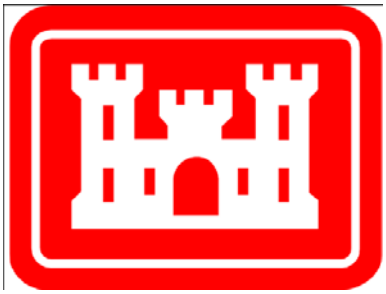
REMEDIAL ACTION OPERATIONS AND LONG TERM MONITORING QUALITY ASSURANCE PROJECT PLAN

Defense Depot Memphis, Tennessee
U.S. EPA I.D. Number TN4210020570

Prepared for:

Department of the Army



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Defense Depot Memphis, Tennessee

Prepared for:

U.S. Army Corps of Engineers, Tulsa District
Contract No. W90FYQ-09-D-0005
Task Order No. DS01

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LIST OF ACRONYMS AND ABBREVIATIONS

°C	degrees Celsius
ACSIM	Office of the Assistant Chief of Staff for Installation Management
ALS	ALS Global
AOC	Area of Concern
AS/SVE	Air Sparge Soil Vapor Extraction
BEC	Base Realignment and Closure Environmental Coordinator
bgs	below ground surface
BRAC	Base Realignment and Closure
CD	compact disc
cDCE	cis-1,2-dichloroethene
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CF	chloroform
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	Chain-of-Custody
CT	Carbon Tetrachloride
CVOC	Chlorinated Volatile Organic Compound
CWM	Chemical Warfare Material
CY	cubic yards
DA	Department of the Army
DDMT	Defense Depot Memphis, Tennessee
DLA	Defense Logistics Agency
DO	dissolved oxygen
DoD	Department of Defense
DQE	Data Quality Evaluation
DQI	Data Quality Indicator
DQO	Data Quality Objective
DSA	Diane Short & Associates
e^2M	engineering-environmental Management, Inc.
EBT	Enhanced Bioremediation Treatment
EDD	Electronic Data Deliverable

LIST OF ACRONYMS AND ABBREVIATIONS
(CONTINUED)

EE/CA	Engineering Evaluation and Cost Analysis
EISR	Early Implementation of Selected Remedy
ERPIMS	Environmental Restoration Program Information Management System
ET&D	Excavation, Transportation and Disposal
FFA	Federal Facilities Agreement
FSVE	Fluvial Soil Vapor Extraction
FTL	Field Team Leader
GIS	Geographic Information System
GS/MS	Gas Chromatography/Mass Spectrometry
HRS	Hazard Ranking System
HSWA	Hazardous and Solid Waste Amendment
IAQ	Intermediate Aquifer
lb/hr	pounds per hour
ID	Identification
IRA	Interim Remedial Action
IRACR	Interim Remedial Action Completion Report
IS	Internal Standard
IW	Injection Well
LCS	Laboratory Calibration Standard or Laboratory Control Sample
LIMS	Laboratory Information Management System
LTM	Long-Term Monitoring
LUC	Land Use Control
MAQ	Memphis Aquifer
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
MFA	Metabolic Fatty Acid
mg/L	milligrams per liter
MI	Main Installation
MLGW	Memphis Light Gas & Water
MNA	Monitoring Natural Attenuation
MPC	Measurement Performance Criteria
MSCHD	Memphis Shelby County Health Department

LIST OF ACRONYMS AND ABBREVIATIONS
(CONTINUED)

MS/MSD	Matrix Spike/Matrix Spike Duplicate
msl	mean sea level
MSR	Management Systems Review
MW	Monitoring Well
ND	Not Detected
NELAP	National Environmental Laboratory Accreditation Program
NPL	National Priorities List
ODB	Base Realignment and Closure Division
ODSVE	Off-Depot Soil Vapor Extraction
OPS	Operating Properly and Successfully
ORP	oxygen reduction potential
OU	Operable Unit
PARCCS	Precision, Accuracy/bias, Representativeness, Comparability, Completeness, and Sensitivity
PC	Project Chemist
PCB	polychlorinated biphenyls
PCE	tetrachloroethene
PCP	pentachlorophenol
PDB	passive diffusion bag
PID	Photoionization Detector
PM	Project Manager
PMW	Performance Monitoring Well
ppbv	parts per billion by volume
PQO	Project Quality Objectives
PRB	Permeable Reactive Barrier
PT	Performance Testing
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
RA	Remedial Action
RA-O	Remedial Action Operation

LIST OF ACRONYMS AND ABBREVIATIONS
(CONTINUED)

RAO	Remedial Action Objective
RA SAP	Remedial Action Sampling and Analysis Plan
RCRA	Resource Conservation and Recovery Act
RFA	RCRA Facility Assessment
RG	Remediation Goal
RIC	Reconstructed Total Ion Chromatogram
RL	Reporting Limit
ROD	Record of Decision
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
RW	Recovery Well
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SPD	Sampling Plan Details
SQL	Sample Quantitation Limit
SVE	Soil Vapor Extraction
SVOC	semi-volatile organic compound
SWMU	Solid Waste Management Unit
TA	Treatment Area
TCE	trichloroethene
TDEC	Tennessee Department of Environmental and Conservation
TeCA	1,1,2,2-tetrachloroethane
TOC	Total Organic Carbon
TSA	Technical Systems Audit
TSVE	Thermal Soil Vapor Extraction
TTA	Target Treatment Area
U	Undetected
UFP	Uniform Federal Policy
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
VC	Vinyl Chloride
VMP	Vapor Monitoring Wells

LIST OF ACRONYMS AND ABBREVIATIONS
(CONTINUED)

VOC	volatile organic compound
WMI	Waste Management, Inc.
ZVI	Zero Valent Iron
µg/L	micrograms per liter
%R	percent Recovery

1.0 PROJECT MANAGEMENT AND OBJECTIVES

1.1 INTRODUCTION

HDR has prepared this Quality Assurance Project Plan (QAPP) for Remedial Action Operations (RA-O) and Long Term Monitoring (LTM) at Defense Depot Memphis, Tennessee (DDMT) under Contract W90FYQ-09-D-0005, Task Order DS01 to the United States Army Corps of Engineers (USACE), Tulsa District. The environmental restoration program at DDMT is directed by the Department of the Army, Office of the Assistant Chief of Staff for Installation Management (ACSIM), Base Realignment and Closure Division (ODB). The regulatory oversight agencies are United States Environmental Protection Agency (USEPA) Region 4 and the Tennessee Department of Environment and Conservation (TDEC). DDMT has USEPA Identification Number TN4210020570.

DDMT was added to the National Priorities List in 1992 and environmental restoration is being conducted under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA). The decision documents for DDMT are complete and the selected remedies have been implemented. The *Preliminary Close Out Report* (USEPA, 2010) was approved in May 2010, and the DDMT National Priorities List (NPL) site status was revised to Construction Complete. Interim remedial action completion reports (IRACRs) have been approved for all actions. USEPA has concurred with operating properly and successfully (OPS) determinations for the remedies implemented on Federal property. The data collection activities for RA-O and LTM at DDMT are being conducted in accordance with approved plans.

This QAPP describes the planned environmental data collection activities and provides guidance for obtaining the type and quality of data needed to evaluate the effectiveness of RAs and to document the achievement of remediation goals. The QAPP replaces the *Remedial Action Sampling and Analysis Plan, Revision 1* (MACTEC, 2005a), *Volume 1: Field Sampling Plan* and *Volume 2: Quality Assurance Project Plan*. This QAPP has been prepared in general accordance with *Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP), Version 1, Part 1: UFP-QAPP Manual*, (Intergovernmental Data Quality Task Force, 2005). Worksheets from *Part 2A: UFP-QAPP Workbook* have been completed where applicable and are referenced in the text; all completed worksheets are included in Appendix A.

Review and approval of this QAPP is documented on Worksheet #1. QAPP Identifying Information is provided on Worksheet #2. The approved QAPP will be distributed to the individuals and organizations shown on Worksheet #3. The listed individuals are to ensure that necessary personnel review and understand the QAPP prior to beginning each phase of work. The HDR project manager (PM) will have

project staff and subcontractors review and sign-off on the applicable QAPP sections prior to each field investigation; review will be documented by Worksheet #4 and a copy will be provided to the HDR quality assurance (QA) manager.

1.2 PROJECT ORGANIZATION

The project organization chart is shown on Worksheet #5 shows reporting relationships between all organizations involved in the project, including the lead organization and all contractors and subcontractors.

Worksheets #6-Communication Pathways is not included; the required information is provided herein. Coordination between Department of the Army (DA), USEPA and TDEC is described in the *Federal Facilities Agreement between USEPA Region IV, TDEC, and Defense Logistics Agency at Defense Distribution Depot Memphis, Tennessee.* (FFA) (USEPA, 1995). Project documents are prepared under direction of the HDR PM and submitted to the DA PM and the Base Realignment and Closure (BRAC) Environmental Coordinator (BEC) and the USACE technical PM for review; following approval from DA, documents are submitted to USEPA and TDEC for review and comment. Communications between HDR and USEPA/TDEC are coordinated through DA.

Communications during field activities are coordinated by the HDR PM through the HDR project chemist (PC) and HDR field team leader (FTL). The HDR PC is the primary contact with the analytical laboratory and the data validation contractor. The HDR PC coordinates sampling tasks with the subcontract laboratory and the HDR FTL to ensure proper sample containers and other laboratory-supplied materials arrive at DDMT when needed and in the proper quantity. The HDR PC receives the analytical reports and transmits them to the data validation contractor, the HDR database/geographic information systems (GIS) manager and the PM. The HDR FTL directs field personnel and subcontractors (e.g. soil sampling or well installation) in sampling activities; field problems, including deviations from the work plans, are reported to the PC and PM for discussion and reporting to DA/USACE and to USEPA/TDEC as necessary.

Worksheet #7-Personnel Responsibility and Qualifications is attached. Worksheet 8-Special Personnel Training is not included; the required information is provided herein. HDR project personnel are selected based on education and experience necessary for the planned activities. Required health and safety training is documented in accordance with project specific health and safety plans; no other special training is required. Proper training of analytical laboratory personnel is in accordance with laboratory certification requirements. Additional information will be provided in project-specific work plans, where necessary.

1.3 PROJECT PLANNING AND PROBLEM DEFINITION

Based on the status of environmental restoration and the approved remedial action work plans and LTM plans, scoping sessions were not held. Worksheet #9-Scoping Session Participants is therefore not included.

The environmental questions to be answered by the RA-O and LTM activities covered in this QAPP are:

- Are remedies operating effectively and within discharge limits as applicable?
- Are remedies meeting cleanup goals, including objectives for active remedies and final remediation goals for groundwater specified in the Record of Decisions (RODs)?

The following sections describe the site history and regulatory status, environmental restoration activities performed to date, and the remedial action and monitoring activities covered by the QAPP. The information is summarized on Worksheet #10-Problem Definition.

1.3.1 Site Location and Description

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

DDMT covers approximately 632 acres and includes the Main Installation (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 north-northwest portion of the MI, contains approximately 65 acres and includes former mineral storage and waste disposal areas.

1.3.2 Regulatory Status

In 1990, USEPA Region 4 and TDEC issued the Depot a Resource Conservation and Recovery Act (RCRA) Part B permit for the storage of hazardous waste (No. TN 4210020570). The Hazardous and Solid Waste Amendment (HSWA) portion of the permit issued by USEPA included requirements for the identification and, if necessary, corrective action of Solid Waste Management Units (SWMUs) and Areas of Concern (AOCs). A RCRA Facility Assessment completed in 1990 identified 49 SWMUs and 8 AOCs. Subsequent to issuing the RCRA permit, USEPA prepared a final Hazard Ranking System (HRS)

Scoring Package for the facility. In October 1992, USEPA added the Depot to the NPL (57 Federal Register 47180 No. 199).

On 6 March 1995, USEPA, TDEC, and the Depot entered into the FFA (USEPA, 1995) under CERCLA, Section 120, and RCRA, Sections 3008(h) and 3004(u) and (v). The FFA outlines the process for investigation and cleanup of the Depot sites under CERCLA. The parties agreed that investigation and cleanup of releases from the sites (including formerly identified SWMUs/AOCs) would satisfy any RCRA corrective action obligation.

Site designations were developed for overlapping environmental programs and for facility reuse. Environmental restoration sites were first identified during the 1990 RCRA Facility Assessment (RFA), and additional sites were added over time. During FFA negotiations after DDMT was placed on the NPL, the Depot was divided into four Operable Units (OUs): Dunn Field, OU 1; Southwest Quadrant MI, OU 2; Southeastern Watershed and Golf Course, OU 3; and North-Central Area MI, OU 4.

Upon completion of the Air Sparging and Soil Vapor Extraction (AS/SVE) system for the Dunn Field Off Depot area in 2009, construction of the selected remedies was completed. The *Preliminary Close Out Report* (USEPA, 2010) was approved in May 2010 and the DDMT NPL site status was revised to Construction Complete.

1.3.3 Geology and Hydrogeology

The geologic units of interest at Dunn Field are (from youngest to oldest): loess, including surface soil; fluvial deposits; Jackson Formation/Upper Claiborne Group; 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 feet thick and are continuous throughout the Dunn Field area.

The fluvial (terrace) deposits consist of two general layers. The upper layer is a silty, sandy clay that transitions to a clayey sand and ranges from about 10 to 36 feet thick. The lower layer is composed of interlayered sand, sandy gravel, and gravelly sand, and has an average thickness of approximately 40 feet. 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 of the fluvial aquifer ranges from 3 to 50 feet and is controlled by the configuration of the uppermost clay in the Jackson Formation/Upper Claiborne Group. The groundwater in the fluvial aquifer is not a drinking water source for area residents but is classified by TDEC as General Use (TDEC Chapter 1200-04-03).

The Jackson Formation/Upper Claiborne Group consists of clays, silts, and sands. The uppermost clay unit appears to be continuous, except in the southwestern area of Dunn Field. Off site, to the west and northwest of Dunn Field, there are possible gaps in the clay. Where present, these gaps create connections to the underlying intermediate aquifer (IAQ) from the fluvial deposits. The IAQ is locally developed in deposits of the Jackson Formation/Upper Claiborne Group.

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 feet in thickness, and begins at a depth below ground surface (bgs) of approximately 120 to 300 feet. The top of the Memphis Sand was identified at 255 feet bgs (elevation of 21 feet above mean sea level [msl]) in monitoring well (MW)-67, the first monitoring well completed in the Memphis Sand at DDMT. The Memphis aquifer (MAQ) is confined by overlying clays and silts in the Cook Mountain Formation (part of the Jackson/Upper Claiborne Group) and contains groundwater under strong artesian (confined) conditions regionally. The City of Memphis obtains the majority of its drinking water from this unit. The Allen Well Field, which is operated by Memphis Light Gas & Water (MLGW), is located approximately two miles west of Dunn Field.

1.3.4 Main Installation (OU-2, 3 and 4)

The MI contains approximately 567 acres with open storage areas, warehouses, former military family housing, and outdoor recreational areas. Investigations from 1989 to 2001 identified contamination in surface soil and ground water. Surface soil contamination requiring response consisted primarily of metals, polychlorinated biphenyls (PCBs), semi-volatile organic compounds (SVOCs), and a pesticide, dieldrin. Groundwater contamination requiring response was limited to chlorinated volatile organic compounds (CVOCs) primarily tetrachloroethene (PCE), trichloroethene (TCE), carbon tetrachloride (CT), and chloroform (CF).

1.3.4.1 Prior Removal Activities

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

- Approximately 602 cubic yards (CY) of surface and subsurface soil was removed from the pentachlorophenol (PCP) dip vat area (Building 737) because of elevated levels of PCP (completed in 1985).
- Approximately 60,000 gallons of hazardous and petroleum/oil/lubricants materials from damaged drums were reclaimed and repackaged at Building 873 in 1985. Approximately 800 55-gallon

drums were recouped in this open storage area and then returned to their original location for storage and distribution.

- Approximately 5,000 tons (3,700 CY) of surface soil in the Housing Area was removed because of the presence of dieldrin (began in June 1998; completed in October 1998). The Housing Area is an exception to the overall industrial land use for MI and remediation levels were based on residential reuse.
- Approximately 530 tons (400 CY) of surface soil surrounding the cafeteria (Building 274) was removed because of elevated levels of polychlorinated biphenyls (began in October 1998; completed in November 1998).
- Approximately 980 CY of surface and subsurface soil from near Buildings 1084, 1085, 1087, 1088, 1089 and 1090 was removed because of elevated levels of metals and polyaromatic hydrocarbons (began in May 2000; completed in August 2000).

1.3.4.2 Record of Decision

The *Memphis Depot Main Installation Record of Decision* (MI ROD) (CH2M HILL, 2001) includes OUs 2 (Southwest Quadrant MI), 3 (Southeastern Watershed and Golf Course), and 4 (North-Central Area MI). The MI ROD received final approval on 6 September 2001. The remedial action objectives (RAOs) are:

<u>Surface Soil</u>

- to prevent direct contact/ ingestion of surface soils contaminated with lead in excess of industrial worker risk-based criteria.
- to prevent direct contact/ ingestion of surface soils contaminated with dieldrin and arsenic in excess of human health risk assessment criteria for residents.
- to prevent direct contact/ ingestion of surface soils contaminated with lead in excess of risk-based criteria for protection of residential children.

<u>Groundwater</u>

- to prevent human ingestion of water contaminated with volatile organic compounds (VOCs) in excess of maximum contaminant levels (MCLs) from potential future onsite wells.
- to reduce concentrations of contaminants of concern to MCLs or lower.

- to prevent horizontal and vertical offsite migration of groundwater contaminants in excess of MCLs.

The selected remedy contained the following components:

- Excavation, transport and offsite disposal of lead contaminated surface soil near Building 949.
- Deed restrictions and site controls in the form of land use controls (LUCs) to prevent residential land use on the MI, except at the existing housing area; daycare restriction controls; production/consumptive use groundwater controls for the fluvial aquifer and for drilling into deeper aquifers on the MI; and elimination of casual access through maintenance of a boundary fence around the golf course.
- Enhanced bioremediation treatment (EBT) of CVOCs in the most contaminated part of the groundwater plume.
- Long-term groundwater monitoring to document changes in plume concentrations and to detect potential plume migration to off-site areas or into deeper aquifers.

The area of lead contamination in soil near Building 949 (approximately 300 CY) was excavated and disposed off-site prior to final execution of the ROD. The action was taken under Defense Logistic Agency's (DLA's) removal authority under CERCLA Section 104 in order to accommodate the economic redevelopment of the site. The action was documented in *Remediation Report, Removal Action at Building 949* (Jacobs Federal Programs, 2002) and noted as a significant change in the ROD.

1.3.4.3 Remedial Actions

The EBT system was constructed from May to August 2006. Construction included installation of 49 injection wells (IWs) and 30 performance monitoring wells (PMWs), construction of the lactate-storage and transfer facility, construction of two trailer-mounted injection systems, and baseline groundwater sampling and analysis.

Sodium lactate was injected biweekly to monthly from September 2006 through February 2009. Changes to injection procedures were made based on field observations and measurements. Performance monitoring was performed quarterly from October 2006 through March 2009. System operations and monitoring results were described in annual reports. CVOC concentrations for parent compounds (PCE, TCE, CT and CF) were reduced over 90 percent in injection wells and over 80 percent in monitoring wells at locations with baseline concentrations above 100 micrograms per liter (µg/L).

The *Main Installation Interim Remedial Action Completion Report, Revision 1* (MI IRACR) (HDR|engineering environmental Management, Inc. [e^2M], 2010), including an OPS determination, was submitted to USEPA and TDEC in February 2010. Although EBT did not achieve the goal of reducing concentrations below MCLs, additional field investigation, groundwater modeling and trend analysis presented in the MI IRACR indicated that additional remedial action (RA) was not necessary. The OPS determination and the MI IRACR were approved by USEPA in March 2010. Additional monitoring wells installed in the IAQ and the upper portion of the MAQ supported the groundwater model results.

EBT components were planned to be removed following a period of monitoring after the last injection in February 2009. However, system removal and well abandonment was delayed because of rebound in CVOC concentrations observed in LTM samples. New baseline groundwater samples were collected from IWs and PMWs in December 2011. The sample results confirmed rebound in concentrations of parent compounds in the target treatment areas (TTAs), and additional EBT was recommended utilizing some of the existing IWs and PMWs. Following consultation with USEPA and TDEC, 20 IWs and 11 PMWs not needed for additional EBT were abandoned in June 2012.

The *Main Installation Remedial Action Work Plan Addendum, Revision 0* (MI RAWP Addendum) (HDR, 2012a) describing procedures for a new round of EBT was approved by USEPA and TDEC in January 2013. EBT is being used in five areas where individual CVOC concentrations of parent compounds (PCE, TCE and CT) exceed 100 µg/L: TTA-1N, TTA-1S, TTA-2, the West-Central plume and the Building 835 plume. Additional EBT was initiated in November 2012 and is being implemented through quarterly injections of sodium lactate solution into the fluvial aquifer.

1.3.5 Dunn Field (OU-1)

Dunn Field, which is located across Dunn Avenue from the MI, contains approximately 64 acres and includes former mineral storage and waste disposal areas. Soil samples collected for the RI showed significant levels of CVOCs: 1,1,2,2-tetrachloroethane (TeCA); 1,2-dichloroethane; total 1,2-dichloroethene; CT; CF; methylene chloride; PCE; TCE; and vinyl chloride (VC). TCE and TeCA were the CVOCs most frequently detected in soil samples at elevated concentrations. Three contaminant plumes in the fluvial aquifer were identified at Dunn Field. The CVOCs detected in soil samples were also detected most frequently in groundwater samples. The individual CVOCs with the highest concentrations were TeCA and TCE.

1.3.5.1 Prior Removal and Remedial Activities

The *Record of Decision for Interim Remedial Action of the Groundwater at Dunn Field (OU-1)* (CH2M HILL, 1996) was signed in April 1996, with the objective of hydraulic containment to prevent further contaminant plume migration and reduce contaminant mass in groundwater. The interim remedial action (IRA) groundwater recovery system included 11 recovery wells (RWs) screened in the fluvial aquifer and located along the western boundary of Dunn Field. The system began operation in November 1998 with groundwater discharge to the city sewer system without treatment under an industrial discharge agreement. Based on reduction of CVOC concentrations in groundwater following implementation of the Source Areas RA, the final RWs were shutdown in January 2009. Approximately 918 pounds of total VOCs, including 369 pounds of TCE, were discharged by the IRA in just over 10 years of operation. The IRA system was removed and the RWs abandoned in July 2010. The final year of IRA groundwater monitoring and closure activities were described in the *Dunn Field Groundwater Interim Remedial Action 2009 Operations and Closure Report* (HDR, 2010).

Following completion of an engineering evaluation and cost analysis (EE/CA), a non-time critical removal action was conducted to reduce or eliminate the potential risk posed by chemical warfare material (CWM) wastes at Sites 1, 24-A, and 24-B. The removal action was completed in March 2001 and documented in the *Final Chemical Warfare Materiel Investigation/Removal Action Report* (UXB, 2001). Approximately 914 CY of soil contaminated with mustard degradation by-products, and 19 CY of mustard-contaminated soil were excavated, transported, and disposed offsite. Twenty-nine bomb casings were recovered from Site 24-A.

