	No	NA

Effective Date: July 27, 2017 Page 32 of 32

CHEMTECH 284 Sheffield Street, Mountainside, NJ 07092 (908) 789-8900

READ RECEIPT

Employee Name:

Department:

P250-Log-in Procedure

Method or Document Read (Include Title, Number, Revision, as applicable)

Employee Statement:

I have read and understood the information in the above mentioned method or document.

Employee S	Signature
------------	-----------

Date

Supervisory Statement: I have reviewed this document or method with the employee.

Supervisor Signature

Date

Note: This receipt is to be returned to the Quality Assurance/Quality Control Department for incorporation into employee training record files. If you have questions or would like to review your train record files, see QA/QC Director.

Appendix C Responses to USEPA Comments, USEPA and TDEC Approval Letters

EPA General Comments:

1. The Supplemental Remedial Investigation Phase 3 Quality Assurance Project Plan (QAPP) does not identify quality assurance (QA) personnel or show the independence of the QA function on this project. Please revise the SRI QAPP to identify all QA personnel involved in this investigation and demonstrate the independence of the QA function on this project.

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. The method in which the soil cuttings investigation derived waste (IDW) will be sampled indicates the results of the soil samples collected and submitted for laboratory analysis could be biased low. For example, in QAPP Worksheet #14/16: Project Tasks & Schedule, Investigation Derived Waste, Page 26, the test states "Soil cuttings from the well borings will be transported to Dunn Field and placed on plastic sheeting; the cuttings will be covered by plastic sheeting held in place by perimeter weights. Grab samples will be collected from the soil cuttings for analysis of VOCs by standard (8260) analysis and by TCLP." As such, volatilization of contaminants could occur during the process of transporting the soil cuttings from the Main Installation (MI) to Dunn Field, placing the cuttings on, and covering them with plastic sheeting, then, collecting and submitting the sample for standard (8260) analysis. Additionally, it is not clear why the IDW soil samples submitted for laboratory analysis will not be collected using a sampler such as an EncoreTM to determine the presence of low level volatile organic compounds (VOCs)..
 - a. It is strongly recommended the soil cuttings generated from the Phase 3 sampling investigation be containerized prior to transportation and an Encore[™]-type soil sampler be used to collect a portion of the IDW directly from the container to ensure representative and quality results are obtained for decision making.

Response: Army believes the current sampling procedure for IDW soil provides representative results for disposal requirements. The samples are not intended to be representative of in situ soil.

The Phase 3 soil cuttings will be generated through rotasonic drilling of well borings and the heat of the drill rod will likely volatilize a significant portion of any CVOCs present in the soil. Continuous soil cores will be collected for logging and PID screening. As stated on page 23 of the QAPP "Cuttings with suspected contamination due to elevated photoionization detector (PID) readings or visible staining will be segregated". Containerizing soil cuttings that do not have apparent contamination is unnecessary and imposes additional cost on the Army from handling the drums and collecting samples from each drum. Upon completion of drilling, one soil sample will be collected for approximately each 5 cubic yards of soil cuttings. At each sample location, approximately 6 inches of surface soil will be removed and samples will be collected for VOC analysis by standard extraction and for toxicity characteristic leaching procedure (TCLP) extraction. Soil samples for standard extraction will be collected with an EncoreTM sampler. Soil samples for TCLP extraction, which requires greater sample volume, will be collected by completely filling a 4-ounce jar for each location. The TCLP analyses will be used if CVOC concentrations from standard analysis exceed the remediation goals.

The QAPP will be revised to provide the above information and to state that cuttings with apparent contamination will be placed in a drum after logging and screening the soil core. Worksheets #12, #14&16, #15, #17, #18, #19&30, #23, and #29 in the QAPP will be revised to add information for soil IDW analyses.

3. QAPP Worksheet #20: Field QC Summary provides a summary of groundwater samples to be collected. However, no additional information regarding IDW water samples collected for volatile organic compounds (VOCs) analysis is provided. Please revise the SRI QAPP to include all required information for the collection and analysis of IDW water for VOCs.

Response: A note will be added to Worksheet #20 stating "Samples of soil and water IDW will be collected at

completion of field work or before if storage capacity is reached; field quality control (QC) samples are not required by the disposal facility and will not be collected."

The QAPP will be revised to add necessary information for IDW water to Worksheets #12, #15, #18, #19&30, #23 and #29. A statement will be added to Worksheet #17: Grab samples of IDW water and soil will be collected from the containers/soil pile as described on Worksheet #14&16.