A non-time critical removal action to address lead contaminated surface soil at Site 60, a former pistol range in the Northeast Open Area, was completed in March 2003, pursuant to an EE/CA completed in July 2002. Approximately 930 CY of lead contaminated surface soil was excavated, transported, and disposed offsite at an approved, permitted landfill.

Locations of the pre-ROD removal actions and the IRA are shown on Figure 3.

1.3.5.2 Record of Decision and Amendment

The *Memphis Depot Dunn Field Record of Decision* (Dunn Field ROD) (CH2M HILL, 2004a) for OU 1, the only known and documented waste burial area, was finalized in April 2004. Remediation goals (RGs) for the contaminants of concern are shown on Table 1. The RAOs and the selected remedy address surface soil, material within disposal sites and associated soil, and CVOCs in subsurface soil and groundwater. The RAOs are:

Surface Soil

- Limit use of the surface soil in the Disposal Area to activities consistent with light industrial land use and prevent residential use through land controls

Disposal Sites

- Prevent groundwater impacts from a release of buried containerized hazardous liquids and the leaching of contaminants from buried hazardous solids
- Prevent unacceptable risk of direct contact with buried hazardous liquids and/or solids due to intrusive activities during future land use or site development

Subsurface Soil Impacted with VOCs

- Prevent direct inhalation of indoor air vapors from subsurface soils in excess of industrial worker criteria
- Reduce or eliminate further impacts to the shallow fluvial aquifer from VOCs in the subsurface soil

Groundwater

- Prevent human exposure to contaminated groundwater (i.e., exceeding protective target concentrations)
- Prevent further off-site migration of VOCs in excess of protective target levels
- Remediate fluvial aquifer groundwater to drinking water quality to be protective of the deeper MAQ

The components of the selected remedy from the Dunn Field ROD are:

- Excavation, transportation, and disposal (ET&D) of soil and material contained within disposal sites based upon results from a pre-design investigation
- Soil vapor extraction (SVE) to reduce VOC concentrations in subsurface soils to levels that are protective of the intended land use and groundwater
- Injection of zero valent iron (ZVI) within Dunn Field to treat CVOCs in the most contaminated part of the groundwater plume, and installation of a permeable reactive barrier (PRB) to remediate CVOCs within the off-site areas of the groundwater plume

- Monitored natural attenuation (MNA) and LTM of groundwater to document changes in plume concentrations, detect potential plume migration to off-site areas or into deeper aquifers, and track progress toward RGs
- Implementation of LUCs, which consist of the following institutional controls: Deed and/or lease restrictions; Notice of Land Use Restrictions; City of Memphis/Shelby County zoning restrictions and the Memphis and Shelby County Health Department (MSCHD) groundwater well restrictions.

The selected remedies were modified through the *Dunn Field Record of Decision Amendment 3* (ROD Amendment) (engineering-environmental Management, Inc. [e^{2}M], 2009a) approved in January 2009. The fundamental change was the use of AS/SVE instead of a PRB for the Off Depot groundwater plume. The ROD Amendment also revised the criteria for extent of the AS/SVE system and clarified the treatment objective. The AS/SVE system was selected to cross the core of the plume near the downgradient end and to reduce the individual CVOC concentrations in groundwater to 50 µg/L or less. Groundwater modeling results indicate that the AS/SVE system in combination with natural attenuation processes would reduce groundwater concentrations to MCLs in accordance with the RAOs within a reasonable period of time. The RGs for the contaminants of concern were not changed

1.3.5.3 Remedial Actions

Three RAs were planned to implement the selected remedies for OU 1, Dunn Field: Disposal Sites RA (ET&D); Source Areas RA (SVE, ZVI injections and LUCs); and Off-Depot Groundwater RA (AS/SVE, MNA, and LTM). Locations of the Disposal Sites RA are shown on Figure 3, and locations of the Source Areas and Off-Depot Groundwater RAs are shown on Figure 4.

1.3.5.3.1 Disposal Sites Remedial Action

The Disposal Sites RA was performed during two separate mobilizations. During the first mobilization from March 2005 to May 2005, Disposal Sites 4.1, 13, 31, and the majority of Disposal Site 10 were completed. Disposal Site 3 and the remaining materials from Disposal Site 10 were completed during the second mobilization in February and March 2006. Approximately 2,700 CY of non-hazardous materials were transported off-site and disposed of at the BFI South Shelby County Landfill. Approximately 234 CY of hazardous materials from Disposal Site 3 was disposed at the Clean Harbors Lambton Secure Landfill in Canada. The confirmation samples met the remediation goals at each excavation. The *Dunn Field Disposal Site RA Completion Report* (MACTEC, 2006) was approved by USEPA on 25 August 2006.

1.3.5.3.2 Early Implementation of Selected Remedy

An Early Implementation of Selected Remedy (EISR) using ZVI was performed to reduce contaminant mass downgradient of the planned PRB location in order to ensure that the portion of the plume slated for MNA in the ROD was not unduly extensive or high in concentration. ZVI injections were made November 2004 to January 2005. Injections were made in 14 borings at 2-foot intervals over the fluvial aquifer thickness; the injection locations were spaced approximately 60 to 80 feet apart. The total mass of ZVI injected was approximately 192,500 pounds. The *EISR Interim Remedial Action Completion Report* (MACTEC, 2005b) noted that the injections did not achieve the goal of 90 percent or greater reduction of TCE and 1,1,2,2 tetrachloroethane and included recommendations for decreased spacing between injection locations to achieve increased reduction in CVOCs. The report was approved by USEPA in September 2005.

1.3.5.3.3 Source Areas Remedial Action

The Source Areas RA included conventional SVE in the coarse-grained fluvial soils; ET&D for two shallow areas containing waste materials (treatment area [TA]-1F) and buried drums containing petroleum hydrocarbons (TA-3); thermal SVE (TSVE) (in situ thermal desorption) in the fine grained loess; and ZVI injection in the fluvial aquifer.

The Fluvial SVE (FSVE) system was installed to remove CVOCs from the fluvial sands at Dunn Field. The system consists of two blowers connected to seven SVE wells with screened intervals at approximately 30 to 70 feet bgs, 20 vapor monitoring points (VMPs) located 15 to 80 feet from the SVE wells and an equipment building for the blowers, heat exchangers and controls. Ten additional SVE wells were installed in borings for confirmation soil samples in November 2010. The FSVE system began operation in July 2007 and was shutdown in July 2012 after soil RGs were met. Approximately 4,000 pounds of VOCs were removed during system operations.

The initial excavations at TA-1F and TA-3 were performed October 2007 to January 2008. Further excavation was delayed in order to proceed with construction and operation of the TSVE system. The excavations were completed February to June 2009. Approximately 7,400 CY of waste material were disposed as non-hazardous waste at the Waste Management, Inc (WMI) landfill in Tunica MS, a CERCLA-approved facility. Soil confirmation samples met remediation goals in both areas.

TSVE treatment was performed in four areas with a total area of about 1.25 acres and a treatment interval of approximately 5 to 30 feet bgs. System components included 367 heater-only wells; 68 vapor extraction wells, 62 multi-level temperature monitoring points, 25 pressure monitoring points and a

Shotcrete surface cover to limit water infiltration and improve vapor capture. The system operated continuously from May until the heaters were shutdown in the final TA in November 2008. Approximately 12,500 pounds of VOCs were removed during treatment. Confirmation soil samples, collected at various depths from 35 soil borings, demonstrated that clean-up standards were met; the average concentration for CVOCs in each TA was below the RG and none of the final samples exceeded an RG by a factor of 10 or more.

ZVI injections were not required because groundwater objectives for the Source Areas remedy were achieved through the subsurface soil remedies.

The memorandum, *Operating Properly and Successfully Demonstration, Source Areas Remedial Action* (e^2M, 2009b), was approved by USEPA on 21 October 2009. The *Source Areas Interim Remedial Action Completion Report, Rev. 1* (HDR|e^2M, 2009) was approved by USEPA on 2 November and by TDEC on 10 November 2009.

CVOC concentrations in groundwater began to decrease significantly after FSVE start-up, indicating that further contaminant migration from subsurface soils to groundwater was prevented and that continuing sources of contamination, such as pockets of free product below the water table, were not present. Reduction in CVOC concentrations is shown in total CVOC plume maps for April 2007, April 2009, April 2011, and April 2013 on Figure 5.

1.3.5.3.4 Off Depot Remedial Action

AS/SVE is being conducted near the leading edge of the groundwater plume west of Dunn Field to volatilize CVOCs and prevent further plume migration. The AS/SVE system consists of 90 AS points, 12 SVE wells, 10 pairs of VMPs and control buildings for the AS compressor, SVE blowers and system controls. AS/SVE operations began 21 December 2009. The *Off Depot Groundwater Interim Remedial Action Completion Report, Rev. 1* (HDR, 2011a) was approved by USEPA in August 2011. Approximately 83 pounds of VOCs were removed through December 2013 and CVOC concentrations in downgradient wells were reduced below the treatment goal of 50 µg/L for individual CVOCs.

1.3.6 Remedial Action Operations and Long-Term Monitoring

The FSVE system was shutdown in 2012 but remains in place in the event concentrations rebound above 50 µg/L for individual CVOCs, and operations are resumed. RA-O is continuing for AS/SVE in the Off Depot area and EBT on the MI. LTM is being performed for the MI and Dunn Field to evaluate progress

toward the groundwater RAOs. Performance monitoring data for EBT and the AS/SVE systems and LTM results are used to evaluate continuing operations and to recommend changes.

1.3.6.1 Fluvial SVE

The FSVE system was shutdown in July 2012 after emissions reduced asymptotically below 1 ppm and soil RGs were met in confirmation samples. The system operated under MSCHD Permit #01030-01P, which required VOC emissions below 5.71 pounds per hour (lb/hr) or 25 tons per year with documentation provided in an annual emissions report. Following shutdown, the conveyance lines were closed, and the air intakes and exhausts for the blowers were sealed. BaroBall™ caps were installed on 11 SVE wells for increased efficiency during passive venting; the other FSVE wells were sealed. The final year of system operations and monitoring was described in *Dunn Field Source Areas Fluvial Soil Vapor Extraction System Annual Operations Report, Year Five* (HDR, 2012b).

Vadose zone modeling (SESOIL) was used to estimate the time required for migration of CVOCs (TCE and TeCA) from the loess to groundwater in order to provide a conservative estimate of the groundwater monitoring period necessary following shutdown of the SVE system. The model indicated rebound in CVOC concentrations in groundwater due to leachate from the fluvial sand would be observed 60 to 90 days after shutdown, but maximum concentrations due to leachate from the loess would not be observed for two to four years.

Fourteen LTM wells were selected for monitoring rebound after shutdown of the FSVE system and were designated Performance-FSVE wells. Only two of these wells (MW-03 and MW-220) have exceeded an MCL since rebound monitoring began in 2012; both wells are in the northwest corner of Dunn Field and are impacted by an off-site plume originating northeast of Dunn Field. An increase in chloroform concentrations in two of the wells (MW-06 and MW-87) in April 2014 is considered a possible indication of rebound. The FSVE system and the Performance-FSVE wells are shown on Figure 6.

1.3.6.2 AS/SVE

The AS/SVE system consists of 90 AS points, 12 SVE wells, 10 pairs of VMPs and control buildings for the AS compressor, SVE blowers and system controls. The system design was based on pulsed AS operation with 1/3 of the 90 AS wells operating at one time with all 12 SVE wells online (Figure 7). The system is operated through programmable logic controllers and each AS group is operated for four hours before the system switches to the next group. AS/SVE operations were incorporated in MSCHD Permit #01030-01P issued for the FSVE with permit conditions applicable to the combined operations. System operations and monitoring during 2013 is described in *Off Depot Air Sparge - Soil Vapor Extraction*

System Annual Operations Report, Year Four (HDR, 2014a). The *Dunn Field Off Depot Groundwater Air Sparge and Soil Vapor Extraction System Operations and Maintenance Manual* (HDR, 2011b) describes system components, maintenance requirements and operating procedures and contains as-built drawings of system components, equipment specifications and manufacturer's equipment manuals.

System operations have been adjusted periodically to reduce potential for plume diversion and to target areas with higher CVOC concentrations. At present, the AS system operates12 hours per day with 48 AS points open 4 hours per day (full-time), 18 AS points open 2 hours per day (half-time) and 24 AS points closed. One SVE blower operates 24 hours per day with all 12 SVE wells open. The AS/SVE system layout is shown on Figure 7.

System uptime averaged 95% from startup in December 2009 through December 2013. The system has been down since February 2014 due to equipment damage from a power surge during a thunder storm. Equipment repairs and installation of an electrical surge suppression system are in progress; the AS/SVE system is expected to be restarted in December 2014.

AS/SVE system operations and performance monitoring include the following activities:

- Monthly system inspections for repair or replacement of components as required and for system readings to include:
  - Pressure, temperature, air flow rates and operating hours at the AS compressor and the SVE blowers;
  - Air pressure and flow rates at operating AS wells;
  - Air flow rate, vacuum and photoionization detector (PID) measurements at SVE wells and system effluent;
  - PID and vacuum measurements at VMPs.
- Quarterly laboratory samples from system influent analyzed for VOCs.

The SVE wells are adjusted at the manifold to balance individual flow rates. System flow rates are measured using a pitot tube and flow rates at individual wells are measured by vane-type meters at the well manifold. Vacuum measurements are made using a digital manometer. The VOC mass removed by the AS/SVE system is estimated from the average VOC concentrations in the effluent sample, system operating hours and flow rates.

Overall effectiveness of the AS/SVE system is evaluated through LTM (Section 1.3.6.3). The treatment goal for the AS/SVE system is to reduce groundwater concentrations downgradient of AS/SVE barrier below 50 µg/L for individual CVOCs. The AS/SVE system will be shutdown when upgradient groundwater concentrations reach 50 µg/L for individual CVOCs. Further treatment should not be necessary unless upgradient concentrations rebound.

CVOC concentrations in all wells downgradient of the AS/SVE system have been below 50 µg/L for individual CVOCs since 2010. The only LTM well in the Source Areas on Dunn Field or the Off Depot area to exceed the active treatment objective since April 2012 has been MW-159, which is located just upgradient of the AS/SVE system. Once equipment repairs are completed, the AS/SVE system will be operated in alternating months to restore the northerly groundwater flow observed prior to system operation. That will allow groundwater to flow from MW-159 into the treatment zone.

1.3.6.3 EBT

EBT is being used in five areas where individual CVOC concentrations of parent compounds (PCE, TCE and CT) exceed 100 µg/L: TTA-1N, TTA-1S, TTA-2, the West-Central plume and the Building 835 plume. Field activities consist of quarterly performance monitoring followed by injections of sodium lactate solution into the fluvial aquifer. There are 30 4-inch IWs and 15 2-inch wells used for injections, and 13 2-inch wells used as PMWs. The 58 EBT wells are listed on Table 2 and the well locations are shown on Figure 8.

Additional EBT was performed for two years starting in November 2012. Quarterly sodium lactate injections were conducted through August 2014 and performance monitoring through November 2014. Field activities and monitoring results to date are described in *Main Installation Year Three Enhanced Bioremediation Treatment Report* (HDR, 2014b). The *Main Installation Remedial Action Enhanced Bioremediation Treatment Operations and Maintenance Manual* (e^2M, 2007) describes system operations and maintenance requirements and contains as-built drawings of system components, equipment specifications and manufacturer's equipment manuals.

Each monitoring/injection event includes the following activities:

- Clear access to injection and monitoring well locations.
- Measure water quality and collect groundwater samples at all IWs and PMWs.
- Prepare sodium lactate injection fluid in the trailer-mounted, 500-gallon storage tank.
- Inject sodium lactate solution at designated wells.

- Clean injection trailers and transfer area with Alconox/water solution.

Performance monitoring is used to evaluate success in expanding anaerobic conditions within the fluvial aquifer in the EBT zones and in decreasing CVOC concentrations through reductive dechlorination. Monitoring includes field measurements of water quality and groundwater sampling at the IWs and PMWs. Water quality parameters are dissolved oxygen (DO), oxygen reduction potential (ORP), pH, temperature, and conductivity. Groundwater samples are analyzed for VOCs, total organic carbon (TOC), metabolic fatty acids and dissolved gases.

An injection plan is developed for each quarterly event based on ORP measurements for the previous and current events, and the analytical results and lactate volume from the previous quarter. The target volume for injections during November 2012 and February 2013 was 250 gallons per IW with lactate concentrations of 15 to 21 percent. The injection volumes were increased and lactate concentrations decreased during later events to improve distribution of the lactate; the target volume was 350 to 500 gallons per IW with lactate concentrations of 6 to 12 percent.

The RAOs for groundwater at the MI include reduction in concentrations of CVOCs to MCLs or lower. Comparison of results from the updated baseline samples in December 2011 and the EBT-14 samples in November 2013 showed decreases of PCE, TCE and CT above 80% in IWs and 30% to 73% in PMWs. The number of EBT wells with concentrations above the MCL decreased from 55 in December 2011 to 30 wells in November 2013. The number of wells exceeding MCLs for parent CVOCs decreased from 51 to 18 for PCE, from 31 to 9 for TCE and from 8 to 1 for CT; there was a corresponding but lesser increase in wells exceeding MCLs for daughter CVOCs, with an increase from 7 to 13 for cis-1,2-dichloroethene (cDCE) and from 1 to 3 for VC. There was good correlation with reductive dechlorination and TOC concentrations above 50 milligrams per liter (mg/L). Overall progress toward RAOs will be evaluated through LTM.

1.3.6.4 Dunn Field LTM

Dunn Field LTM is being performed to evaluate overall progress toward the groundwater RAOs and in accordance with the LTM Plan in Appendix C of the *Off Depot Groundwater Final Remedial Design, Rev. 1* (CH2M HILL, 2008). Recommendations for changes to LTM wells and sample frequency are made in annual LTM reports.

The LTM plan classified the monitoring wells in three categories:

- Background – wells screened in the fluvial aquifer located along or outside of the Dunn Field boundary; located upgradient to or at a distance from contaminant plumes on Dunn Field; no (or only low-level) previous detections of site contaminants in well samples.
- Sentinel – wells screened within either the fluvial or intermediate aquifers adjacent to or within the window to the IAQ.
- Performance – wells screened in the fluvial aquifer; located within the limits of known contaminant plumes; or repeatedly have contaminants in samples; located in areas targeted for treatment during the RA.

Dunn Field LTM includes 86 wells classified as Background (8), Background-NE (5), Performance (50), Performance-FSVE (14) or Sentinel (9). The Background-NE wells are located on or bordering the northeast section of Dunn Field and have CVOC concentrations from an off-site source(s) upgradient of Dunn Field. The performance-FSVE wells are performance wells selected for rebound monitoring after shut down of the FSVE system in July 2012. The LTM wells have the following sample frequency: semiannual (42), annual (29) or biennial (15) sample frequencies. The Dunn Field LTM wells are listed on Table 3 and the locations are shown on Figure 9.

TeCA, TCE and PCE isoconcentration maps for the April 2014 LTM samples are shown on Figures 10, 11 and 12. In April 2014, 13 performance wells and 5 Background-NE had concentrations above the MCL or TC for the most commonly detected CVOCs (TeCA, TCE, PCE, DCE and CF).

1.3.6.5 MI LTM

MI LTM is performed to evaluate overall progress toward the groundwater RAOs and in accordance with the LTM plan in Appendix B of *Main Installation Final Remedial Design, Rev. 1* (CH2MHILL, 2004b). Recommendations for changes to LTM wells and sample frequency are made in annual LTM reports.

MI LTM wells are classified in four categories:

- Background – wells screened in the fluvial aquifer located along or outside the MI boundary; wells upgradient of or at a distance from groundwater plumes on the MI and Dunn Field; and wells with no, or low, previous detections of site constituents.
- Boundary – wells screened in the fluvial aquifer located along or outside the MI boundary to monitor constituent migration from off-site sources.
- Sentinel – wells screened within either the fluvial or intermediate aquifer adjacent to or within the window to the IAQ.

- Performance – wells screened in the fluvial aquifer and within the limits of known groundwater plumes.

MI LTM includes 99 wells classified as Background (6), Boundary (7), Performance (62) and Sentinel (24). The number of performance wells changed in 2013; 20 LTM wells are being used for EBT injections and performance monitoring and 5 former EBT PMWs were added to LTM. Two wells were installed in 2013, and their sample frequency will be selected in accordance with the LTM plan after four semiannual events. The remaining wells have the following sample frequency: biennial (13), annual (33) and semiannual (59). The MI LTM wells are listed on Table 4 and are shown on Figure 13.

There are 57 LTM performance wells screened in the fluvial aquifer within the limits of seven designated groundwater plumes: TTA-1 North, TTA-1 South, TTA-2, West Central, Building 835, North Central and South-Central. The designated plumes, primary CVOCs and associated LTM wells are listed below.

Plume	CVOC	LTM Wells
TTA-1 North	PCE	DR1-2, DR1-7, DR1-8, MW-21, MW-100B, PMW21-03 and PMW21-05
TTA-1 South	PCE, TCE	DR1-1/1A, DR1-3, DR1-4, MW-101, PMW101-03A/B, PMW101-06A/B
TTA-2	PCE, CT, CF	DR2-1, DR2-3, DR2-4, DR2-6, MW-26, MW-64, MW-88, MW-92, MW-96, MW-217, MW-218, MW-259, PMW92-02
West-Central	PCE	MW-39/39A, MW-94A, MW-98, MW-200, MW-204A/B, MW-205A/B, MW-206A/B, MW-208A/B, MW-210B
Bldg 835	TCE	MW-142, MW-143, MW-198, MW199B, MW-209B, MW-257
North-Central	TCE	MW-103, MW-104, MW-214A/B, MW-258, MW-260
South-Central	TCE	MW-97, MW-216, MW-261

There are five LTM performance wells outside the designated plumes (MW-25A, MW-52, MW-215A/B and PZ-03).

PCE and TCE isoconcentration maps for the October 2013 LTM samples are shown on Figures 14 and 15. In 2013, 55 LTM wells had concentrations above the MCL for one or more parent CVOCs (PCE, TCE and CT); one additional well exceeded only the MCL for cDCE. There are 48 LTM performance wells in the five areas where EBT is currently being performed, and 34 of these wells exceeded MCLs. In addition to the 14 performance wells associated with the North-Central and South-Central areas or in isolated locations, many of the wells associated with the current EBT areas may be outside the TAs.

1.3.6.6 Main Installation Supplemental Remedial Investigation and Focused Feasibility Study

The latest five-year review for DDMT, *Third Five-Year Review, Revision 1* (HDR, 2012d), identified two issues, both related to the MI: rebound in CVOC concentrations and the time required to achieve groundwater RAOs. Additional EBT was initiated in November 2012 to address the rebound. The schedule to achieve RAOs on the MI was addressed in the Year Three EBT report (HDR, 2014a), which stated it was unlikely that contaminant concentrations would be reduced below MCLs by the target date of December 2015. The 2013 LTM report (HDR, 2014b) also addressed the schedule for meeting RAOs on the MI and included a general recommendation for additional wells to improve delineation of contaminant plumes and to clarify site hydrogeology.

A USEPA comment on the 2013 LTM report stated a ROD Amendment or Explanation of Significant Differences (ESD) may be necessary to address: the need for response action in the IAQ; to clarify selection of monitored natural attenuation as an MI remedy component for fluvial groundwater; to address potential on-site impacts from a possible southeast off-site location; and possibly to select response actions in addition to, or other than, EBT in the fluvial aquifer at the MI. The scope of additional investigation in support of an MI ROD Amendment/ESD was discussed during the May 2014 project review meeting.

A Supplemental Remedial Investigation and Focused Feasibility Study (SRI/FFS) will be performed to develop a remedial strategy to achieve RAOs throughout the MI. A project-specific QAPP will be prepared for the SRI.

1.4 PROJECT QUALITY OBJECTIVES AND MEASUREMENT PERFORMANCE CRITERIA

This section identifies the environmental decisions to be made and the level of data quality needed to ensure that those decisions are based on sound scientific data.

1.4.1 Project Quality Objectives

Project quality objectives (PQOs) define the type, quantity, and quality of data that are needed to answer specific environmental questions and support proper environmental decisions. Site-specific PQOs are described below and summarized on Worksheet #11- Project Quality Objectives/Systematic Planning Process Statements.

1.4.1.1 AS/SVE Operations

Operations and performance data for the AS/SVE system are collected by HDR to monitor system performance and evaluate system effectiveness. The data requirements are described in the approved RA work plan and updated in annual reports.

- Operations data consist of vapor flow rate and vacuum at SVE wells and system discharge, flow rates and pressure at the compressor and AS points, and vacuum measurements at VMPs and are collected from direct reading of gauges. The data are used to track vapor extraction and capture throughout the treatment area. The data are collected monthly and included in quarterly and annual reports.
- Performance data for the AS/SVE consist of monthly PID measurements at VMPs, SVE wells and system discharge and quarterly laboratory analysis of vapor samples from system discharge. The data are used to monitor removal of VOCs from groundwater and comply with permit limits for the system discharge. The PID measurements provide screening level data; the VOCs in the discharge are confirmed by definitive laboratory analyses. AS/SVE system effectiveness is determined primarily through groundwater monitoring.
- The vapor analytical results, operating hours and flow rates for the system discharge are used to estimate the VOC mass removed at each system.
- Summary data are provided in quarterly reports and validated laboratory analytical results are included in annual reports. Laboratory analytical results are added to the DDMT analytical database with the final data quality evaluation (DQE) flags.

PQOs for AS/SVE operations are to evaluate:

- Removal of VOCs from groundwater by the AS/SVE system; and
- Compliance with effluent discharge limits.

1.4.1.2 EBT Operations

Operations and performance data for EBT are collected by HDR to monitor injection activities and evaluate changes in aquifer conditions. The data requirements are described in the approved RA work plan amendment and updated in annual reports.

- Operations data consist of mixing and injections records. The data are used to track volume of carbon source (lactate) in the injection solution for each well and to evaluate success in completing injections by well and area.

- Performance data for EBT consist of field and analytical results. Field measurements of water quality are DO, ORP, pH, temperature, and conductivity; results are used to confirm aquifer conditions are appropriate for bioremediation. Groundwater samples are analyzed for VOCs, TOC, metabolic fatty acids and dissolved gases to evaluate distribution of carbon source in the aquifer and to confirm reductive dechlorination through concentration of parent CVOCs (PCE, TCE and CT) and breakdown products (cDCE, VC, CF, ethene and ethane).
- Summary data are provided in quarterly reports and validated laboratory analytical results are included in annual reports. Laboratory analytical results are added to the DDMT analytical database with the final DQE flags.

PQOs for EBT operations are to evaluate:

- Extent of anaerobic conditions
- Distribution of injected carbon source
- Reduction in CVOCs

1.4.1.3 Groundwater Monitoring at Dunn Field

Groundwater monitoring at Dunn Field is conducted by HDR to evaluate effectiveness of remedial actions in meeting the RAOs to prevent further offsite migration of VOCs in groundwater in excess of protective target levels and remediate fluvial aquifer groundwater to drinking water quality to be protective of the deeper MAQ. The data requirements are described in the approved LTM plans and are updated in annual reports.

- Groundwater data consist of water level measurements and laboratory analyses for VOCs in groundwater samples from PMWs and LTM wells. The data are collected at designated wells in accordance with the approved plans. Groundwater samples are collected using either passive diffusion bags (PDBs) or low-flow sampling. When low-flow sampling is used, screening level field measurements are used to confirm stabilization of the groundwater parameters prior to sampling. Summary data are provided in reports after each sample event and validated laboratory analytical results are included in annual reports. Laboratory analytical results are added to the DDMT analytical database with the final DQE flags.
- PQOs for Dunn Field groundwater analytical data are to evaluate:
    - Effectiveness of the FSVE system in preventing further groundwater impacts by removing CVOCs in soil and, following system shutdown, confirmation that VOC concentrations in the

vadose zone have been reduced sufficiently to prevent rebound of groundwater concentrations in excess of the treatment goal of 50 µg/L for individual CVOCs.

- Effectiveness of the AS/SVE system in reducing CVOC concentrations within the TA below 50 µg/L for individual CVOCs.
- Progress of FSVE and AS/SVE systems in combination with natural attenuation processes in reducing groundwater concentrations to target concentrations within a reasonable period of time.

1.4.1.4 Groundwater Monitoring at the Main Installation

MI LTM is conducted by HDR to evaluate progress in meeting the RAOs to restore groundwater to concentrations at or less than MCLs and to prevent contaminant migration horizontally and vertically offsite at concentrations in excess of MCLs. The data requirements are described in the approved LTM plan and are updated in annual reports.

- Groundwater data consist of water level measurements and laboratory analyses for VOCs in groundwater samples from LTM wells. The data are collected at designated wells per the schedule updated in annual LTM reports. Groundwater samples are collected using PDBs low-flow sampling and screening level field measurements are used to confirm stabilization of the groundwater parameters prior to sampling. Summary data are provided in reports after each sample event and validated laboratory analytical results are included in annual reports. Laboratory analytical results are added to the DDMT analytical database with the final DQE flags.
- PQOs for MI LTM data are to evaluate:
  - Rebound of CVOCs in the TAs.
  - Migration of CVOCs into deeper aquifers.
  - Progress in reducing groundwater concentrations to MCLs.

1.4.2 Measurement Performance Criteria

Measurement performance criteria are determined for each matrix, analytical group, concentration level, and analyte, if applicable. The criteria relate to the parameters of precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity (PARCCS). The parameters indicate the qualitative and quantitative degree of quality associated with measurement data and, hence, are referred to as data quality indicators (DQIs).

PARCCS criteria measure the usability of environmental data as it relates to project objectives. Evaluation of these criteria ultimately reveals the representativeness and bias, if any, present in the sampling and analytical processes. The field program will be assessed through the collection and analysis of field duplicates, rinsate blanks, trip blanks, and matrix spike/matrix spike duplicates (MS/MSDs). The analytical program will be assessed through the internal laboratory quality control (QC) performed, including method blanks, laboratory calibration standards (LCSs), surrogate standards, internal standards (ISs), and calibration standards.

Measurement Performance Criteria for groundwater and vapor sample analyses are shown on Worksheet #12. PARCCS criteria are discussed in the following sections.

1.4.2.1 Precision

Precision is the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. *Precision data* indicate how consistent and reproducible the field sampling or analytical procedures have been. Analytical precision for a single analyte is expressed as a percentage of the difference between results of duplicate samples for a given analyte. The relative percent difference (RPD) for each compound is calculated using the equation below. The closer the numerical values of the measurement, the more precise the measurement. Comparing overall project precision and laboratory precision will help to identify sources of imprecision if a problem exists.

Precision is expressed either as relative standard deviation (RSD) for more than two replicate measurements or as RPD for duplicate measurements. The RSD for each compound or element is calculated using the following equation:

$$s = \sqrt{\frac{\sum_{i=1}^{n} (x_i - x)^2}{n - 1}}$$

Where:

s	=	sample standard deviation
x	=	the mean
x_i	=	the i_{th} data value
n	=	number of data values
Σ	=	sum of

The RPD for each compound or element is calculated using the following equation:

$$\text{RPD} = \frac{\text{A - B}}{(\text{A} + \text{B})/2} \text{ x } 100$$

Where:

A = Replicate value 1

B = Replicate value 2

RPD = Relative percent difference

To measure precision in environmental samples, field duplicate samples are collected concurrently with the parent sample under the same field conditions. Although spiked in the laboratory, MS/MSD samples also provide field precision data. Field duplicates will be collected at a frequency of ten percent (i.e., one duplicate for every ten field samples).

Precision determination will be performed in the laboratory by the analysis of laboratory duplicate vapor samples and MS/MSD groundwater samples.

1.4.2.2 Accuracy

Accuracy is the degree of agreement between an observed value (sample result) and an accepted reference value; *bias* describes the systematic or persistent distortion associated with a measurement process.

Analyte accuracy/bias is evaluated using different types of QC samples. A LCS that contains a known concentration of analyte(s) spiked into contaminant-free water or other blank matrix provides information about how accurately the laboratory (analysts, equipment, reagents, etc.) can analyze for a specific analyte(s) using a selected method. The cumulative laboratory and method accuracy/bias is calculated as a percentage using the following equation:

Accuracy/*Bias* = (*Measured Value/True Value)* × 100%

Because environmental samples contain interferences, the accuracy/bias for a specific analyte is evaluated in relation to the sample matrix, by analyzing matrix spike samples. A known concentration of the analyte is added to an aliquot of the sample. The difference between the concentration of the analyte in the unspiked sample and the concentration of the analyte in the spiked sample should be equal to the concentration of the analyte that was spiked into the sample.

The spike recovery is calculated as a percentage using the following equation:

$$\%R = \frac{X - T}{K} \times 100$$

Where:

X	=	Analytical result from the spiked sample
T	=	Analytical result from the unspiked aliquot
K	=	Known value of the spike
%R	=	Percent recovery

The percent recovery (%R) of the compounds spiked into a matrix (via both LCSs, MS/MSDs, or surrogates) is used to evaluate the accuracy of the environmental sampling process. The recovery of an analyte from the LCS, an MS/MSD, and/or surrogate spikes is indicative of the impact a specific matrix may have on the accuracy of a specific compound or element. The compounds to be spiked for in LCSs and MS/MSDs include the full list of analytes to be reported for the individual analysis.

The accuracy and bias of field protocols are difficult to assess quantitatively. Sampling accuracy can be maximized, however, by the adoption of and adherence to a strict field QA program. Specifically, procedures will be performed following standard protocols. Equipment and instrumentation will be properly calibrated and well maintained. Trip blanks will be included in each groundwater sample batch and equipment rinsates will be provided as needed to provide data to assess the potential for cross-contamination. Through regular review of field procedures, deficiencies will be documented and corrected in a timely manner.

Exceedances of matrix-specific QC samples (MS/MSDs) may be problematic because of matrix effect (signal enhancement or suppression) on the analysis, but should not be viewed as an indicator of poor laboratory performance. Necessary corrective actions will vary depending on the type of interference and are subject to analyst professional judgment. When these departures indicate potential for false negatives, lack of sensitivity, or inability to accurately detect the target analyte(s), the analyst will inform the laboratory project manager, who will contact the HDR project chemist for direction regarding finding possible alternatives. Other options, such as taking measures to decrease the matrix effect by such techniques as implementing cleanup procedures, diluting the samples, or processing a smaller amount of sample, may be considered. However, consequences to the data (e.g., higher detection limits or less representative sample aliquot) must be assessed comparatively with project objectives.

An LCS will be analyzed with each laboratory analytical batch. Surrogates will be spiked into every sample, standard, and blank. Groundwater MS/MSD samples will be collected at a frequency of five percent (i.e., one pair of MS/MSD samples for every 20 field samples.)

1.4.2.3 Representativeness

Representativeness is a qualitative term that describes the extent to which a sampling design adequately reflects the environmental conditions of a site. It takes into consideration the magnitude of the site area represented by one sample and indicates the feasibility and reasonableness of that design rationale. Representativeness also reflects the ability of the sample team to collect samples and the ability of the laboratory personnel to analyze those samples so that the generated data accurately and precisely reflect site conditions.

If the results are reproducible, the data obtained can be said to represent the environmental condition. Representativeness is ensured by collecting sufficient samples of an environmental medium, properly chosen with respect to place and time. The precision of a representative set of samples reflects the degree of variability of the sampled medium as well as the effectiveness of the sampling techniques and laboratory analysis. Samples that are not properly preserved or analyzed beyond holding times may not be considered representative. Review of sampling procedures, laboratory preparation, analysis holding times, trip blank analysis, and field blank analysis are required for this assessment.

1.4.2.4 Completeness

Completeness is a measure of the amount of valid data collected using a measurement system. Completeness is expressed as the percentage of usable data obtained from a measurement system compared to the amount that was expected to be obtained under correct or normal conditions. For data to be considered usable, they must meet some or all of the acceptance criteria specified in the analytical method used and must not result in “rejected” data points.

Field sampling conditions are often unpredictable and non-uniform. However, the objective of the field sampling program is to obtain samples for each analysis required at each site, provide sufficient sample material to complete those analyses, and collect the QC samples necessary to fully implement the field and laboratory QA/QC program.

Samples, for which critical data points fail the data quality objectives (DQOs), may be reanalyzed (providing adequate sample volume and holding times are met) or re-sampled (with approval of the project manager) to meet the completeness goal. Critical data points are those points that are needed to

meet the established DQOs, and include the contaminants of concern as well as the field activities necessary to achieve the DQOs.

Completeness will be calculated on a per-analysis-method, per-matrix, and per-site basis. The percent complete is used to evaluate whether sufficient data were acquired from the sampling event. The overall DQO for field sampling and analytical completeness during field investigations is 90 percent for waters and air (soil vapor) samples.

1.4.2.5 Comparability

Comparability is the degree to which different methods or data agree or can be represented as similar. It describes the confidence with which one dataset can be compared to another dataset.

Field and laboratory procedures affect comparability. To optimize comparability, only USEPA established methods and established protocols have been selected or specified as appropriate for these investigations. By using standard sampling and analytical procedures, datasets will be comparable among DDMT sites.

1.4.2.6 Sensitivity

Sensitivity is the ability of the method or instrument to detect the target analytes at the level of interest.

In order to meet DDMT project-specific DQOs, screening (where confirmed by definitive data) and definitive data will be compared to DDMT risk-based screening levels or RG. Definitive data will be generated using USEPA methods with method detection limits (MDLs) and/or reporting limits (RLs) at or below screening levels to allow for sufficient qualitative and quantitative results (where achievable and feasible).

Worksheet #15 presents comparisons of each of the method MDLs and RLs for target analytes to the DDMT background values and risk-based screening levels.

Laboratory-established MDLs are updated annually per analytical method and matrix. The RLs used for DDMT will remain unchanged for the duration of the project. Updated MDL information will be provided by the laboratory on sample result forms but will not be updated in the QAPP, as the MDLs presented in the QAPP reflect the laboratory's MDLs at the time the QAPP was developed. Method MDLs, RLs, and sample quantitation limits (SQLs) are defined below.

RL values meet the general DQOs as long as the RL is below the DDMT risk-based screening level. Contaminants of concern with an RL above the screening level will be evaluated by comparison to the MDL. If the MDL is below or equal to the screening level, the MDL will be considered to meet the general DQOs. Some contaminants have MDLs above the screening level; this issue will be noted in reports, but no corrective action is suggested, as long as the same analytes consistently have MDLs above the screening levels and the data sets remain comparable.

1.4.2.6.1 Method Detection Limit and Limit of Detection

MDL studies are conducted using spiked reagent water for water matrices processed through the appropriate analytical procedure. The RL is derived from the MDL and is set at the project-requested RL to meet project-specific DQOs.

The MDL is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the value is above zero. The MDLs are established using the required USEPA procedure specified in 40 Code of Federal Regulations (CFR) 136, Appendix B (*Definition and Procedure for the Determination of the Method Detection Limit* (40 CFR 136, 1986). A data pool of at least seven spiked replicates analyzed at a concentration approximately three times the anticipated MDL is generated. The MDL is estimated by employing the "t" distribution with a 99 percent confidence interval using the following equation:

$$MDL = \sqrt{(t)(S)}$$

Where:

t = a factor for n-1 degrees of freedom at the 99 percent confidence factor
S = the standard deviation of the data pool

The laboratories perform annual MDL studies on each instrument. Limit of detection verifications are performed after each MDL study according to the laboratories' Standard Operating Procedures (SOPs) L1 and L2.

1.4.2.6.2 Reporting Limit and Sample Quantitation Limit

The RL is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions as defined by SW-846. Project-specific RLs were reviewed in comparison to risk-based screening levels. The SQL is the RL adjusted by the sample volume analyzed, and/or dilution.

Each analytical concentration will be reported as a numeric value at or greater than the MDL. Samples with no detections below the MDL are reported as less than the MDL, on not detected (ND), or undetected (U), depending on the laboratory's reporting system. Detections below the SQL but above the MDL will be reported as estimated values. Water results will be reported in µg/L or mg/L concentrations, and air (soil vapor) samples will be reported in parts per billion by volume (ppbv).

1.5 SECONDARY DATA EVALUATION

Secondary data includes data generated by external, independent parties; data collected in other investigations designed to answer different questions than the current investigation; or data from computer models. Secondary data such as operational records and interviews with former employees was used to guide initial investigations at DDMT. Boring logs from previous investigations have been used to develop subsurface cross-sections used to estimate groundwater flow and well installation data. Computer models were used to evaluate potential impacts to the Intermediate and Memphis aquifers on the MI and Dunn Field, to identify sentinel well locations and to estimate the groundwater monitoring period necessary to observe groundwater impacts following shutdown of the FSVE system. The model results and other secondary data used at DDMT have been confirmed through sampling and laboratory analyses. Key historical documents are listed on Worksheet #13-Secondary Data Criteria and Limitations Table.

1.6 PROJECT OVERVIEW AND SCHEDULE

Worksheet #14-Summary of Project Tasks is attached.

Worksheet #15-Reference Limits and Evaluation Table is attached. This worksheet presents comparisons of each of the method MDLs and RLs for target analytes to the DDMT background values and risk-based screening levels as discussed in section 1.4.2.6, Sensitivity.

Operations and performance data for the AS/SVE system and for EBT will be collected as long as treatment continues. The AS/SVE system is currently expected to operate through December 2016. EBT injections are currently scheduled to end in August 2014 and performance monitoring in November 2014.

LTM will continue until groundwater RGs are achieved, MCLs on the MI and target concentrations from the ROD on Dunn Field. LTM will be performed in accordance with the approved LTM plans with modifications documented in annual reports. Groundwater monitoring is conducted in April and October for Dunn Field LTM and the MI.

The project master schedule is provided in the annual site management plan update; the current update is *2014 Site Management Plan, Revision 1* (HDR, 2014c). RA-O and LTM activities are summarized on Worksheet #16-Project Schedule/Timeline.

2.0 MEASUREMENT AND DATA ACQUISITION

This section describes how project data will be collected, measured and documented. Proper implementation of these activities will help ensure that project data are scientifically sound, of known and documented quality, and suitable for the intended use. QA activities will be performed during each phase of data collection and generation. Sampling and analysis procedures to be used in project activities are discussed below.

2.1 SAMPLING TASKS

This section includes all components of the project-specific sampling system, including process design and rationale, procedures, and requirements.

2.1.1 Sampling Design and Rationale

The sampling design, rationale and procedures are described in the approved RA work plans and LTM plans and are updated in annual reports. The information is summarized on Worksheet #17-Sampling Design and Rationale and Worksheet #18-Sampling Locations and Methods/SOP Requirements.

2.1.2 Sampling Procedures and Requirements

SOPs in Appendix B include procedures for sampling groundwater for VOCs, TOC, metabolic fatty acids (MFAs) and dissolved gases, and air for VOCs, and procedures for use of the required equipment or technique (low flow bladder pumps, bailers and PDBs for groundwater and Summa canisters for vapor). Worksheet #19-Summary of Analytical SOP Requirements, which lists sample containers and holding times required for each analytical method, is attached.

Worksheet #20-Field QC Sample Table is attached. The number of field QC samples for each matrix (groundwater and air), analytical group (VOCs), and concentration level are provided.

2.1.2.1 Sample Collection Procedures

Sampling procedures that will be used in the project are described in SOP 4 and SOP 5, shown in Worksheet #21-Project Sampling SOP References Table.

2.1.2.2 Sample Containers, Volume and Preservation Procedures

Worksheet #19-Summary of Analytical SOP Requirements lists requirements for sample containers, volume, preservation and holding time requirements, as specified in the applicable USEPA SW-846 or TO-15 method.

SOP 4, SOP 7 and SOP 8 describe preservation procedures that maintain sample integrity in the field, prior to and during shipment to, the off-site laboratory. Laboratory SOPs L-8 and L-10, listed in Worksheet #21, describe sample receipt, log-in and storage procedures at the off-site laboratories.

2.1.2.3 Equipment and Sample Containers Cleaning and Decontamination Procedures

Procedures for the initial cleaning of sampling equipment and subsequent decontamination procedures that will be followed during the sampling event are described in SOP 9, listed in Worksheet #21. These procedures will help ensure that collected samples are representative of the sampling location by verifying that sampling equipment is clean and free of target analytes or interferences.

2.1.2.4 Field Equipment Calibration, Maintenance, Testing and Inspection Procedures

Worksheet #22-Field Equipment Calibration, Maintenance, Testing, and Inspection Table is attached and describes procedures and documentation activities to ensure that field sampling equipment is available and in working order.

2.1.2.5 Sampling Supply Inspection and Acceptance Procedures

Procedures for inspection and acceptance of sampling supplies, to ensure adequate sampling supplies were received, are described in SOP 4 and SOP 5.

2.1.2.6 Field Documentation Procedures

Field documentation procedures are described in SOP 1. Field data are recorded in field logbooks and on data collection forms to provide a permanent record of field activities. The records are maintained in project files.

2.2 ANALYTICAL TASKS

The following sections address all components of the project-specific analytical measurement system, including on-site and off-site laboratory analytical SOPs; method- and laboratory-specific QC measurements, acceptance criteria, and corrective actions; calibration procedures; and instrument, equipment, and supply maintenance, testing, and inspection requirements. Planned on-site analysis at

DDMT consists of semi-quantitative field screening techniques. Off-site analyses will be performed by Microbac (VOCs, TOC, MFAs and dissolved gases in water) and ALS Laboratories (VOCs in air). Laboratory SOPs are included in Appendix C.

2.2.1 Analytical SOPs

Worksheet #23-Analytical SOP Reference Table is attached and lists on-site and off-site analytical procedures to be used during RAO and LTM.