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.

Response: Worksheet #6 will be revised to add 'EPA Oversight' and 'TDEC Oversight' as separate drivers with RPMs the 'Responsible Entities' and the 'Procedure' as "Review and comment on SRI Phase 3 documents, participation in monthly Site Management Team calls to discuss recent project activities, document review, task schedule and issue resolution".

a. Please revise the worksheet to specify that the EPA will be notified when significant corrective actions or changes occur.

Response: Will add "Field Activities" as a 'Driver', with HDR PM as 'Responsible Entity'; 'Procedure' Notify BEC of planned changes and corrective actions for field activities for discussion with USEPA and TDEC RPMs and regulatory buy-in". Will also add "monitoring compliance with the FFA" to the 'Procedure' for the Regulatory Interface driver; the FFA specifies required notification to EPA and TDEC.

b. Please revise the worksheet to also include the form of communication and timeframe for notification for all communication drivers.

Response: Will add type of communication (call, email or letter) and timeframe to Worksheet #6.

2. Worksheet #10: Conceptual Site Model, Land Use Considerations, Page 10: The text in this section indicates land use controls (LUCs) are not required in Functional Unit 6, where unrestricted residential land use is permissible. However, Functional Unit 6 is not presented on any figures presented in the SRI QAPP. Please revise the SRI QAPP to identify the location of Functional Unit 6 in the relevant figures.

Response: Referenced text will be revised to "... except in the eastern section of Functional Unit 6 (Figure _), which is ..." A new figure will be added showing functional unit locations and the area where unrestricted use is allowed.

3. Worksheet #14/16: Project Tasks & Schedule, Pages 25-26: Worksheet #14/16 describes the general groundwater well construction under the subheadings for Fluvial Aquifer Wells and Intermediate Aquifer Wells. However, the SRI QAPP does not provide the proposed depth and well construction information for each of the twenty (20) proposed fluvial wells and three (3) proposed intermediate wells to be installed, as listed in Worksheet #17, Pages 29-30. Revise the SRI QAPP to include more detailed well construction information for all proposed well locations including but not limited to depth of the well and proposed screened intervals. Additionally, provide the well construction information for all existing wells on the Main Installation (MI) along with geologic cross-sections of the MI which will be included in the upcoming SRI Phase 3 report.

Response: A table listing the Phase 3 wells with planned location coordinates, aquifer to be screened and estimated depths for groundwater, top of clay, surface casing (where planned) and top of screen; the planned

screen length (10 feet) will also be listed. A table listing existing MI wells will also be provided. A location map with existing wells and planned Phase 3 wells is provided on Figure 9.

Numerous cross-sections for the MI are included in the SRI Phases 1 and 2 Report, which has been submitted to EPA. The Phase 3 wells were not planned based on location relative to cross-sections; cross-sections for the Phase 3 report will be identified and prepared after the wells are installed. Inclusion of existing cross-section in the QAPP is not considered necessary.

- 4. Worksheet #14/16: Project Tasks & Schedule, Page 25: Worksheet #14/16 states under the subheading Investigation Derived Waste (IDW), "If standard analysis demonstrates the VOC concentrations are less than the Dunn Field remedial goals (RGs), the soil cuttings will be spread on Dunn Field"; however, Worksheet #10: Conceptual Site Model, Page 11 states, "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 CVOCs in groundwater at the MI." According to the SRI QAPP, IDW soils are not proposed to be sampled and analyzed for PCBs, pesticides/herbicides, and PCP. Therefore, it is unclear if soil cuttings, which may be potentially spread on Dunn Field, may contain PCBs, pesticides/herbicides, and PCP.
 - a. Revise the SRI QAPP to include analytical sampling for PCBs, pesticides/herbicides, and PCP for soils associated with IDW or provide an explanation for why these analyses are not necessary.

Response: Army does not consider analysis of IDW soil samples for PCBs, pesticides/herbicides, and PCP to be necessary.