2.2.2 Analytical Instrument Calibration Procedures

Worksheet #24-Analytical Instrument Calibration Table is attached and lists the calibration procedures for each analytical instrument, which allows the analytical technique to accurately and precisely identify and quantitate the target analytes at the required quantitation limits (QLs) and within the required measurement ranges. The worksheet also lists the flags the laboratory should apply to sample results if the calibration criteria are not met.

2.2.3 Analytical Instrument and Equipment Maintenance, Testing and Inspection Procedures

Worksheet #25-Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table is attached and lists the procedures and documentation activities that will be performed to ensure all analytical instrumentation and equipment are available and in working order when needed.

2.2.4 Analytical Supply Inspection and Acceptance Procedures

SOP 4, SOP 5 and SOP 7 include procedures that field sampling personnel will follow upon receipt of sampling supplies or supplies for field instrumentation, in order to ensure correct supplies were received.

Laboratory receipt of supplies is discussed in SOPs L-8 and L-10.

2.3 SAMPLE COLLECTION, HANDLING, TRACKING AND CUSTODY PROCEDURES

This section describes sample collection documentation and sample handling, tracking, and custody procedures used to ensure that sample integrity and custody are maintained. The procedures address sample collection, packaging, handling, and shipping, as well as records, receipt of laboratory samples, archiving, and disposal. Chain-of-custody (COC) SOPs include procedures associated with sampling and off-site laboratory analysis. Use of on-site laboratory analysis is not planned at DDMT.

2.3.1 Sample Collection Documentation

Proper field sampling and analytical documentation help ensure sample authenticity and data integrity. SOP 4 and SOP 5 describe sample collection activities. SOP 7 and SOP 8 describe associated sample handling activities such as sample documentation, packing and shipping. SOP 1 contains information on field documentation procedures.

2.3.2 Sample Handling and Tracking System

Proper sample tracking systems support the COC procedures, which in turn help to ensure sample authenticity and data defensibility.

Sampling schedules and requirements are described in approved work plans and annual reports. The PC will prepare Sampling Plan Details (SPDs) in Excel identifying samples to be collected and will confirm the list with the PM.

For LTM and EBT sampling events, the PC will prepare an SPD that lists the sample locations, analyses and sample identifications (IDs) and includes field QC samples (field duplicates, MS/MSD samples, trip blanks and rinsate blanks). The PC will coordinate the sampling event ID with the DDMT Database Manager. The PC will verify the accuracy of the SPD with the PM and FTL, then send the SPD, as a bottle order, to Microbac, and to the FTL and PM.

Microbac will use the SPD to prepare pre-printed labels, load the sample IDs into a bar code system, and send the sample vials, labels, bar code reader, coolers, COC forms, custody seals, and bubble wrap, as detailed in SOP L-8 to the Memphis site, by FedEx, using this address:

2241 Truitt Street
Building 265
Memphis TN 38114

MI LTM samples will be collected semiannually, with some samples collected only annually or biennially. Sample events will be sequentially numbered, and sample IDs will be in the format:

Well ID-LX-## (e.g., MW-256-LA-14)

where: L = long term monitoring
X = event (A for annual, B for biennial, S for semiannual)
= sequential event number

MI EBT samples will be collected quarterly, with some samples collected only annually or biennially. Sample events will be sequentially numbered, and sample IDs will be in the format:

Well ID-EBT-## (e.g., MW-256-EBT14)

where: L = long term monitoring
EBT = EBT event
= sequential event number

Dunn Field LTM samples will be collected semiannually with some samples collected only annually or biennially. Sample events will be sequentially numbered, and sample IDs will be in the format:

Well ID-DFLX-##- (e.g., MW-132-ODLS-4)

where: DF = Dunn Field
L = long term monitoring
X = event (A for annual, B for biennial, S for semiannual)
= sequential event number

For an air sampling event, the PC will prepare an SPD that lists the sample locations, analyses and sample IDs and includes field QC samples (field duplicates). The FTL will inform the PC as to the number of Summa™ canisters, flow controllers, and tubing and swage fittings required. The PC will verify the accuracy of the SPD with the PM and FTL, then send the SPD, as a canister order, to the FTL, the PM, and Microbac, who will forward it to ALS Global (ALS).

ALS will clean, evacuate, and pack the canisters as detailed in SOP L-6 and send them to the Memphis site, by FedEx, using the address above.

The AS/SVE (also called Off-Depot soil vapor extraction [ODSVE]) effluent air sample will be collected quarterly. Sample events will be identified by the sampling quarter and year, and sample IDs will be in the format:

ODSVE-EFF-XQYY-NS (e.g., ODSVE-EFF-3Q11-NS)

where: ODSVE = off depot SVE system
EFF = effluent sample
X = quarter number (e.g., 1 for first quarter)
Q = quarter
YY = last two digits of the year
NS = normal sample

2.3.2.1 Sample Handling

Worksheet #26-Sample Handling System shows the flow of samples from the time of collection to laboratory delivery to final sample disposal.

2.3.2.2 Sample Delivery

Samples will be grouped in sample delivery groups (SDGs), defined as a group of 20 or fewer field samples within a project task. If practical, samples from one task will not be placed in the same cooler with samples from another task.

Samples will be packed into coolers, or cartons for the Summa™ canisters, as described in SOP 7 and shipped overnight to the laboratory via Federal Express. For DDMT, the shipping costs are covered by the laboratories, and their account number will be used on the airbill.

2.3.3 Sample Custody

The evidentiary trail from sample collection through data generation and archiving is maintained using sample custody procedures and documented by complete COC records. A sample is in "custody" if it is in the actual physical possession of authorized personnel or in a secured area that is restricted to authorized personnel. Since it is often difficult to predict what samples or projects will require proof of custody after the fact, all data collection events should employ documented COC procedures to ensure data authenticity and defensibility. Worksheet #27-Sample Custody Requirements describes field and laboratory custody procedures.

2.3.3.1 Sample Custody – Field

Sample custody in the field includes labeling each sample container, collecting and preserving the samples, and packaging samples for shipment to the designated laboratory. Proper documentation of field samples includes completing the logbook, the Daily Field Report, and the COC record for each sample shipment. The FTL will retain a copy of the COC record. The PC will receive emails from the laboratories that verify that samples were received intact and properly preserved.

Complete sample custody procedures for the field are described in SOP 7.

2.3.3.2 Sample Custody – Laboratory

Samples will be logged upon receipt at the laboratory by a sample custodian. The laboratory will assign a unique identification code to each sample container received. Sample receipt protocols and storage conditions include the following:

- Verify that sample holding times have not been exceeded.
- Determine whether the temperature requirement has been maintained during shipment and document the shipping container temperature on the COC. If the temperature is not between 0 degrees and 6 degrees Celsius (°C), the lab will notify the PC immediately.
- Compare samples received to those listed on the COC and examine all shipping records for accuracy and completeness.
- Sign each COC and record the date and time of sample receipt immediately.
- Note any problems associated with the coolers and samples on the cooler receipt form.
- Log each sample into the master logbook and computer file according to the laboratory's SOP.
- Record sample numbers on each sample container and attach laboratory sample container labels with the unique laboratory identification number and test.
- Place the samples in proper laboratory storage. The primary considerations for sample storage are:
  - Maintaining the sample at the method-required temperature, and
  - Maintaining sample integrity through adequate protection from constituents from outside sources or from cross-contamination between samples.

The laboratory will maintain the integrity of the samples received, their associated extracts, and the data generated. Limited and controlled access to laboratory areas will be maintained.

Complete sample custody procedures for the laboratories are described in SOPs L-8 and L-10.

2.4 QUALITY CONTROL SAMPLES

QC is the set of activities that are performed for the purposes of monitoring, measuring, and controlling the performance of a measurement process. QC samples provide measurable data quality indicators used to evaluate the different components of the measurement system, including sampling and analysis.

Worksheet #28-QC Samples Table identifies the QC samples, acceptance limits, required analysis frequency and corrective actions.

2.4.1 Field Quality Control Samples

Groundwater QC samples that commonly originate in the field and will be collected at DDMT are trip blanks, field (ambient) blanks, rinsate blanks, and field duplicates. Air QC samples that commonly originate in the field and will be collected at DDMT are field duplicates. The air field duplicates are collected with a T-fitting manifold so they are true field duplicates, collected at the same time as the parent samples, rather than sequential field replicates.

2.4.2 Analytical Quality Control Samples

Groundwater QC samples that commonly originate in the laboratory and will be used for DDMT are method blanks, ISs, MS/MSDs, surrogate spikes, LCSs, initial calibrations, second source standards, and continuing calibrations. Air QC samples that commonly originate in the laboratory and will be used for DDMT are method blanks, laboratory duplicates, ISs, surrogate spikes, LCSs, initial calibrations, second source standards, and continuing calibrations.

2.5 DATA MANAGEMENT TASKS

All project data and information must be documented in a format that is usable by project personnel. Project data and information will be documented, tracked, and managed, from generation in the field to final use and storage, in a manner that ensures data integrity, defensibility, and retrieval. Raw data (including electronic media) of all field samples, QC samples, standards, and blanks will be archived, where applicable, and maintained for 10 years from the date of generation.

2.5.1 Project Documentation and Records

Worksheet #29-Project Documents and Records Table lists project documents and records that will be generated for every aspect of the project.

2.5.2 Data Package Deliverables

Data package deliverables for on-site and off-site analyses are listed below.

2.5.2.1 Sample Collection and Field Measurements Data Package Deliverables

Sample collection documentation will include field logbook and field form entries, field measurements, and COCs. Field measurements will be taken for groundwater samples collected by low-flow sampling. The measurements are specific conductance, temperature, dissolved oxygen, pH, turbidity and oxidation/reduction potential. All field and QC sample results, calibrations, and calibration verifications

will be recorded in a field logbook or on field forms. The logbook pages and forms will be scanned and stored with other project data. The hardcopy versions of the field data will be filed with other project data.

The COCs will be scanned, and the scanned file will be sent to the PC, who will save it with other project files. The hardcopies will be filed with other project data.

2.5.2.2 On-Site Analysis Data Package Deliverables

There will be no on-site analytical data packages for DDMT.

2.5.2.3 Off-Site Laboratory Data Package Deliverables

Laboratory data packages will be provided for each set of samples designated as a SDG. Data and summary forms sufficient for the data validator to perform a Step I Verification and Steps IIa and IIb verification (commonly known as a Level III data package) are to be sent by email to the PC within 15 business days of sample receipt. Data and summary forms as listed above plus data sufficient for a Step III (data usability) assessment (commonly known as a Level IV data package) are to be sent on a compact disc (CD) to the PC within 20 business days of sample receipt. Hardcopy data packages are not required.

Level III Data Package Contents (Summary Forms)		**Level IV Data Package Additional Contents**
Case narrative	MS/MSD results	Reconstructed total ion chromatogram (RIC) for each sample
Sample results	Lab duplicate results	Raw spectra of target compound and background-subtracted spectrum of target compound for each sample
Gas chromatography/mass spectrometry (GS/MS) tuning	LCS results	RICs and quantitation reports for all GC/MS standards
Initial calibration	Instrument run logs	QC Raw Data - RICs, chromatograms, quantitation reports, integration reports, mass spectra, etc.
Second source calibration	Sample preparation	
Continuing calibration	Chain-of-custody	
Method blank results	Sample receipt	
Surrogate recoveries	Holding times summary	
Internal standard areas and RTs	Data qualifiers (flags)	
	Other records (e.g., telephone communication log)	

The lab will also send to the PC an electronic data deliverable (EDD) in the format currently being used, and Environmental Restoration Program Information Management System (ERPIMS) lab project files using the ERPIMS format that is in use at the time of data generation. The EDD and ERPIMS files will be sent via email and included on the CD with the Level IV data package.

Worksheet #30-Analytical Services Table, which lists the laboratories that will perform off-site analytical services, is attached.

2.5.3 Data Reporting Formats

Data and summary forms in the Level III data package will be in a format similar to the USEPA contract laboratory program (CLP) forms, and will include the same information. The EDD file will be an Excel flat file, in the format currently being used. The ERPIMS lab project files will use the ERPIMS format that is in use at the time of data generation.

Handwritten records will be written legibly in ink. Any changes or corrections that are made to hardcopy data will have the original entry crossed out with a single line and initialed and dated by the responsible person.

Examples of hard-copy data reporting forms and all verification checklists and forms are included in SOP 1, SOP 5 and lab SOPs SOP L-1 through L-10.

2.5.4 Data Handling and Management

This section describes computerized and manual procedures that trace the paths of all data from generation to final use and storage, as well as the associated quality checks for error detection that are performed to ensure data integrity.

2.5.4.1 Data Recording

Data recording in the field will be performed as described, and using the forms and formats included in, SOP 4 and SOP 5.

2.5.4.2 Data Transformations and Reduction

Data transformations and reduction will take place at the off-site laboratories as described in SOPs L-1 and L-6.

2.5.4.3 Data Transfer and Transmittal

Laboratory data packages will be provided for each set of samples designated as a SDG.

The laboratories will send to the PC:

- Level III data package in PDF format (see section 2.5.2.3) by email,
- Level IV data package on a CD,
- An EDD in Excel by email and on a CD, and
- ERPIMS lab project files by email and on a CD.

The laboratories will send directly to Diane Short & Associates (DSA):

- A Level III data package in PDF format.

Microbac, will send to these items to the PC. The PC will forward the files as appropriate to the PM and the Database Manager.

2.5.4.4 Data Analysis

The laboratories will perform data analysis as described in SOPs L-1 and L-6.

2.5.4.5 Data Review

The laboratories will perform data review on each data package as described in SOPs L-1 and L-6. The data review as performed by the laboratory information management system (LIMS) will indicate sample or QC results outside control limit. The data review performed by the laboratory's PM will make note of deficiencies and discrepancies and will be used to develop the case narrative that will be included in each data package.

The Step I data verification and Steps IIa/IIb data validation will be performed by DSA. DSA will use the report template as their SOP and will assign qualifiers as described in the list of qualifiers attached to the SOP.

Data review is discussed in more detail in section 4.0.

2.5.5 Data Tracking and Control

The PM or PC will set up on the computer network the folders necessary for storing project sample data. The PC will use an Excel spreadsheet to track all data packages and their status. The file will include project ID, package number, date sampled, dates of the various files received from the laboratories, and

location of the hardcopy files. CDs with the data files for each data package will be stored in the appropriate CD box in the PC's office. Hardcopies of the data packages are not required. Previously-required hardcopies of data packages are not required to be stored as long as there is a CD with the same information and the information has been loaded onto the HDR computer network and backed up. This storage is in accordance with the Office of Information Resource Management requirements.

3.0 ASSESSMENT AND OVERSIGHT

Assessment and oversight ensure that planned project activities are implemented as described in the QAPP and that reports are provided to apprise management of the project status and any QA issues that arise during implementation. Assessment activities help to ensure that data quality is adequate for its intended use, and that appropriate responses are in place to address non-conformances and deviations from the QAPP. Reports will be prepared to present assessment finding, document the need for corrective action, and describe the process for implementation of corrective action and evaluation of its effectiveness.

3.1 ASSESSMENTS AND RESPONSE ACTIONS

Periodic assessments will be conducted to ensure that usable data are being generated. Deviations from the QAPP may also be identified by project personnel without the benefit of formal assessments.

The number, frequency, and types of planned assessment activities to be performed were considered during development of this QAPP. Assessments will be performed for the activities with the most influence on data quality and provide information about potential problems and mistakes. Sampling error is generally thought to contribute the majority of the measurement error associated with project data. Assessments will include activities for identifying and correcting problems encountered during the project.

The assessments described in the UFP-QAPP Manual were considered:

- Readiness Review – A systematic, documented review of readiness for the startup or continued use of a facility, process, or activity, typically conducted before proceeding with a major phase of work. All RAs required by the RODs have been implemented. System operations and LTM have been performed for several years based on approved plans. A Readiness Review is not necessary.
- Field Sampling Technical Systems Audit (TSA) – A thorough on-site audit including equipment, supplies, personnel training, sampling procedures, sample handling and tracking, and data handling, management, and review procedures. The primary sampling activity is groundwater monitoring which is performed on a biennial cycle. One Field Sampling TSA will be performed biennially. Vapor sampling will be reviewed at the same time.
- On-site Analytical TSA – A thorough audit of on-site analytical procedures. No on-site analyses are performed except for screening level analysis of water quality parameters during low-flow

purging prior to groundwater sampling; these analytical procedures will be reviewed during the Field Sampling TSA.

- Off-site Laboratory TSA – A thorough audit of an off-site laboratory. The off-site laboratories are audited under the National Environmental Laboratory Accreditation Program (NELAP); current certifications will be provided in annual reports. The QA manager will review the latest annual audits performed through NELAP and perform a laboratory performance audit on a Level 4 data package annually for each matrix (groundwater and vapor); the NELAP audit summary and performance audit findings will be provided in annual project reports.
- Split Sampling and Analysis Audit – A comparison study to assess inter-laboratory precision and accuracy. No split sampling and analysis audits are planned. Vapor and groundwater sample analyses performed by different laboratories over several years at DDMT provide sufficient basis for review of precision and accuracy. Current results are reviewed against past analytical results in annual project reports. Split sampling and analysis is not necessary.
- Performance testing (PT) Sample Tracking and Analysis – Statistical analysis of PT sample results to provide information on routine laboratory performance and the overall accuracy and bias of the analytical method. PT sample tracking and analyses are performed as part of the NELAP certification and no further assessment is necessary.
- Data Review TSA – A thorough review of the complete data review process. Data review is performed by an independent contractor and the reports are reviewed by the PC using the procedures described in Section 4.2.
- Management Systems Review (MSR) – A review of organizational management structure, policies, and procedures. An MSR is not necessary based on the current level of activity for RAO-LTM at DDMT.

Worksheet #31-Planned Project Assessment Table is attached. Project-specific questionnaires and audit checklists used for performing assessments are provided in SOP 11. Completed checklists will be attached to the QA management reports.

Worksheet #32- Assessment Findings and Response Actions is attached. Assessment findings that require corrective action will initiate a sequence of events to include documentation of deficiencies, notification of findings, request for corrective action, implementation of corrective action, and follow-up assessment of the corrective action's effectiveness.

3.2 QA MANAGEMENT REPORTS

Worksheet #33-QA Management Reports Table is attached.

QA management reports will be provided to update managers and stakeholders on project status and results of all QA assessments. Assessment checklists, reports, requests for corrective action, and the corrective response summaries will be included as attachments to the QA management reports. Summaries of QA management reports will be included in annual project reports.

4.0 DATA REVIEW

Data review is the process by which data are examined and evaluated to varying levels of detail and specificity. It includes verification, validation, and usability assessment. The data review activities are used to ensure that only scientifically sound data that are of known and documented quality and meet PQOs are used in making environmental decisions.

4.1 OVERVIEW

There are three distinct evaluative steps that are used to ensure that project data quality needs are met.

- **Step I – Verification (review for completeness).** Confirmation by examination and provision of objective evidence that the specified sampling and analytical requirements have been completed.
- **Step II – Validation.** Confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. The validation process includes evaluating compliance with method, procedure, or contract requirements and evaluating against criteria based on the quality objectives developed in the QAPP (e.g., the measurement performance criteria [MPC]). The purpose of validation is to assess the performance of the sampling and analysis processes to determine the quality of specified data. It is divided into two subparts:
  - **Step IIa** assesses and documents compliance with methods, procedures, and contracts.
  - **Step IIb** assesses and documents a comparison with MPC in the QAPP.
- **Step III – Usability Assessment.** Determination of the adequacy of data, based on the results of validation and verification, for the decisions being made. The usability step involves assessing whether the process execution and resulting data meet project quality objectives documented in the QAPP.

In order to perform the data review steps described above, reported analytical data must be supported by complete data packages, as described in Section 2.5.2. If relevant raw data or sample information are not available or adequate to document data quality, then data review cannot be performed, and re-sampling or re-analysis must be considered.

4.2 DATA REVIEW STEPS

This section presents procedures for implementing each of the three data review steps.

4.2.1 Verification

Verification is a completeness check that is performed before the data review process continues in order to determine whether the required information is available for further review. It involves a review of all data inputs to ensure that they are present.

Worksheet #34-Verification (Step I) Process Table describes verification procedures to ensure that data are evaluated properly, completely, and consistently for use in meeting PQOs. Verification is not designed for use in qualitative review but ensures the availability of sufficient information for subsequent steps of the data review process. Example inputs for conducting the completeness check are listed in the worksheet.

4.2.2 Validation

Project-specific validation procedures are developed to identify and qualify data that do not meet the measurement performance criteria. Worksheet #35-Validation (Steps IIa and IIb) Process Table is attached.

Worksheet #36-Validation (Steps IIa and IIb) Summary Table is attached and lists the matrices, analytical groups, and concentration levels that each entity performing validation will be responsible for, as well as the criteria that will be used to validate those data.

SOP 10 details the final qualifiers that will be applied to the validated data. The DSA data review reports, included as Attachments 10-1 and 10-2 of SOP 10, are considered their SOPs. DSA uses an extended descriptive set of data qualifiers (included as Attachment 10-3 of SOP 10) that do not match the final qualifiers (U, J, UJ, B, R) that will be used for this project. Data validated by DSA will be further reviewed by the PC for conformance with the data review SOP (SOP 10). The final qualifier definitions are:

- U Not detected above the MDL
- J Detected, value is estimated
- UJ Not detected, MDL is estimated
- B Analyte also detected in an associated blank
- R Result is rejected

4.2.3 Usability Assessment

Worksheet #37-Usability Assessment is attached. The usability assessment considers whether data meet PQOs as they relate to the decision to be made, and evaluates whether data are suitable for making that decision. The usability assessment is the final step of data review and can be performed only on data of known and documented quality (i.e., verified and validated data). Data quality indicators (precision, accuracy/bias, representativeness, comparability, completeness, and sensitivity) are important components of validation and usability assessment. In the usability step, reviewers assess whether the process execution and resulting data meets quality objectives based on criteria (DQIs) established in the QAPP.

The PC will prepare a short summary of final qualifiers applied to the data and a usability assessment for each data package. The criteria for usability will be that data that are not rejected (final qualifier R) are usable. There may be exceptions to this general determination that may be sample-specific. These exceptions will be determined by the PC using professional judgment.

4.3 STREAMLINING DATA REVIEW

Streamlining data review is a process of eliminating some requirements for validation (steps IIa and IIb) that are deemed no longer necessary to preserve data integrity the review is meant to reduce time and costs while still confirming the quality of the data. Streamlining data review is not being incorporated at this time for RAO and LTM at DDMT.

5.0 REFERENCES

CH2M HILL, 1996. *Record of Decision for Interim Remedial Action of the Groundwater at Dunn Field (OU-1) at the Defense Distribution Depot Memphis, Tennessee*. Prepared for the U.S. Army Engineering and Support Center, Huntsville, Alabama. April 1996.

CH2M HILL, 2001. *Final Main Installation Record of Decision.* Prepared for the Defense Logistics Agency and presented to the U.S. Army Engineering and Support Center, Huntsville, Alabama. February, 2001.

CH2M HILL, 2004a. *Memphis Depot Dunn Field Record of Decision.* Prepared for the U.S. Army Engineering and Support Center, Huntsville, March, 2004.

CH2M HILL, 2004b. *Memphis Depot Main Installation Final Remedial Design, Rev. 1.* Prepared for the U.S. Army Engineering and Support Center, Huntsville, July, 2004.

CH2M HILL, 2008. *Memphis Depot Dunn Field Off Depot Groundwater Final Remedial Design.* Prepared for the U.S. Army Engineering and Support Center, Huntsville, Alabama. September 2008.

engineering-environmental Management, Inc. (e²M), 2007. *Main Installation Remedial Action Enhanced Bioremediation Treatment Operations and Maintenance Manual*

e²M, 2009a. *Dunn Field Record of Decision Amendment, Revision 3.* Prepared for the Air Force Center for Engineering and the Environment. January 2009.

e^{2}M, 2009b. *Operating Properly and Successfully Demonstration, Source Areas Remedial Action.* Prepared for the Air Force Center for Engineering and the Environment. June 2009.

40 Code of Federal Regulations (CFR) 136, 1986. Definition and Procedure for the Determination of the Method Detection Limit. Title 40, Chapter 1, Subchapter D, Part 136, Appendix B, June 30, 1986.

HDR, 2010. *Dunn Field Groundwater Interim Remedial Action 2009 Operations and Closure Report.* Prepared for the Air Force Center for Engineering and the Environment. December, 2010.

HDR, 2011a. *Off Depot Groundwater Interim Remedial Action Completion Report, Revision 1.* Prepared for the Air Force Center for Engineering and the Environment. July, 2011.

HDR, 2011b. *Dunn Field Off Depot Groundwater Air Sparge and Soil Vapor Extraction System Operations and Maintenance Manual*. Prepared for the Air Force Center for Engineering and the Environment. March, 2011.

HDR, 2012a. *Main Installation Remedial Action Work Plan Addendum, Revision 0.* Prepared for the United States Army Corps of Engineers, Tulsa District. October, 2012.

HDR, 2012b. *Dunn Field Source Areas Fluvial Soil Vapor Extraction System Annual Operations Report, Year Five.* Prepared for the United States Army Corps of Engineers. September, 2012.

HDR, 2014a. *Off Depot Air Sparge – Soil Vapor Extraction System Annual Operations Report, Year Four, Revision 1*. Prepared for United States Army Corps of Engineers, Tulsa District. August, 2014.

HDR, 2014b. *Main Installation Year Three Enhanced Bioremediation Treatment Report*. Prepared for Prepared for United States Army Corps of Engineers, Tulsa District. April, 2014.

HDR, 2014c. *2014 Site Management Plan, Revision 1*. Prepared for Prepared for United States Army Corps of Engineers, Tulsa District. January, 2014.

HDR, 2014d. *Annual Long-Term Monitoring Report-2013, Revision 1*. Prepared for the United States Army Corps of Engineers, Tulsa District. July 2014.

HDR|e^{2}M, 2009. *Source Areas Interim Remedial Action Completion Report, Revision 1*. Prepared for the Air Force Center for Engineering and the Environment. September, 2009.

HDR|e^{2}M, 2010. *Main Installation Interim Remedial Action Completion Report, Revision 1*. Prepared for the Air Force Center for Engineering and the Environment. February, 2010.

Intergovernmental Data Quality Task Force (IDQTF). *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual, Final Version 1*. March 2005.

Jacobs Federal Programs, 2002. *Remediation Report, Removal Action at Building 949, Former Defense Distribution Depot, Memphis*. Prepared for U.S. Army Corps of Engineers, Mobile. February 2002.

MACTEC Engineering and Consulting, Inc. (MACTEC), 2005a. *Remedial Action Sampling and Analysis Plan, Volume I: Field Sampling Plan and Volume II: Quality Assurance Project Plan*. Prepared for the Air Force Center for Environmental Excellence. November, 2005.

MACTEC, 2005b. *Early Implementation of Selected Remedy Interim Remedial Action Completion Report, Revision 1*. Prepared for the Air Force Center for Environmental Excellence. September, 2005.

MACTEC, 2006. *Dunn Field Disposal Site Remedial Action Completion Report.* Prepared for the Air Force Center for Environmental Excellence. July, 2006.

United States Environmental Protection Agency (USEPA), 1995. *Federal Facilities Agreement Between USEPA Region IV, TDEC, and Defense Logistics Agency at Defense Distribution Depot Memphis, Tennessee*. Effective March 6, 1995.

USEPA, 2010. *Preliminary Close Out Report. February, 2010.*

UXB International, 2001. *Final Chemical Warfare Material Investigations/Removal Report*. 2001.

TABLES

TABLE 1
REMEDIATION GOALS FROM DUNN FIELD RECORD OF DECISION
RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN
Defense Depot Memphis, Tennessee

	Remedial Goal Objectives				
	Site-Specific Soil Screening Levels to be Protective		Protective Soil Vapor Concentration		Groundwater Target Concentrations at 10-4 Target Risk Levels and Target HI=1.0 (µg/L)
Parameter	Loess Specific Values (mg/kg)	Fluvial Deposit Specific Values (mg/kg)	Loess Specific Values (ppbv)	Fluvial Deposit Specific Values (ppbv)	
Carbon Tetrachloride	0.2150	0.1086	28.14	14.22	3.0
Chloroform	0.9170	0.4860	61.57	32.63	12.0
Dichloroethane, 1,2-	0.0329	0.0189	1.12	0.64	—
Dichloroethene, 1,1-	0.1500	0.0764	57.00	29.03	7/340
Dichloroethene, cis-1,2-	0.7550	0.4040	73.86	39.52	35.0
Dichloroethene, trans-1,2-	1.5200	0.7910	256.53	133.50	50.0
Methylene Chloride	0.0305	0.0169	5.14	2.85	—
Tetrachloroethane, 1,1,2,2-	0.0112	0.0066	0.03	0.55	2.2
Tetrachloroethene	0.1806	0.0920	15.18	0.99	2.5
Trichloroethane, 1,1,2	0.0627	0.0355	0.84	2.03	1.9
Trichloroethene	0.1820	0.0932	10.56	2.06	5.0
Vinyl Chloride	0.0294	0.0150	28.94	14.77	—

Notes:
mg/kg = milligrams per kilogram
µg/L = micrograms per liter
ppbv = parts per billion per volume
MCL = maximum contaminant level
HI = hazard index
— = Not available for groundwater cleanup goals because of low number of detections or detected values consistently less than MCLs.

TABLE 2
MAIN INSTALLATION EBT WELLS
RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN
Defense Depot Memphis, Tennessee

Well	Area	Type	Northing (ft)	Easting (ft)	Top of Casing Elevation (ft, msl)	Ground Elevation (ft, msl)	Riser Length (ft)	Screen Length (ft)	Total Well Depth (ft, btoc)
IW21-01A	TTA-1N	IW	276504.77	800599.88	294.34	294.99	98.7	10	108.7
IW21-01B	TTA-1N	IW	276500.95	800605.89	294.61	294.85	89.9	10	99.9
IW21-02A	TTA-1N	IW	276464.81	800594.24	294.62	295.25	100.2	10	110.2
IW21-02B	TTA-1N	IW	276462.20	800598.51	294.65	295.12	91.7	10	101.7
IW21-03A	TTA-1N	IW	276551.96	800698.20	292.81	293.23	99.7	10	109.7
IW21-03B	TTA-1N	IW	276549.21	800705.08	292.50	293.12	89.8	10	99.8
IW21-04A	TTA-1N	IW	276518.82	800711.10	292.69	293.20	99.9	10	109.9
IW21-04B	TTA-1N	IW	276515.66	800715.39	292.79	293.30	89.7	10	99.7
MW-21	TTA-1N	IW	276473.36	800602.47	295.00	295.30	92.1	15	107.1
PMW21-01[1]	TTA-1N	IW	276533.14	800600.14	294.76	295.00	88.4	20	108.4
PMW21-02	TTA-1N	PMW	276574.64	800701.00	292.98	293.19	91.3	20	111.3
PMW21-04[1]	TTA-1N	PMW	276601.83	800771.56	291.87	292.20	89.0	20	109.0
IW101-02A	TTA-1S	IW	276198.80	801107.92	291.12	291.60	124.0	15	139.0
IW101-02B	TTA-1S	IW	276200.62	801111.95	291.14	291.72	110.1	15	125.1
IW101-02C	TTA-1S	IW	276203.32	801116.22	291.53	291.74	93.7	15	108.7
IW101-03A	TTA-1S	IW	276164.62	801104.58	291.94	292.36	125.2	15	140.2
IW101-03B	TTA-1S	IW	276161.58	801106.45	291.91	292.51	109.4	15	124.4
IW101-03C	TTA-1S	IW	276158.05	801108.62	292.04	292.54	93.6	15	108.6
IW101-04A	TTA-1S	IW	276249.13	801142.39	291.72	292.18	123.4	15	138.4
IW101-04B	TTA-1S	IW	276252.94	801142.79	291.59	292.08	107.0	15	122.0
IW101-04C	TTA-1S	IW	276257.10	801143.03	291.47	292.05	92.3	15	107.3
IW101-05A	TTA-1S	IW	276214.93	801126.64	291.52	292.12	121.4	15	136.4
IW101-05B	TTA-1S	IW	276218.44	801125.04	291.41	292.06	107.8	15	122.8
IW101-05C	TTA-1S	IW	276221.88	801122.88	291.27	291.89	92.2	15	107.2
IW101-07A	TTA-1S	IW	276125.77	801099.90	292.83	293.13	123.0	15	138.0
IW101-07B	TTA-1S	IW	276123.62	801102.61	292.81	293.15	106.6	15	121.6
IW101-07C	TTA-1S	IW	276121.28	801105.60	292.78	293.08	93.3	15	108.3
DR1-5	TTA-1S	IW	276080.00	800828.43	294.46	294.86	124.7	20	144.7
DR1-5A	TTA-1S	IW	276087.00	800835.01	294.51	294.87	90.0	20	110.0
DR1-6	TTA-1S	IW	276043.88	801103.40	293.17	293.50	114.4	20	134.4
DR1-6A	TTA-1S	IW	276035.02	801103.61	293.28	293.58	90.9	20	110.9
PMW101-02A	TTA-1S	IW	276281.93	801144.78	292.00	292.29	117.7	20	137.7
PMW101-02B	TTA-1S	IW	276286.33	801145.41	291.98	292.24	97.8	20	117.8
PMW101-04A	TTA-1S	PMW	276299.41	801182.12	291.07	291.43	117.9	20	137.9
PMW101-04B	TTA-1S	PMW	276296.40	801186.86	291.47	291.75	98.6	20	118.6
PMW101-07A	TTA-1S	PMW	276143.43	801171.78	292.20	292.52	117.9	20	137.9
PMW101-07B	TTA-1S	PMW	276141.84	801176.74	292.36	292.70	98.0	20	118.0
IW85-05	TTA-2	IW	276815.58	806162.75	304.73	305.30	92.4	10	102.4
IW85-06	TTA-2	IW	276779.47	806183.37	304.81	305.45	95.5	10	105.5
IW92-01	TTA-2	IW	276769.42	806506.97	304.51	304.88	80.4	10	90.4
IW92-02	TTA-2	IW	276719.57	806513.90	304.05	304.87	79.5	10	89.5
IW92-03	TTA-2	IW	276669.17	806511.19	304.20	304.72	83.5	10	93.5
IW92-07	TTA-2	IW	276725.81	806366.98	303.78	304.31	87.8	10	97.8
IW92-08	TTA-2	IW	276784.63	806289.19	304.55	304.93	85.3	10	95.3
DR2-2	TTA-2	IW	276770.85	806658.86	304.30	304.67	78.4	15	93.4
DR2-5	TTA-2	IW	276830.98	806180.40	305.41	305.72	84.5	15	99.5
PMW92-03[1]	TTA-2	IW	276678.91	806438.66	303.91	304.17	92.5	10	102.5
MW-113[1]	TTA-2	PMW	276685.34	806279.10	304.81	304.92	96.0	10	106.0
PMW85-01	TTA-2	PMW	276802.18	806146.13	305.08	305.39	93.2	10	103.2
PMW85-05	TTA-2	PMW	276752.08	806222.46	305.12	305.32	93.2	10	103.2
MW-85	TTA-2	PMW	276704.14	806064.51	304.13	304.50	95.9	15	110.9

TABLE 2
MAIN INSTALLATION EBT WELLS
RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN
Defense Depot Memphis, Tennessee

Well	Area	Type	Northing (ft)	Easting (ft)	Top of Casing Elevation (ft, msl)	Ground Elevation (ft, msl)	Riser Length (ft)	Screen Length (ft)	Total Well Depth (ft, btoc)
MW-62	B-835	IW	278289.65	801857.92	293.71	293.90	86.1	10	96.1
MW-213	B-835	IW	278427.08	801669.11	294.22	294.20	77.3	15	92.3
MW-212	B-835	PMW	278028.36	802225.40	295.34	295.68	85.3	15	100.3
MW-203A	W-C	IW	276841.76	801740.43	290.70	290.80	142.9	20	162.9
MW-203B	W-C	IW	276821.40	801741.59	290.87	291.10	93.0	20	113.0
MW-197A	W-C	PMW	276975.42	802042.30	291.26	291.54	161.7	15	176.7
MW-197B	W-C	PMW	276973.14	802036.92	291.03	291.43	93.8	15	108.8

Notes:

1) PMW21-04 and MW-113 changed to PMWs, PMW21-01 and PMW92-03 to IWs in August 2013.

bgs: below ground surface

btoc: below top of casing

ft: feet

msl: mean sea level

TABLE 3
DUNN FIELD LTM WELLS
RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN
Defense Depot Memphis, Tennessee

Well	Aquifer	Well Classification	Sample Frequency	Northing (ft)	Easting (ft)	Top of Casing Elevation (ft, msl)	Ground Elevation (ft, msl)	Riser Length (ft)	Screen Length (ft)	Total Well Depth (ft, btoc)
MW-03	Fluvial	Performance-FSVE	Semiannual	281596.25	802100.69	292.35	290.40	65.5	10	75.5
MW-04	Fluvial	Background	Biennial	281278.87	802369.19	301.61	300.00	60.0	20	80.0
MW-06	Fluvial	Performance-FSVE	Semiannual	280604.17	802069.13	289.11	288.10	51.0	20	71.0
MW-07	Fluvial	Background-NE	Annual	281839.88	802481.70	295.10	293.10	67.0	10	77.0
MW-08	Fluvial	Background-NE	Annual	282001.04	802727.91	292.59	292.74	56.5	10	66.5
MW-13	Fluvial	Background	Biennial	281033.56	802369.21	300.01	300.10	66.0	15	81.0
MW-15	Fluvial	Performance	Annual	280348.88	801985.36	295.12	295.23	63.4	15	78.4
MW-28	Fluvial	Background	Biennial	281568.58	803154.48	294.79	294.89	54.3	15	69.3
MW-31	Fluvial	Performance	Semiannual	281651.53	801783.90	290.37	287.50	64.1	15	79.1
MW-33	Fluvial	Performance	Biennial	280398.10	801561.30	280.71	277.70	44.6	15	59.6
MW-44	Fluvial	Performance	Semiannual	281073.71	800601.09	269.07	269.40	64.0	10	74.0
MW-54	Fluvial	Performance	Semiannual	281159.21	801183.83	295.39	295.57	84.5	10	94.5
MW-57	Fluvial	Performance	Annual	280184.05	802006.19	290.77	291.10	60.0	10	70.0
MW-58	Fluvial	Performance	Biennial	279845.07	802066.44	290.51	290.70	57.0	10	67.0
MW-67	Memphis	Sentinel	Biennial	280473.05	800933.94	278.21	275.53	260.0	15	275.0
MW-68	Fluvial	Performance	Annual	281500.76	802040.04	291.69	291.60	72.5	10	82.5
MW-69	Fluvial	Performance	Annual	281202.55	802011.49	307.02	304.90	82.1	10	92.1
MW-70	Fluvial	Performance	Annual	281029.60	801988.49	304.99	302.80	80.8	10	90.8
MW-71	Fluvial	Performance	Annual	280584.68	801804.71	294.40	291.90	65.5	10	75.5
MW-76	Fluvial	Performance	Annual	281311.98	801642.76	302.71	303.30	73.0	20	93.0
MW-77	Fluvial	Performance	Semiannual	281142.96	801815.29	304.42	304.70	68.0	20	88.0
MW-78	Fluvial	Performance	Biennial	282051.71	802065.28	275.00	275.40	44.5	20	64.5
MW-79	Fluvial	Performance	Semiannual	281794.22	800899.03	285.03	285.40	82.5	20	102.5
MW-80	Fluvial	Background	Biennial	281417.56	800199.07	273.81	274.00	53.0	20	73.0
MW-87	Fluvial	Performance-FSVE	Semiannual	280696.36	802038.55	294.93	292.80	63.0	15	78.0
MW-91	Fluvial	Performance	Annual	280474.97	802014.43	291.99	289.30	55.0	15	70.0
MW-126	Fluvial	Background	Biennial	282390.01	800491.67	252.22	252.49	16.0	10	26.0
MW-129	Fluvial	Background-NE	Annual	282271.08	803128.53	293.01	293.33	65.0	15	80.0
MW-130	Fluvial	Background-NE	Annual	282116.80	803241.45	293.17	293.77	59.5	20	79.5
MW-134	Fluvial	Performance-FSVE	Semiannual	281012.74	802102.58	300.81	301.05	75.0	15	90.0
MW-144	Fluvial	Performance	Semiannual	281138.63	801528.84	291.60	291.89	56.8	20	76.8
MW-145	Fluvial	Performance	Annual	280967.63	800823.18	284.72	284.86	80.1	20	100.1
MW-147	Fluvial	Performance	Annual	281501.94	801674.17	289.76	289.93	60.3	20	80.3
MW-148	Fluvial	Performance	Annual	281377.94	801461.63	294.71	294.87	70.0	20	90.0
MW-149	Fluvial	Performance	Semiannual	281130.04	800982.76	287.18	287.44	81.4	20	101.4
MW-150	Fluvial	Performance	Semiannual	281239.57	801283.62	296.86	297.00	71.2	20	91.2
MW-151	Fluvial	Performance	Semiannual	281290.42	800874.85	284.27	284.42	77.0	20	97.0
MW-152	Fluvial	Performance	Annual	281515.56	800892.84	289.59	289.82	91.0	20	111.0
MW-153	Fluvial	Performance	Biennial	282119.38	800952.34	279.17	279.26	76.1	20	96.1
MW-154	Fluvial	Background	Biennial	280501.53	800919.48	273.81	274.07	53.3	10	63.3
MW-155	Fluvial	Performance	Annual	281325.32	801168.98	291.54	291.83	76.9	20	96.9
MW-157	Fluvial	Performance	Annual	281050.86	801348.37	286.47	286.55	56.7	20	76.7
MW-158	Fluvial	Performance	Annual	281434.42	801005.34	294.07	294.38	91.0	15	106.0
MW-158A	Fluvial	Performance	Annual	281443.51	801005.67	293.95	294.22	77.9	15	92.9
MW-159	Fluvial	Performance	Semiannual	281304.17	801006.69	286.36	286.68	80.5	20	100.5
MW-160	Fluvial	Performance	Annual	281366.70	801304.05	293.84	294.13	65.7	20	85.7
MW-163	Fluvial	Performance	Semiannual	281152.59	801487.27	290.63	290.81	56.2	20	76.2
MW-164	Fluvial	Performance	Semiannual	280997.55	801497.47	287.48	287.71	55.6	20	75.6
MW-165	Fluvial	Performance	Semiannual	281384.63	800855.49	287.06	287.35	88.6	15	103.6
MW-165A	Fluvial	Performance	Semiannual	281383.55	800865.69	287.26	287.53	71.3	15	86.3
MW-166	Fluvial	Performance	Semiannual	281225.00	800927.99	282.72	283.29	84.6	15	100.0
MW-166A	Fluvial	Performance	Semiannual	281213.39	800927.27	282.90	283.36	68.1	15	83.4
MW-167	Fluvial	Background	Biennial	281394.03	800618.54	284.82	285.21	70.5	15	85.5

TABLE 3
DUNN FIELD LTM WELLS
RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN
Defense Depot Memphis, Tennessee

Well	Aquifer	Well Classification	Sample Frequency	Northing (ft)	Easting (ft)	Top of Casing Elevation (ft, msl)	Ground Elevation (ft, msl)	Riser Length (ft)	Screen Length (ft)	Total Well Depth (ft, btoc)
MW-169	Transition	Sentinel	Biennial	282491.23	800956.58	261.90	262.17	68.1	20	88.1
MW-170	Fluvial	Sentinel	Biennial	282443.17	801260.46	273.75	273.98	59.8	20	79.8
MW-171	Fluvial	Sentinel	Biennial	282315.35	801057.83	270.69	271.02	53.3	15	68.3
MW-174	Fluvial	Performance-FSVE	Semiannual	280352.00	802092.07	296.56	296.83	67.0	10	77.0
MW-176	Fluvial	Performance	Annual	280823.77	802032.08	299.68	299.92	76.0	10	86.0
MW-180	Fluvial	Performance	Annual	281476.43	802131.85	296.14	296.39	72.0	10	82.0
MW-182	Fluvial	Sentinel	Annual	280524.22	800623.13	275.40	272.98	62.0	10	72.0
MW-184	Fluvial	Performance	Semiannual	280903.16	801442.29	283.12	283.34	58.0	10	68.0
MW-187	Fluvial	Background	Biennial	280563.18	802348.09	302.74	303.21	76.0	10	86.0
MW-190	Fluvial	Performance	Semiannual	281138.88	801595.73	297.32	297.58	78.0	10	88.0
MW-220	Fluvial	Performance-FSVE	Semiannual	281617.49	802166.87	293.29	290.31	64.9	15	79.9
MW-221	Fluvial	Performance-FSVE	Semiannual	281399.71	802100.05	301.52	298.37	73.1	15	88.1
MW-222	Fluvial	Performance-FSVE	Semiannual	280986.04	802145.54	303.82	301.06	74.2	15	89.2
MW-223	Fluvial	Performance-FSVE	Semiannual	280913.53	802104.29	303.00	300.41	73.9	15	88.9
MW-224	Fluvial	Performance-FSVE	Semiannual	281017.74	802181.62	304.13	301.18	73.7	15	88.7
MW-225	Fluvial	Performance-FSVE	Semiannual	280947.12	802070.50	304.52	301.30	75.0	15	90.0
MW-226	Fluvial	Performance-FSVE	Semiannual	280931.94	802147.21	303.19	300.56	74.2	15	89.2
MW-227	Fluvial	Performance-FSVE	Semiannual	280257.91	802081.00	299.70	296.64	63.6	15	78.6
MW-228	Fluvial	Performance-FSVE	Semiannual	280251.88	802157.40	301.65	298.59	64.1	15	79.1
MW-230	Fluvial	Background-NE	Annual	281842.79	802800.06	286.57	286.66	59.2	15	74.2
MW-235	Fluvial	Sentinel	Annual	280727.57	800447.83	264.00	264.21	50.6	10	60.8
MW-237	Intermediate	Sentinel	Annual	281356.02	800963.99	289.18	289.53	166.5	10	176.7
MW-241	Fluvial	Performance	Annual	281389.82	801396.64	292.97	293.16	73.4	15	88.4
MW-242	Fluvial	Performance	Annual	281297.31	801228.65	295.40	295.94	73.2	16	88.7
MW-243	Fluvial	Performance	Semiannual	281370.62	801116.45	292.26	292.53	80.7	20	100.7
MW-244	Fluvial	Performance	Semiannual	281333.49	801101.07	288.72	289.45	76.3	20	96.3
MW-245	Fluvial	Performance	Semiannual	281379.46	801035.00	290.48	290.62	85.1	20	105.1
MW-246	Fluvial	Performance	Semiannual	281387.26	800951.62	288.17	288.49	85.2	20	105.2
MW-247	Fluvial	Performance	Semiannual	281319.40	800900.12	286.17	286.63	80.5	20	100.5
MW-248	Fluvial	Performance	Annual	281253.66	800720.22	275.45	275.93	67.5	20	87.5
MW-249	Fluvial	Performance	Semiannual	281029.63	800789.83	285.53	285.89	78.0	20	98.0
MW-250	Intermediate	Sentinel	Annual	281045.53	800900.38	289.66	290.19	168.7	15	183.7
MW-251	Intermediate	Sentinel	Annual	281211.70	801021.75	285.83	286.16	160.2	15	175.2