The MI ROD (Section 2.7.1) states the only contaminants of concern (COCs) posing unacceptably high potential risk to human health under industrial and recreational land use scenarios, which are the reasonably anticipated future land uses, are lead in soil within Functional Unit 4, and PCE and TCE in groundwater (Functional Unit 7). The area of lead-contaminated soil was remediated prior to execution of the ROD; completion of the remedial action in full coordination with EPA and TDEC was documented as a significant change in Section 2.13 of the MI ROD. Therefore, only CVOCs are COCs for soil cuttings from the Phase 3 monitoring wells. Industrial use is also the anticipated future land use for Dunn Field. Remedial actions for soil have been completed on Dunn Field and the applicable remediation goals (RGs) were met. The practice for the past several years has been to test samples of soil cuttings for VOCs and, if the RGs are met, the cuttings are spread on Dunn Field. This procedure has been described in work plans and reports approved by EPA.

5. Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times, Page 32: This worksheet lists the accreditation expiration dates for the labs Chemtech and Microbac for VOCs analysis for groundwater/water. However, the expiration date for Microbac's accreditation for VOCs in groundwater/water is listed as December 31, 2017, which is before fieldwork and groundwater sampling will be conducted for Phase 3 activities. Revise the SRI QAPP to address this issue.

Response: The worksheet will be revised to show the current expiration dates for laboratory accreditation. Microbac's expiration date is December 31, 2018 and Chemtech's expiration date is October 20, 2018.

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 and is inconsistent with EPA SESDPROC-105-R3, Groundwater Level and Well Depth Measurement guidance. 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 5.2.2, 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 considered to be maintained while using the Low-Flow method for groundwater sampling. Revise Section 5.2.2 to include the criteria by which water column stabilization is considered to be maintained throughout the sampling process.

Response: SOP-4, Section 5.2.2 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.

8. 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 SRI QAPP does not describe if wells at either the MI or at Dunn Field have dedicated pumps. Revise the SRI QAPP to describe the sampling method (i.e. dedicated pumps, passive diffusion bags, bailers, etc.) that is planned to be used at each well location.

Response: SOP-4 will be updated to describe planned sampling methods. Initial samples from the Phase 3 wells are planned to be collected using bladder pumps and low-flow sampling procedures; new bladders and tubing will be used for each well and stored for later use if necessary. PDBs will be installed in each well after the initial sample and the new wells will be incorporated in following LTM events. Samples at most LTM wells are collected using PDBs, and no wells currently have dedicated pumps.



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY REGION 4 ATLANTA FEDERAL CENTER 61 FORSYTH STREET ATLANTA GEORGIA 30303-8960

April 11, 2018

<u>UPS NEXT DAY AIR</u> RETURN RECEIPT REQUESTED

Mr. James Foster Base Realignment and Closure Division (ACSIM-ODB) 2530 Crystal Drive (Taylor Building), Room 5000 Arlington, VA 22202-3940

Dear Mt. Foster:

The U.S. Environmental Protection Agency (EPA) has received and reviewed the U.S. Department of the Army's Responses to EPA comments for the Defense Depot of Memphis, Tennessee Supplemental Remedial Investigation, Phase 3, Quality Assurance Project Plan (QAPP).

EPA acknowledges and appreciates the DDMT sites efforts throughout the Supplemental Remedial Investigative process and approves the above mentioned report.

Should you have any questions or concerns, please feel free to call me at on my cell number 404-229-9500.

Miedie Mad

Diedre Lloyd Remedial Project Manager Restoration & Sustainability Branch Region 4, Superfund Division

Mr. James Foster, (Signed Original), United Parcel Service, Return Receipt
 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



TENNESSEE DEPARTMENT OF ENVIRONMENT AND CONSERVATION MEMPHIS ENVIRONMENTAL FIELD OFFICE 8383 WOLF LAKE DRIVE BARTLETT, TN 38133-4119 PHONE (901) 371-3000 STATEWIDE 1-888-891-8332 FAX (901) 371-3170

January 11, 2018

James C. Foster BRAC Program Manager Headquarters Department of the Army, Assistant Chief of Staff for Installation Management (DAIM-ODB) Army Pentagon, 2530 Crystal Drive, Arlington, VA 22202-3934

Subject: Supplemental RI Phase 3 QAPP Defense Depot Memphis, Tennessee TDoR ID # 79-736

Mr. Foster,

TDEC-DoR has reviewed the contents of the **Supplemental RI Phase 3 QAPP** for the Memphis Defense Depot, as compiled by T. Holmes (HDR Inc), and approves of the proposals described in the document. 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 Division of Remediation Memphis Environmental Field Office

cc: Tom Holmes (HDR Inc) D. Lloyd (EPA-PM) Joan Hutton (CALIBRE) TDoR NCO: file 79-736 TDoR MEFO: file 79-736

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