Notes:
ft: feet
btoc: below top of casing
msl: mean sea level

TABLE 4
MAIN INSTALLATION LTM WELLS
RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN
Defense Depot Memphis, Tennessee

Well	Aquifer	Well Classification	Sample Frequency	Northing (ft)	Easting (ft)	Top of Casing Elevation	Ground Elevation (ft, msl)	Riser Length (ft)	Screen Length (ft)	Total Well Depth (ft, btoc)
MW-16	Fluvial	Background	Biennial	278837.83	807099.66	299.86	300.19	57.6	15	72.6
MW-19	Fluvial	Background	Biennial	278945.87	800782.26	290.57	290.86	83.1	10	93.1
MW-22	Fluvial	Boundary	Biennial	275912.38	800702.16	298.04	298.49	95.4	10	105.4
MW-23	Fluvial	Boundary	Biennial	275791.02	801817.13	298.99	299.24	101.2	10	111.2
MW-24	Fluvial	Boundary	Biennial	275616.05	803538.81	299.51	299.81	97.3	15	112.3
MW-25A	Fluvial	Performance	Annual	275975.11	805521.27	269.88	270.13	73.0	10	83.0
MW-26	Fluvial	Performance	Semiannual	276508.16	805962.09	303.69	303.89	97.6	10	107.6
MW-34	Intermediate	Sentinel	Biennial	279411.21	801917.96	299.97	300.80	136.6	20	156.6
MW-38	Intermediate	Sentinel	Biennial	279141.38	802450.43	307.45	308.45	139.9	15	154.9
MW-39	Fluvial	Performance	Semiannual	277280.67	802598.11	296.28	296.58	95.5	20	115.5
MW-39A	Fluvial	Performance	Semiannual	277278.11	802607.66	298.61	298.70	148.1	20	168.1
MW-50	Fluvial	Boundary	Biennial	276455.81	807065.28	298.82	299.32	115.0	10	125.0
MW-52	Fluvial	Performance	Annual	275371.97	805897.36	279.26	279.71	94.0	10	104.0
MW-53	Fluvial	Background	Biennial	279176.66	805136.05	306.38	305.58	72.5	10	82.5
MW-55	Fluvial	Background	Biennial	279301.05	801204.62	292.08	292.48	64.0	10	74.0
MW-63A	Fluvial/Intermediate	Sentinel	Annual	278200.31	803572.83	305.96	306.33	140.0	10	150.0
MW-63B	Fluvial/Intermediate	Sentinel	Annual	278201.32	803557.77	305.78	306.22	115.0	10	125.0
MW-64	Fluvial	Performance	Semiannual	276951.52	805005.97	304.21	304.46	102.0	10	112.0
MW-66A	Fluvial	Background	Biennial	276626.02	799792.63	284.22	284.34	74.6	20	94.6
MW-88	Fluvial	Performance	Semiannual	276879.05	806512.88	305.15	305.47	82.0	15	97.0
MW-89	Intermediate	Sentinel	Annual	278286.97	802555.25	303.98	304.38	147.0	30	177.0
MW-90	Intermediate	Sentinel	Semiannual	278283.60	802539.51	304.19	304.64	115.0	30	145.0
MW-92	Fluvial	Performance	Semiannual	276614.20	806489.66	304.41	304.78	93.0	15	108.0
MW-93	Fluvial	Boundary	Biennial	275542.22	804440.10	294.08	294.31	92.0	15	107.0
MW-94A	Fluvial	Performance	Semiannual	276805.80	803085.80	303.00	303.23	109.6	10	119.6
MW-96	Fluvial	Performance	Annual	276310.14	806320.24	289.02	289.67	75.5	20	95.5
MW-97	Fluvial	Performance	Semiannual	276074.23	802139.23	297.44	297.70	97.5	20	117.5
MW-98	Fluvial	Performance	Semiannual	276891.37	802572.77	294.43	294.93	137.0	10	147.0
MW-99	Fluvial	Background	Biennial	277443.37	801114.53	285.33	285.69	91.5	20	111.5
MW-100B	Fluvial	Performance	Semiannual	276600.61	800854.26	290.92	291.47	107.4	20	127.4
MW-101[1]	Fluvial	Performance	Semiannual	276204.09	801110.27	291.74	291.98	89.0	15	104.0
MW-102B	Fluvial	Boundary	Biennial	275760.59	800707.72	311.40	312.07	120.5	20	140.5
MW-103	Fluvial	Performance	Annual	278690.88	805159.83	301.37	301.89	70.0	20	90.0
MW-104	Fluvial	Performance	Annual	278676.47	805417.03	291.98	292.18	70.5	20	90.5
MW-107[1]	Fluvial/Intermediate	Sentinel	Semiannual	278419.07	803009.93	304.92	305.18	128.0	15	143.0
MW-108	Fluvial/Intermediate	Sentinel	Semiannual	277658.02	802985.53	303.07	303.25	160.0	10	170.0
MW-140	Intermediate	Sentinel	Annual	279061.29	801715.68	298.12	298.16	224.6	20	244.6
MW-141	Intermediate	Sentinel	Semiannual	278019.19	802571.25	303.71	303.70	148.7	20	168.7
MW-142	Fluvial	Performance	Annual	278056.03	801629.12	291.18	291.49	85.0	20	105.0
MW-143	Fluvial	Performance	Semiannual	278301.35	801201.48	290.66	290.90	78.6	20	98.6
MW-198	Fluvial	Performance	Annual	277775.91	802142.37	291.78	292.20	90.3	15	105.3
MW-199A	Intermediate	Sentinel	Annual	277756.40	802573.52	301.53	301.84	146.1	15	161.1
MW-199B	Fluvial	Performance	Semiannual	277751.74	802575.66	301.73	302.07	104.6	15	119.6
MW-200	Fluvial	Performance	Semiannual	277006.10	802859.39	300.18	300.51	102.9	15	117.9
MW-202A	Intermediate	Sentinel	Annual	278685.74	802111.27	299.23	299.69	176.2	15	191.2
MW-202B	Intermediate	Sentinel	Semiannual	278692.79	802112.04	299.51	299.74	118.8	15	133.8
MW-204A	Fluvial	Performance	Semiannual	276724.66	802168.25	292.21	292.49	133.3	15	148.3
MW-204B	Fluvial	Performance	Semiannual	276707.81	802167.07	292.71	293.00	94.9	15	109.9
MW-205A	Fluvial	Performance	Semiannual	277157.18	802277.24	292.30	292.40	141.3	15	156.3
MW-205B	Fluvial	Performance	Semiannual	277173.05	802277.84	292.16	292.30	97.3	15	112.3
MW-206A	Fluvial	Performance	Semiannual	277219.28	802792.28	299.92	300.35	127.3	15	142.4
MW-206B	Fluvial	Performance	Semiannual	277200.85	802794.78	299.90	300.12	96.7	15	111.7
MW-207A	Fluvial	Sentinel	Semiannual	277652.76	803192.01	304.05	304.45	149.9	15	164.9
MW-207B	Fluvial	Sentinel	Semiannual	277665.02	803193.27	304.06	304.42	108.5	15	123.5
MW-208A	Fluvial	Performance	Semiannual	277382.22	802799.08	302.21	302.40	184.2	15	199.2
MW-208B	Fluvial	Performance	Semiannual	277396.90	802814.96	301.79	302.08	106.7	15	121.7

TABLE 4
MAIN INSTALLATION LTM WELLS
RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN
Defense Depot Memphis, Tennessee

Well	Aquifer	Well Classification	Sample Frequency	Northing (ft)	Easting (ft)	Top of Casing Elevation	Ground Elevation (ft, msl)	Riser Length (ft)	Screen Length (ft)	Total Well Depth (ft, btoc)
MW-209A	Intermediate	Sentinel	Annual	277574.28	802507.10	298.05	298.36	189.0	15	204.0
MW-209B	Fluvial	Performance	Semiannual	277581.50	802520.13	298.49	298.72	102.3	15	117.3
MW-210A	Intermediate	Sentinel	Semiannual	277238.57	801958.11	289.61	289.70	177.0	15	192.0
MW-210B	Fluvial	Performance	Semiannual	277228.18	801951.94	289.29	289.53	97.0	15	112.0
MW-211	Intermediate	Sentinel	Annual	278000.59	802973.69	303.74	304.09	166.3	15	181.3
MW-214A	Fluvial	Performance	Semiannual	277877.62	803906.94	303.61	303.96	119.1	15	134.1
MW-214B	Fluvial	Performance	Semiannual	277875.84	803922.20	303.70	303.96	101.6	15	116.6
MW-215A	Fluvial	Performance	Annual	277298.37	804164.31	304.50	304.86	128.8	15	143.8
MW-215B	Fluvial	Performance	Annual	277298.27	804177.33	304.56	304.98	105.4	15	120.4
MW-216	Fluvial	Performance	Annual	276024.68	801995.93	297.34	297.63	99.9	15	115.0
MW-217	Fluvial	Performance	Semiannual	276670.60	805213.69	304.18	304.51	101.8	15	116.8
MW-218	Fluvial	Performance	Semiannual	276936.70	805628.44	305.60	306.00	98.9	15	114.0
MW-219	Fluvial	Boundary	Semiannual	276429.49	800460.96	295.13	295.00	98.0	15	113.0
MW-229	Intermediate	Sentinel	Biennial	279294.17	802836.96	311.78	312.09	188.4	20	208.4
MW-252	Intermediate	Sentinel	Annual	278789.21	801364.70	294.16	294.40	126.1	20	146.1
MW-253	Intermediate	Sentinel	Annual	278287.43	801191.42	290.47	290.80	118.3	20	138.3
MW-254	Memphis	Sentinel	Semiannual	279334.36	800857.53	292.84	293.28	285.8	20	305.8
MW-255	Memphis	Sentinel	Annual	279304.76	801226.84	291.84	292.38	284.7	20	304.7
MW-256	Intermediate	Sentinel	Semiannual	279301.82	801243.80	292.68	293.40	127.1	20	147.1
MW-257	Fluvial	Performance	Annual	278549.06	801340.58	292.22	292.67	85.5	15	100.5
MW-258	Fluvial	Performance	Semiannual	278125.81	804426.82	304.37	304.83	79.3	20	99.3
MW-259	Fluvial	Performance	Semiannual	276279.04	804450.97	290.77	291.44	98.6	20	118.6
MW-260	Fluvial	Performance	TBD	278398.46	804376.22	304.16	304.45	68.0	20	88.3
MW-261	Fluvial	Performance	TBD	276390.64	802591.62	293.52	293.79	90.0	20	110.3
DR1-1	Fluvial	Performance	Annual	276300.34	800855.57	293.14	293.42	121.7	20	141.7
DR1-1A	Fluvial	Performance	Annual	276307.34	800863.06	293.00	293.37	89.2	20	109.2
DR1-2	Fluvial	Performance	Annual	276536.64	801152.66	290.00	291.39	97.7	20	117.7
DR1-3	Fluvial	Performance	Semiannual	276527.27	801415.91	290.93	291.11	109.7	20	129.7
DR1-4	Fluvial	Performance	Annual	276231.20	801399.53	292.78	293.00	106.3	20	126.3
DR1-7	Fluvial	Performance	Annual	276791.26	801441.36	289.15	289.46	108.3	20	128.3
DR1-8	Fluvial	Performance	Annual	276752.48	800875.32	290.09	290.47	92.7	20	112.7
DR2-1	Fluvial	Performance	Semiannual	276772.10	806497.62	304.90	305.08	73.9	20	93.9
DR2-3	Fluvial	Performance	Semiannual	276539.12	806203.16	303.44	303.66	93.0	20	113.0
DR2-4	Fluvial	Performance	Annual	276455.62	806633.07	303.55	303.96	88.1	20	108.1
DR2-6	Fluvial	Performance	Semiannual	276643.99	805860.91	304.70	304.92	94.6	20	114.6
PMW21-03	Fluvial	Performance	Semiannual	276573.43	800742.52	292.11	292.72	90.3	20	110.3
PMW21-05	Fluvial	Performance	Semiannual	276628.32	801129.72	288.53	288.92	94.3	20	114.3
PMW92-02	Fluvial	Performance	Semiannual	276667.02	806476.47	304.17	304.35	94.8	10	104.8
PMW101-03A	Fluvial	Performance	Semiannual	276348.46	801198.37	291.61	291.99	119.2	20	139.2
PMW101-03B	Fluvial	Performance	Semiannual	276353.09	801194.14	291.55	291.82	99.3	20	119.3
PMW101-06A	Fluvial	Performance	Semiannual	276191.88	801187.45	292.13	292.72	120.0	20	140.0
PMW101-06B	Fluvial	Performance	Semiannual	276194.93	801183.96	292.17	292.40	99.3	20	119.3
PZ-03	Fluvial	Performance	Annual	276379.33	802941.05	298.51	298.98	108.9	10	118.9

Notes:
1) Samples to be collected from two screened intervals in MW-101 and MW-107
ft: feet
btoc: below top of casing
msl: mean sea level

FIGURES

Figure 1

SITE LOCATION MAP

RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN

DEFENSE DEPOT MEMPHIS, TENNESSEE

Projection: NAD 1927 StatePlane Tennessee
Units: Feet

0 0.6 1.2 1.8 2.4 3 Miles

Installation Location
Memphis, Tennessee

Date: September 2011
Edition: Rev 0

HDR

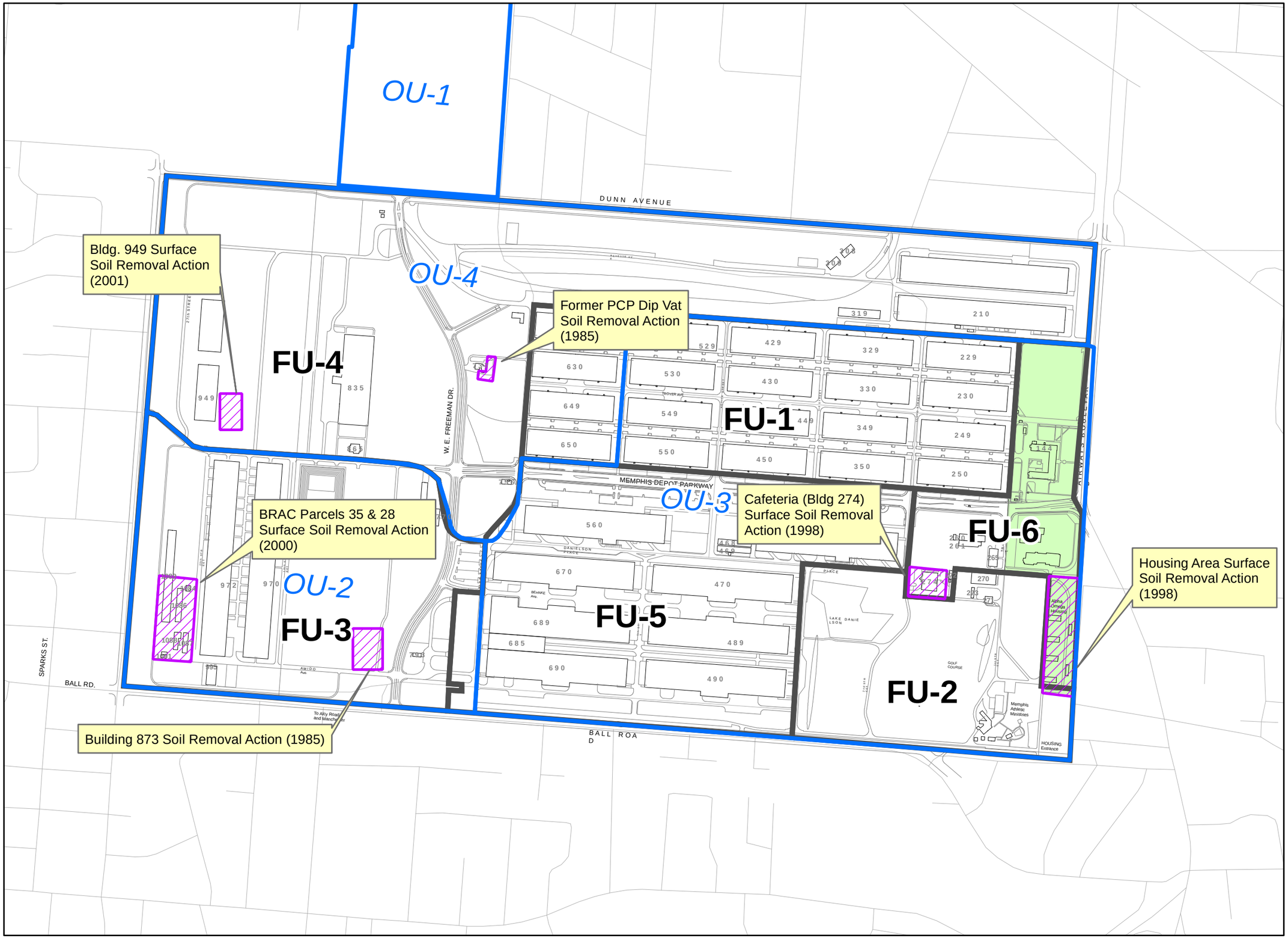

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Figure 2

UNIT BOUNDARIES AND RESPONSE ACTIONS

RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN

DEFENSE DEPOT MEMPHIS, TENNESSEE

Legend

- Operable Units (OU-#)
- Functional Unit Boundary (FU-#)
- Areas Available for Unrestricted Use
- Past Response Actions

Projection: NAD 1927 StatePlane Tennessee
Units: Feet

0 250 500 1,000

Scale in Feet

Installation Location
Memphis, Tennessee

Date: August 2014
Edition: Rev 1

HDR

N
W E
S

Figure 3

DUNN FIELD REMOVAL ACTIONS, INTERIM REMEDIAL ACTION AND DISPOSAL SITE REMEDIAL ACTION

RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN

DEFENSE DEPOT MEMPHIS, TENNESSEE

Legend

- Original Dunn Field Perimeter
- Unrestricted Use Area from ROD
- Area Designations
- IRA Recovery Well
- IRA Discharge Conveyance Line
- Removal Actions
- Disposal Sites Excavation Area

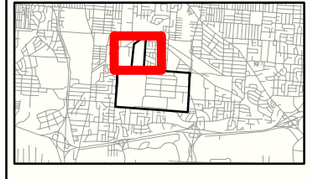
Projection: NAD 1927 StatePlane Tennessee
Units: Feet

Aerial Photo Date: 2006

0 150 300 600

Feet

Installation Location
Memphis, Tennessee



Date: September 2011
Edition: Rev 0

HDR

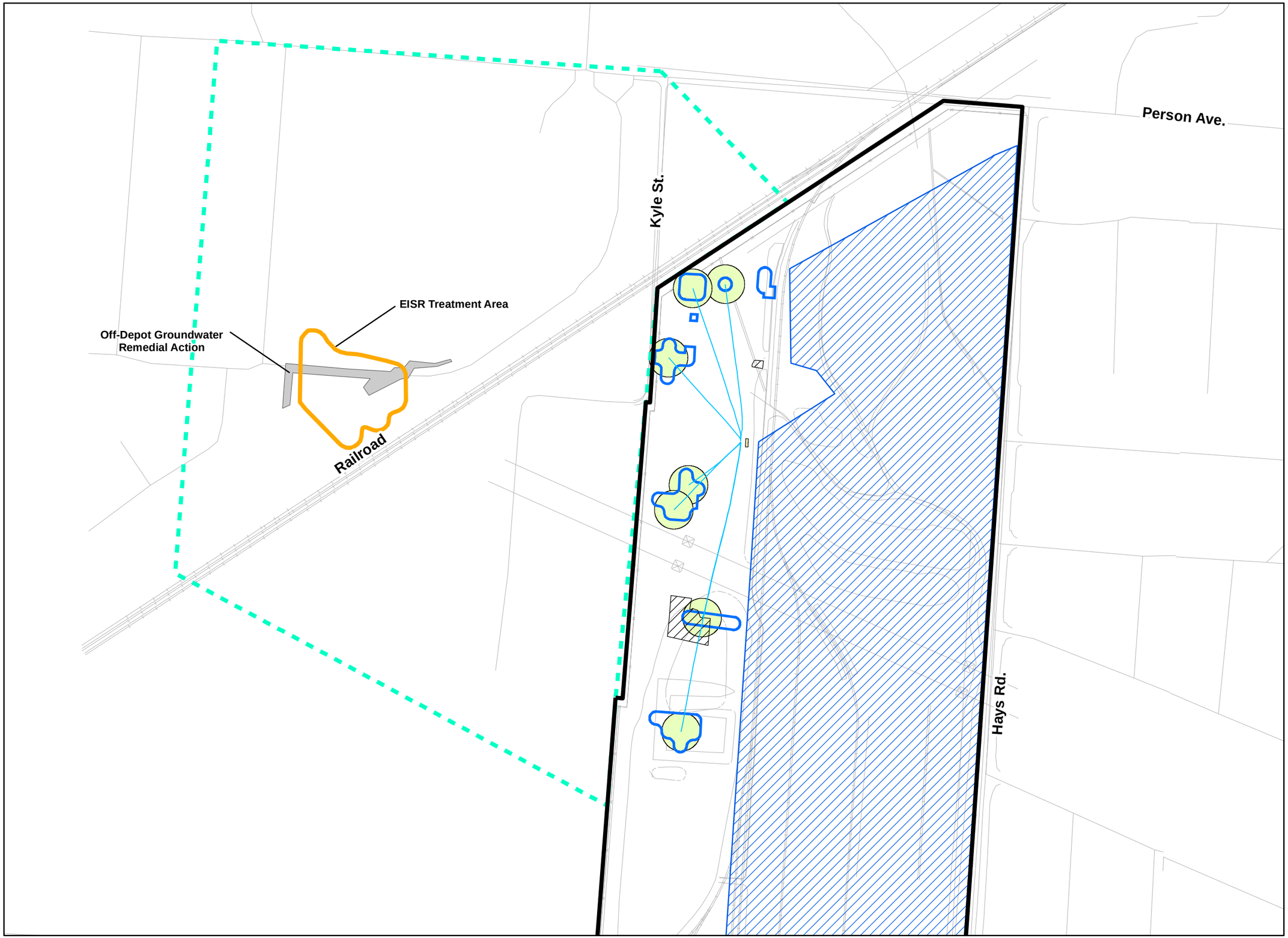


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Figure 4

DUNN FIELD SOURCE AREAS AND OFF-DEPOT GROUNDWATER REMEDIAL ACTIONS

RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN

DEFENSE DEPOT
MEMPHIS, TENNESSEE

Legend

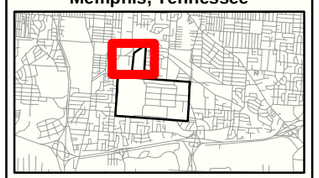
- Original Dunn Field Perimeter
- Unrestricted Use Area from ROD
- Off-Site Treatment Area
- EISR Treatment Area
- Fluvial SVE Well - 60-foot radius of influence
- Fluvial SVE Conveyance Line
- SVE Control Building
- Loess Excavation Areas
- Loess Thermal-Enhanced SVE Treatment Areas
- Air Sparge-SVE Area

Projection: NAD 1927 StatePlane Tennessee
Units: Feet

Aerial Photo Date: 2006

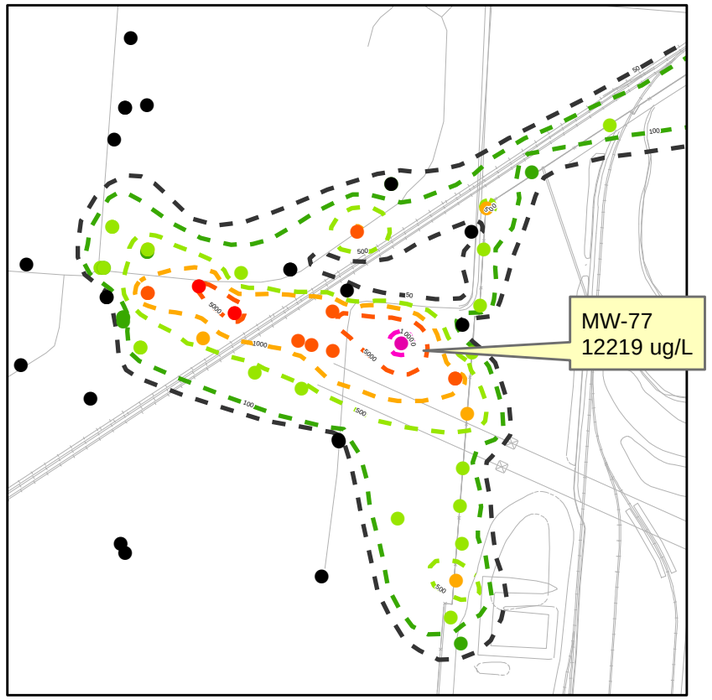
0 100 200 400
Feet

Installation Location
Memphis, Tennessee

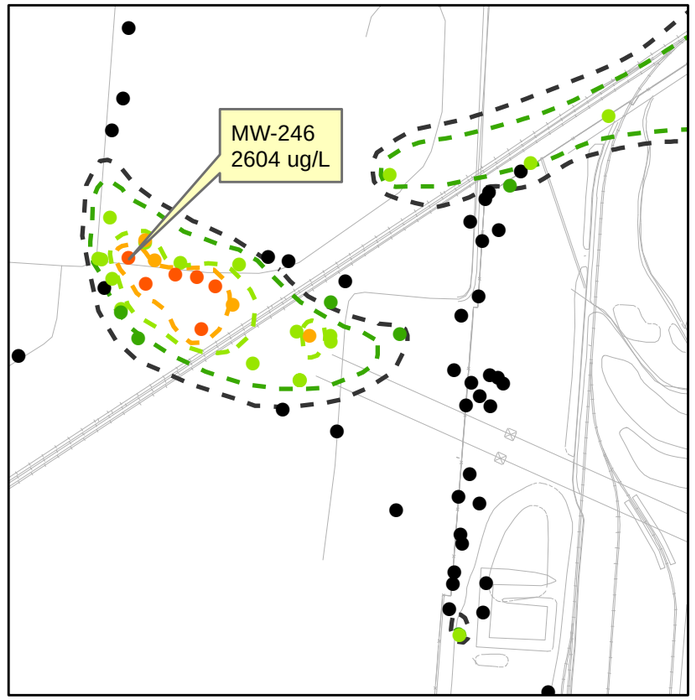


Date: September 2011
Edition: Rev 0

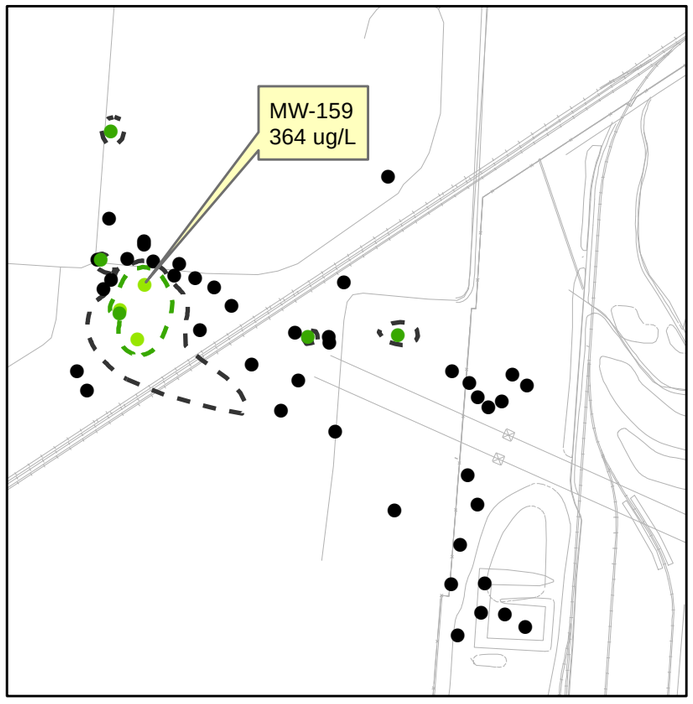
HDR



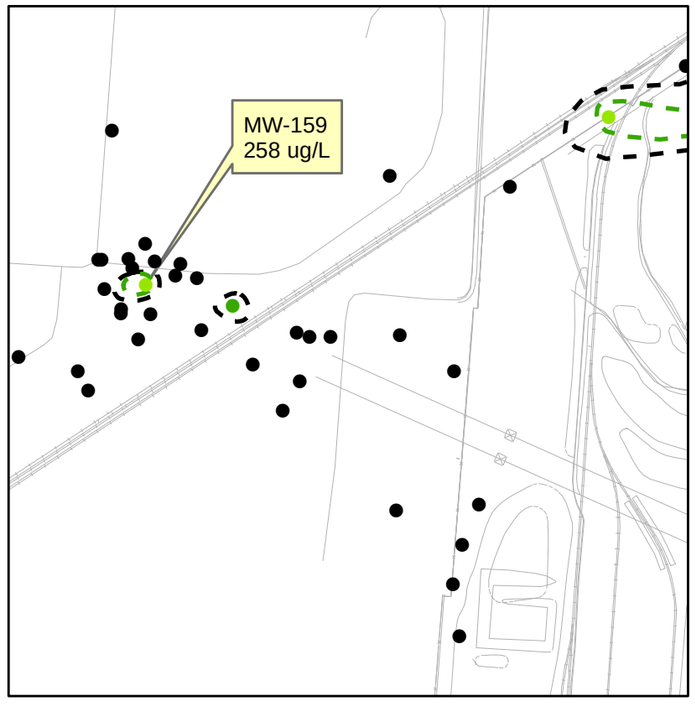
APRIL 2007



APRIL 2009



APRIL 2011



APRIL 2013

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Figure 5

DUNN FIELD TOTAL CVOC CONCENTRATIONS, 2007 - 2013

RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN

DEFENSE DEPOT
MEMPHIS, TENNESSEE

Legend

Total CVOC Isopleth (ug/L)

- 50
- 100
- 500
- 1000
- 5000
- 10000

Total CVOC Ranges (ug/L)

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

0 200 400 600 800
Feet

Installation Location
Memphis, Tennessee

Date: August 2014
Edition: Rev 1

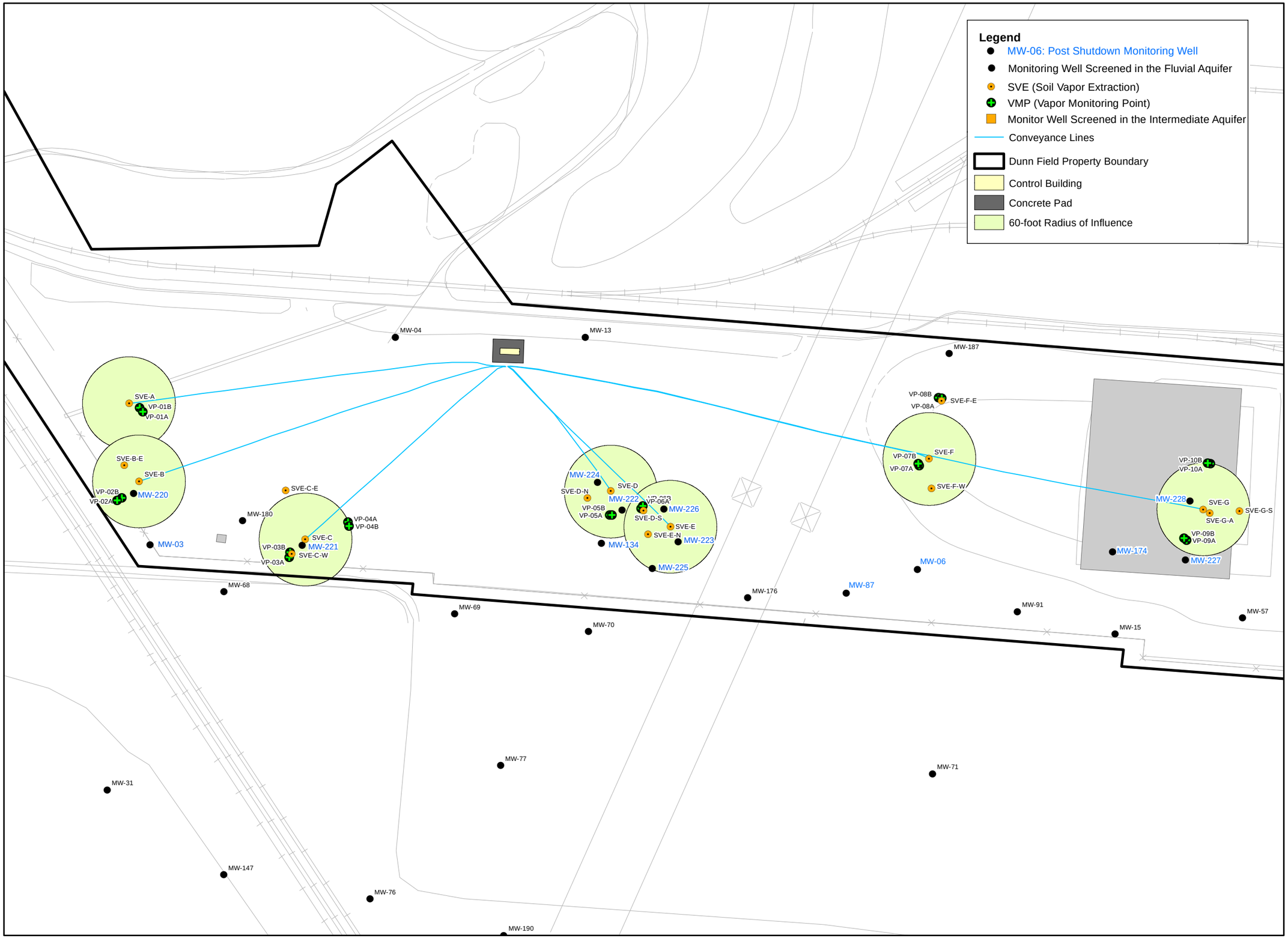

Figure 6

FLUVIAL SVE SYSTEM

RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN

DEFENSE DEPOT MEMPHIS, TENNESSEE

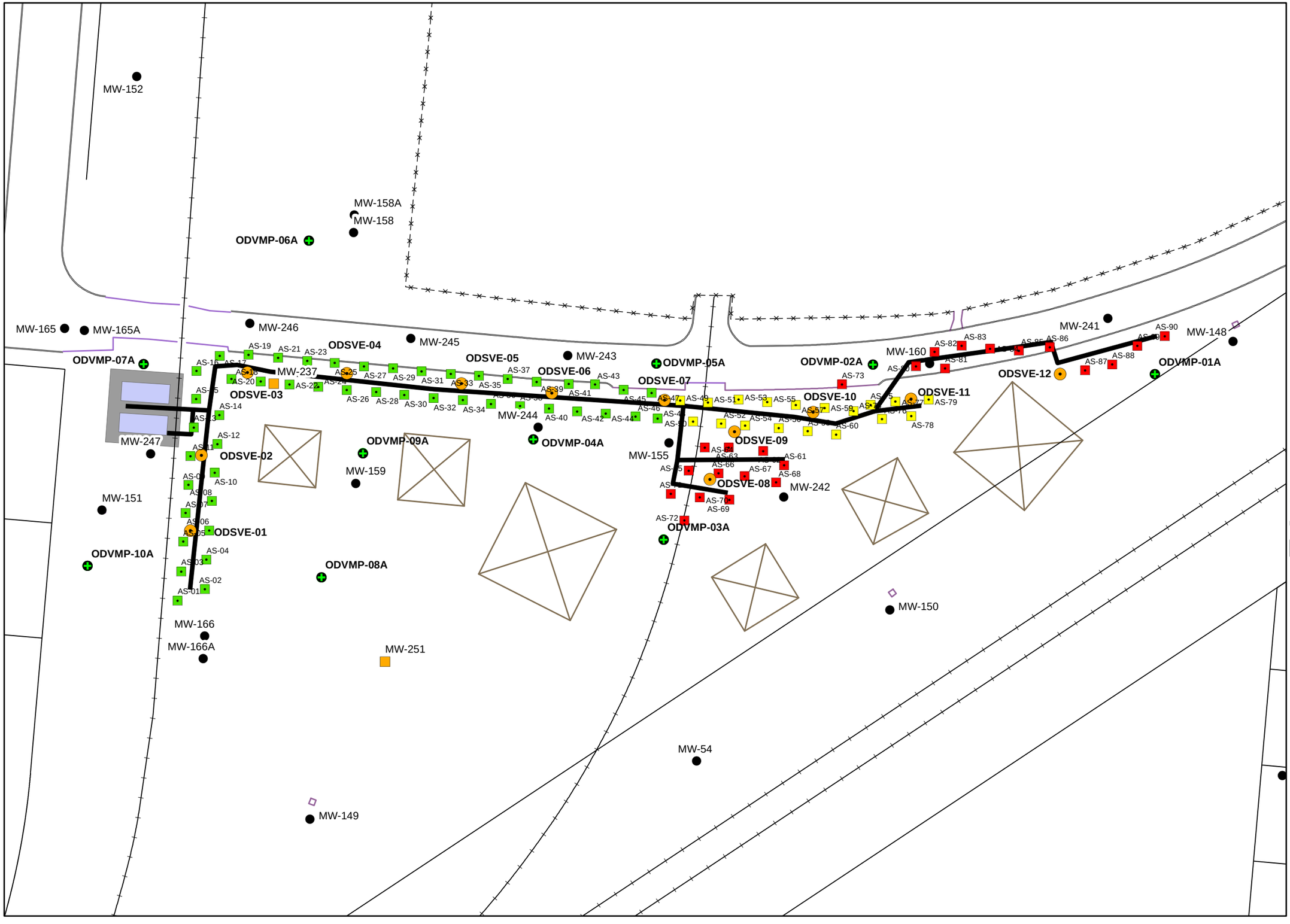

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Figure 7

AS-SVE SYSTEM PLAN MAP

RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN

DEFENSE DEPOT MEMPHIS, TENNESSEE

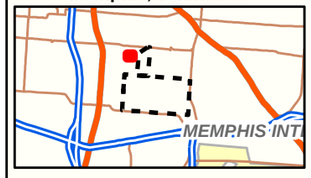
Legend

- Monitoring Well Screened in the Fluvial Aquifer
- Monitoring Well Screened in the Intermediate Aquifer
- AS Location (full time)
- AS Location (1/2 time)
- AS Location (closed)
- VMP Location
- SVE Location
- Conveyance Lines
- SVE Compound

0 25 50 75 100

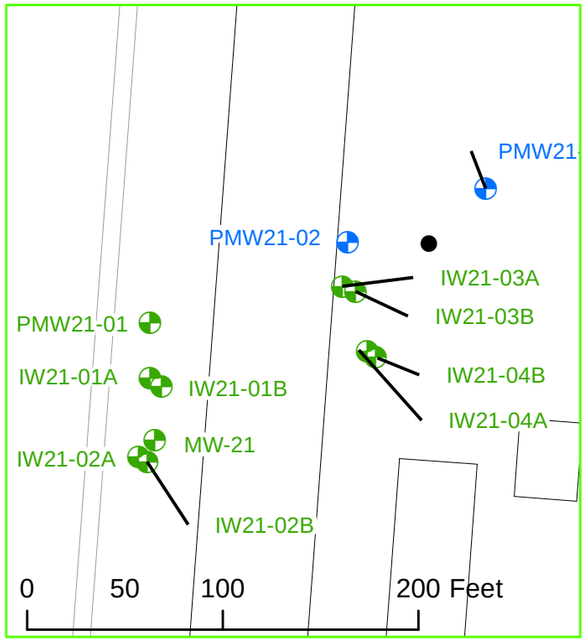
Feet

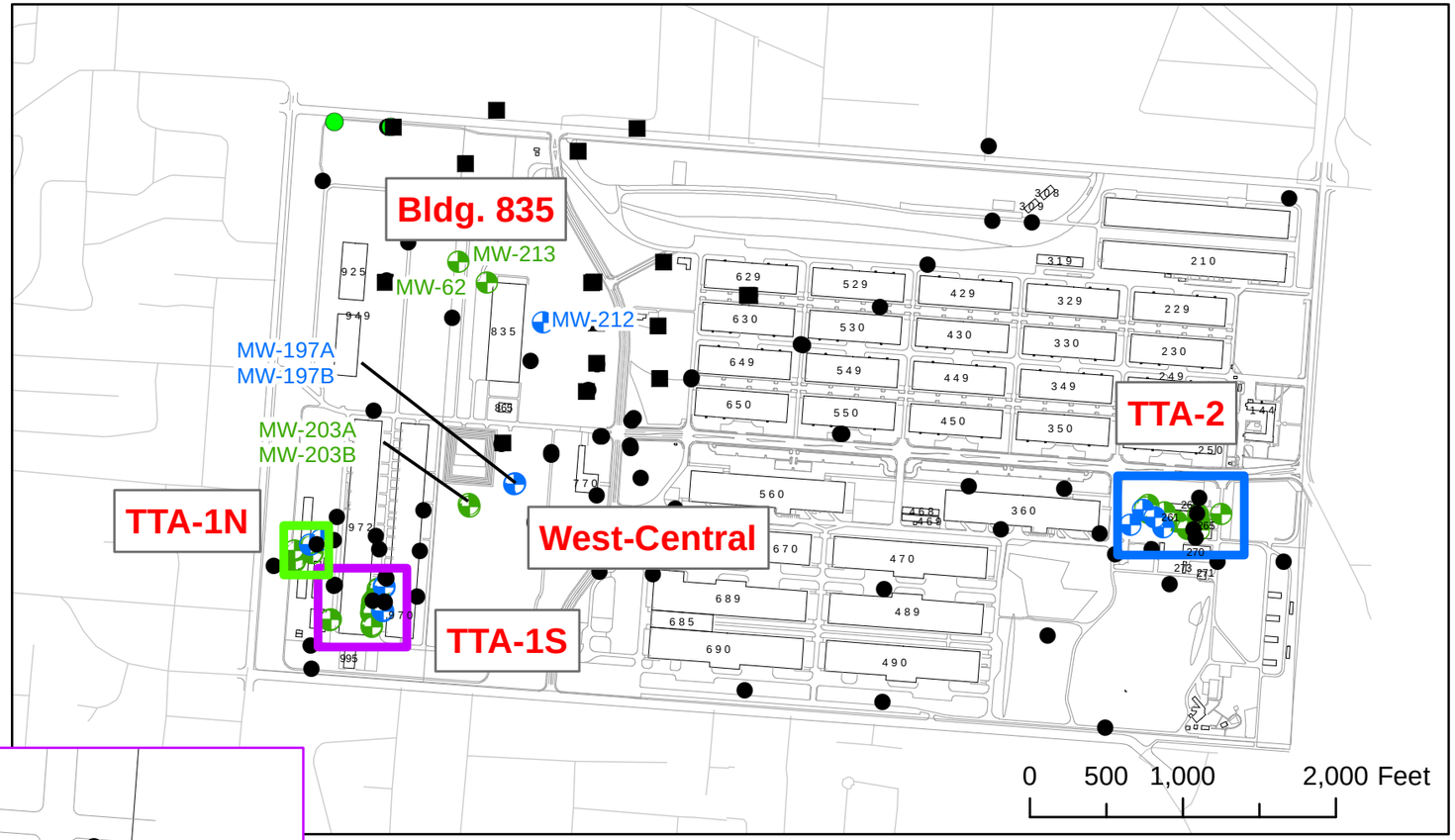
Installation Location
Memphis, Tennessee

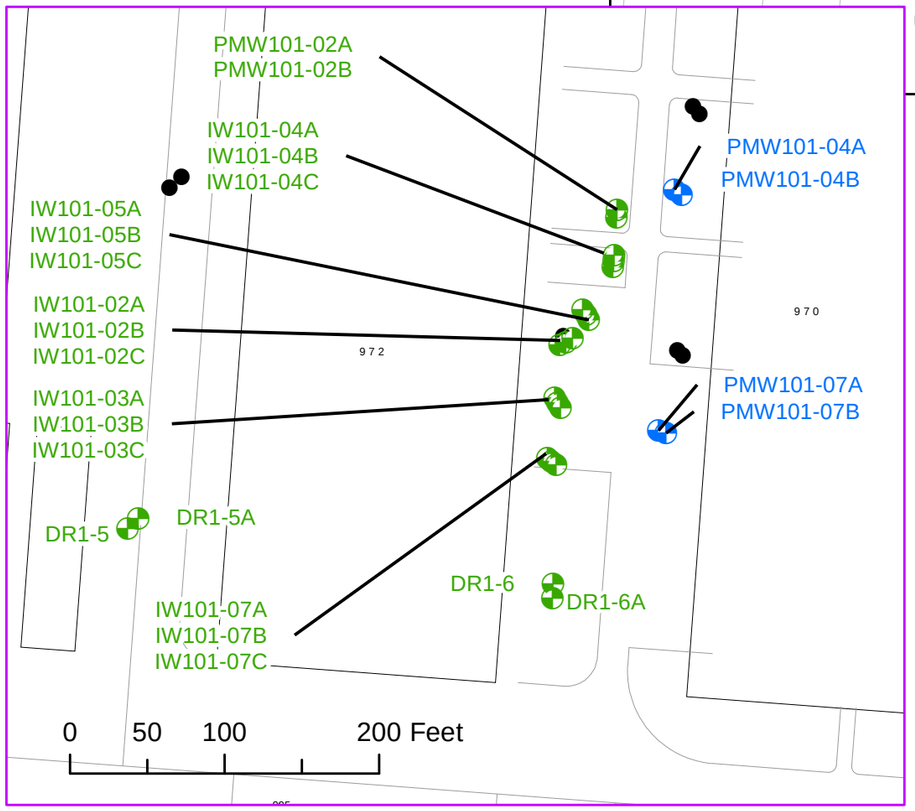


Date: August 2014
Edition: Rev 1

HDR







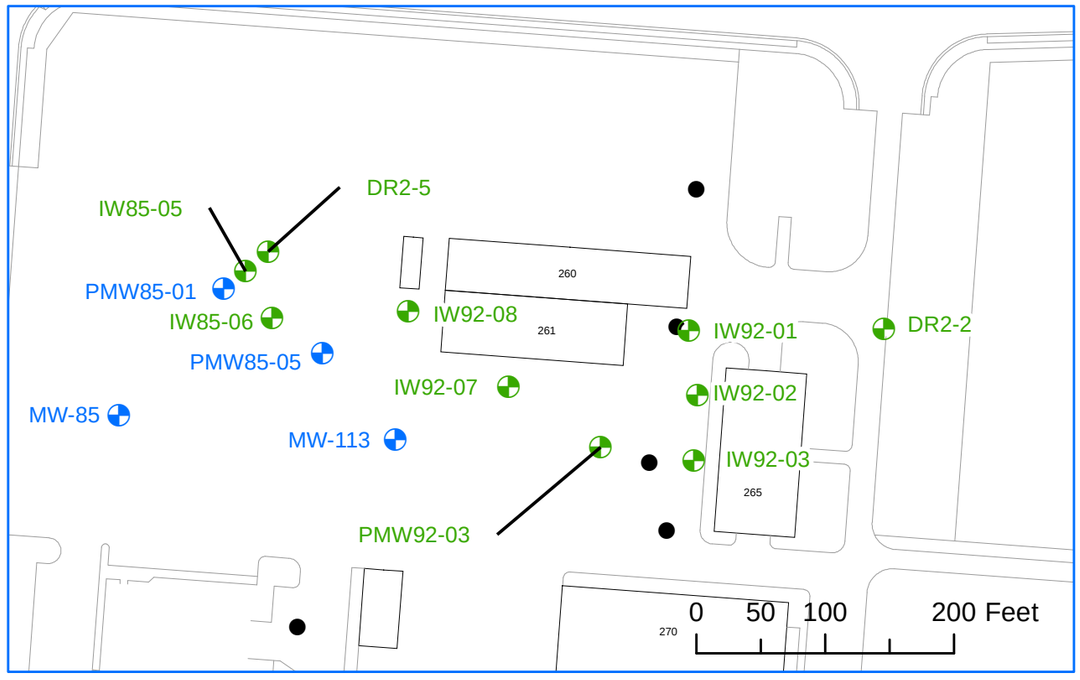


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Figure 8

MAIN INSTALLATION EBT WELLS

RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN

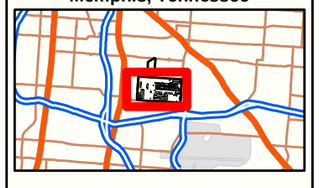
DEFENSE DEPOT MEMPHIS, TENNESSEE

Legend

- Monitoring Well Screened in the Fluvial Aquifer
- Monitoring Well Screened in the Transition Zone or Intermediate Aquifer
- Monitoring Well Screened in the Memphis Aquifer
- EBT Injection Well
- EBT Performance Monitoring Well

Projection: NAD 1927 StatePlane Tennessee
Units: Feet

Installation Location
Memphis, Tennessee



Date: August 2014
Edition: Rev 1

HDR

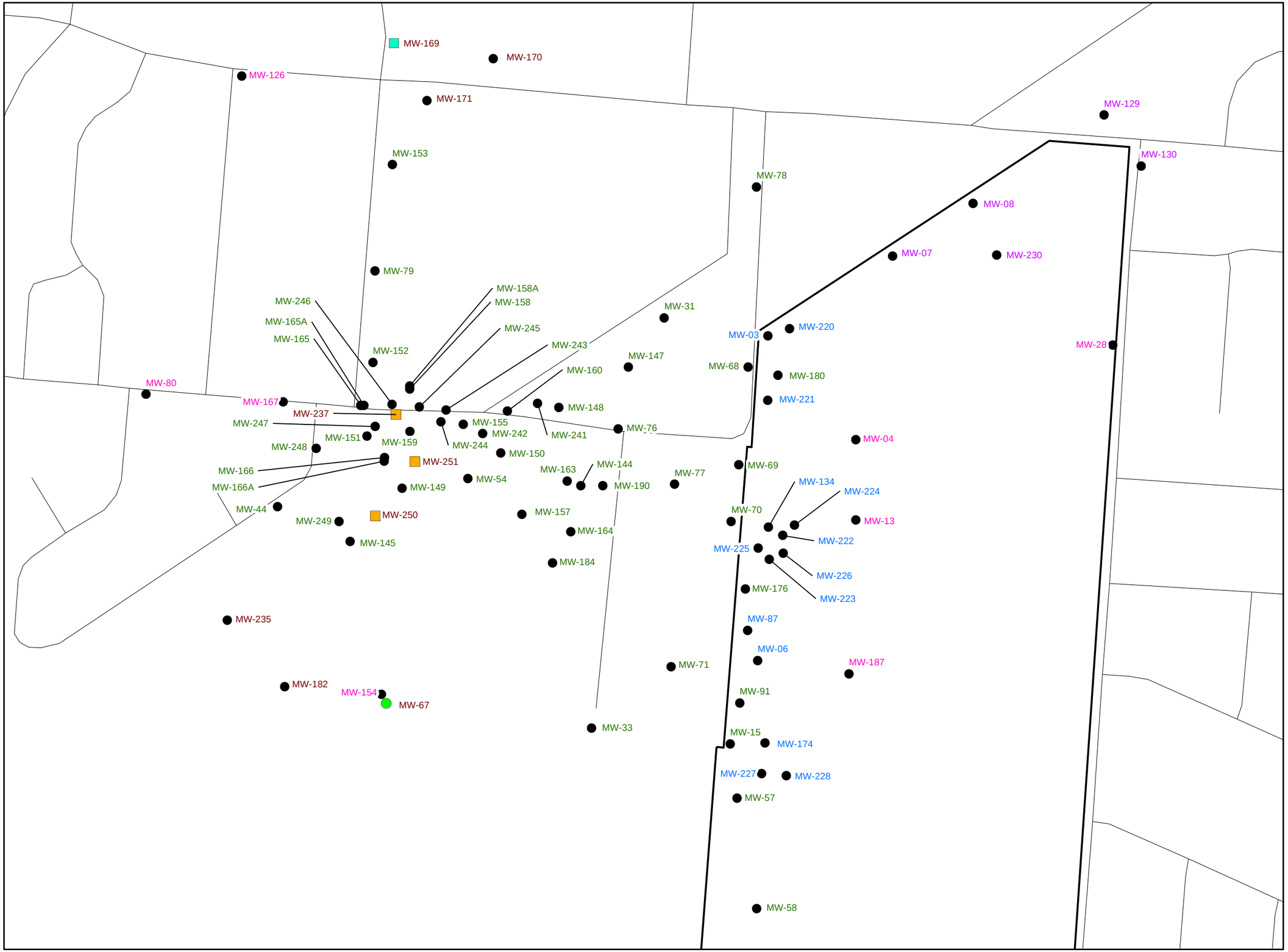


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Figure 9

DUNN FIELD LTM WELLS

RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN

DEFENSE DEPOT MEMPHIS, TENNESSEE

Legend

- Monitoring Well Screened in the Fluvial Aquifer
- Monitoring Well Screened in the Intermediate Aquifer
- Monitoring Well Screened in the Transition Zone
- Monitoring Well Screened in the Memphis Aquifer

Off Depot Well Classification

- MW-04 Background
- MW-07 Background-NE
- MW-250 Sentinel
- MW-15 Performance
- MW-03 Performance-FSVE

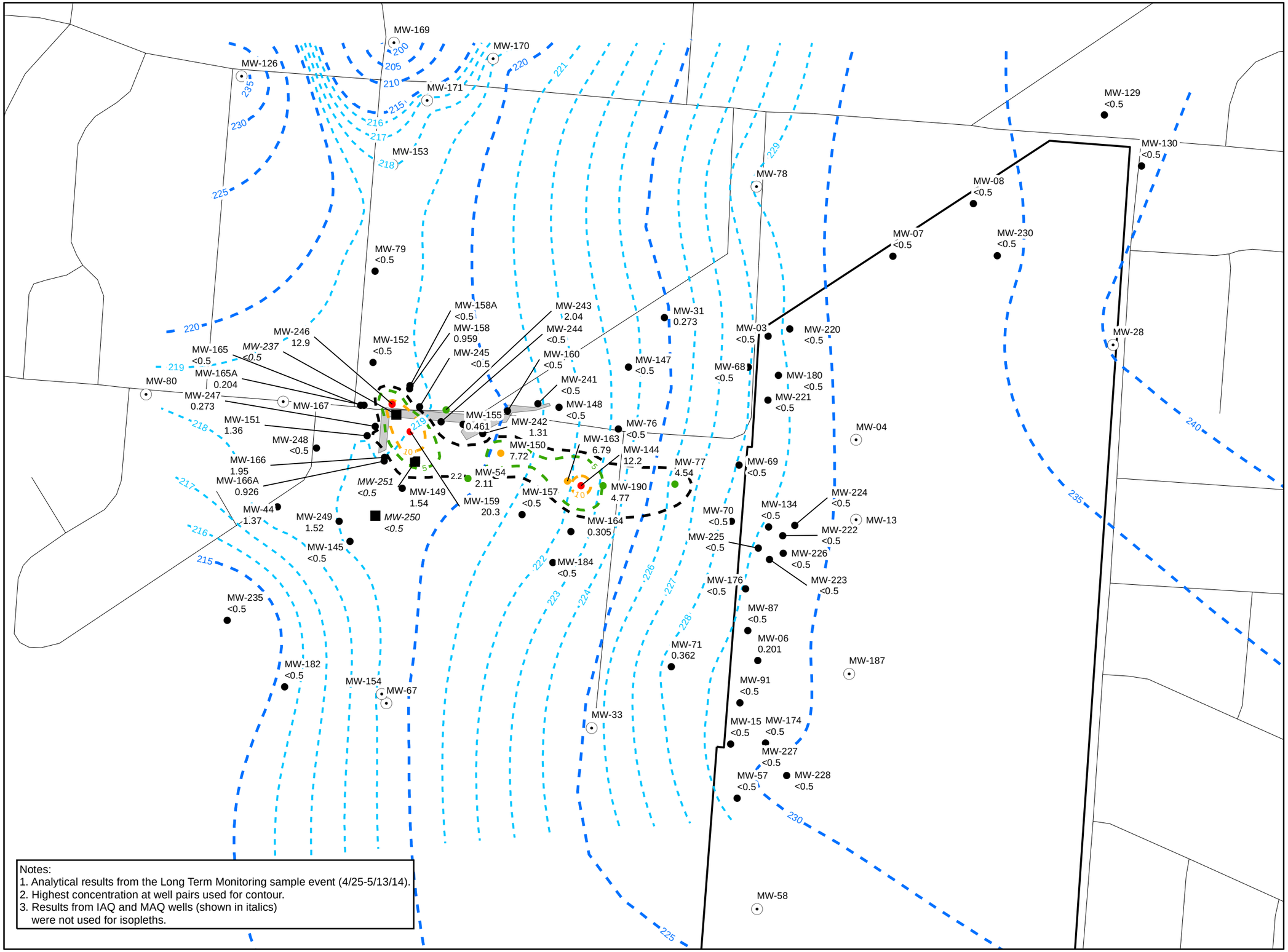
Dunn Field Boundary

0 150 300 450

Feet

Installation Location
Memphis, Tennessee

Date: August 2014
Edition: Rev 1



Notes:
1. Analytical results from the Long Term Monitoring sample event (4/25-5/13/14).
2. Highest concentration at well pairs used for contour.
3. Results from IAQ and MAQ wells (shown in italics) were not used for isopleths.

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Figure 10

DUNN FIELD
TeCA CONCENTRATIONS

RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN

DEFENSE DEPOT MEMPHIS, TENNESSEE

Legend

TeCA Ranges (ug/L)

- 0-2
- 2-5
- 5-10
- 10-50
- Not Sampled

TeCA Isopleth (ug/L)

- 2.2
- 5
- 10

Air Sparge Well Area

Original Dunn Field Property Boundary

Potentiometric surface of the Fluvial Aquifer 1-ft. contour

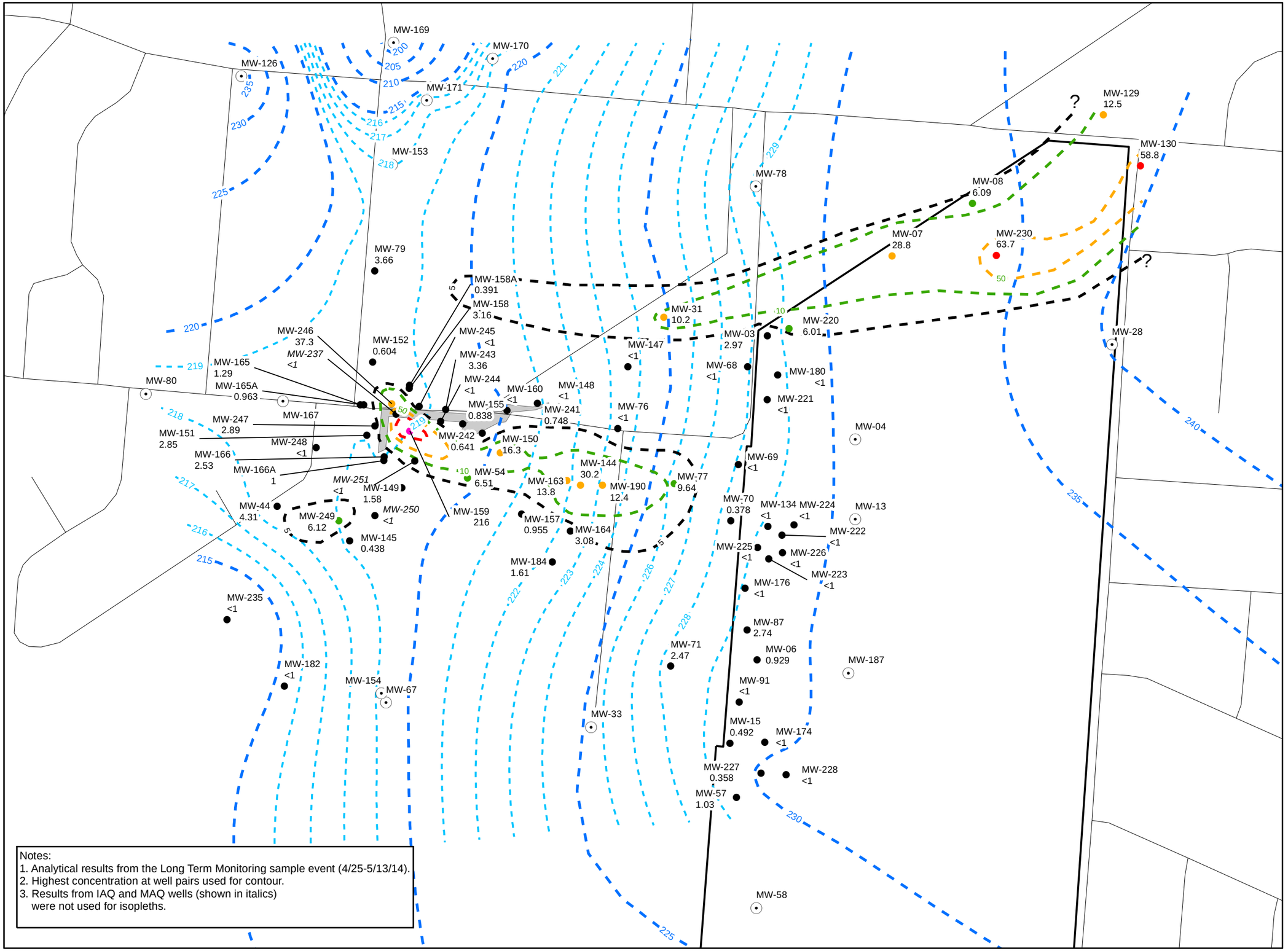
Potentiometric surface of the Fluvial Aquifer 5-ft. contour

0 100 200 300

Feet

Installation Location
Memphis, Tennessee

Date: August 2014
Edition: Rev 1



Notes:
1. Analytical results from the Long Term Monitoring sample event (4/25-5/13/14).
2. Highest concentration at well pairs used for contour.
3. Results from IAQ and MAQ wells (shown in italics) were not used for isopleths.

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Figure 11

DUNN FIELD TCE CONCENTRATIONS

RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN

DEFENSE DEPOT MEMPHIS, TENNESSEE

Legend

TCE Ranges (ug/L)

- 0-5
- 5-10
- 10-50
- 50-100
- 100-300
- Not Sampled

TCE Isopleth (ug/L)

- 5
- 10
- 50
- 100

Air Sparge Well Area

Original Dunn Field Property Boundary

Potentiometric surface of the Fluvial Aquifer 1-ft. contour

Potentiometric surface of the Fluvial Aquifer 5-ft. contour

0 100 200 300

Feet

Installation Location
Memphis, Tennessee

Date: August 2014
Edition: Rev 1

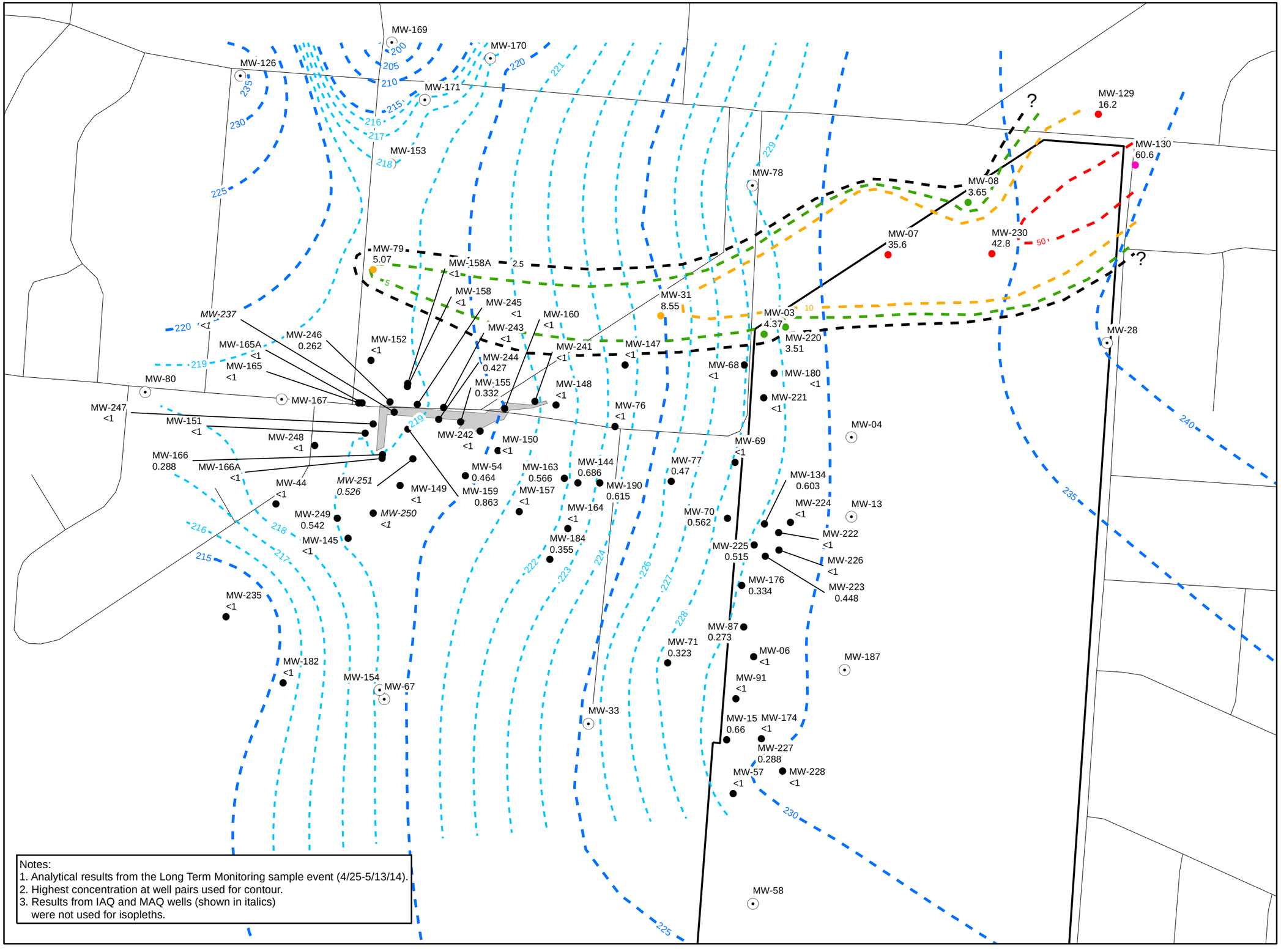

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Figure 12

DUNN FIELD
PCE CONCENTRATIONS

RA-O AND LTM QUALITY
ASSURANCE PROJECT PLAN

DEFENSE DEPOT
MEMPHIS, TENNESSEE

Legend

PCE Ranges (ug/L)

- 0 - 2.5
- 2.5 - 5
- 5 - 10
- 10 - 50
- 50 - 250
- Not Sampled

PCE Isopleth (ug/L)

- 2.5
- 5
- 10
- 50

Air Sparge Well Area

Original Dunn Field
Property Boundary

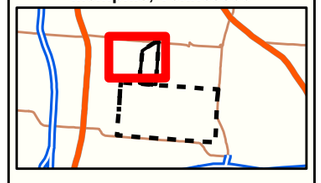
Potentiometric surface of the Fluvial Aquifer 1-ft. contour

Potentiometric surface of the Fluvial Aquifer 5-ft. contour

0 100 200 300

Feet

Installation Location
Memphis, Tennessee



Date: August 2014
Edition: Rev 1

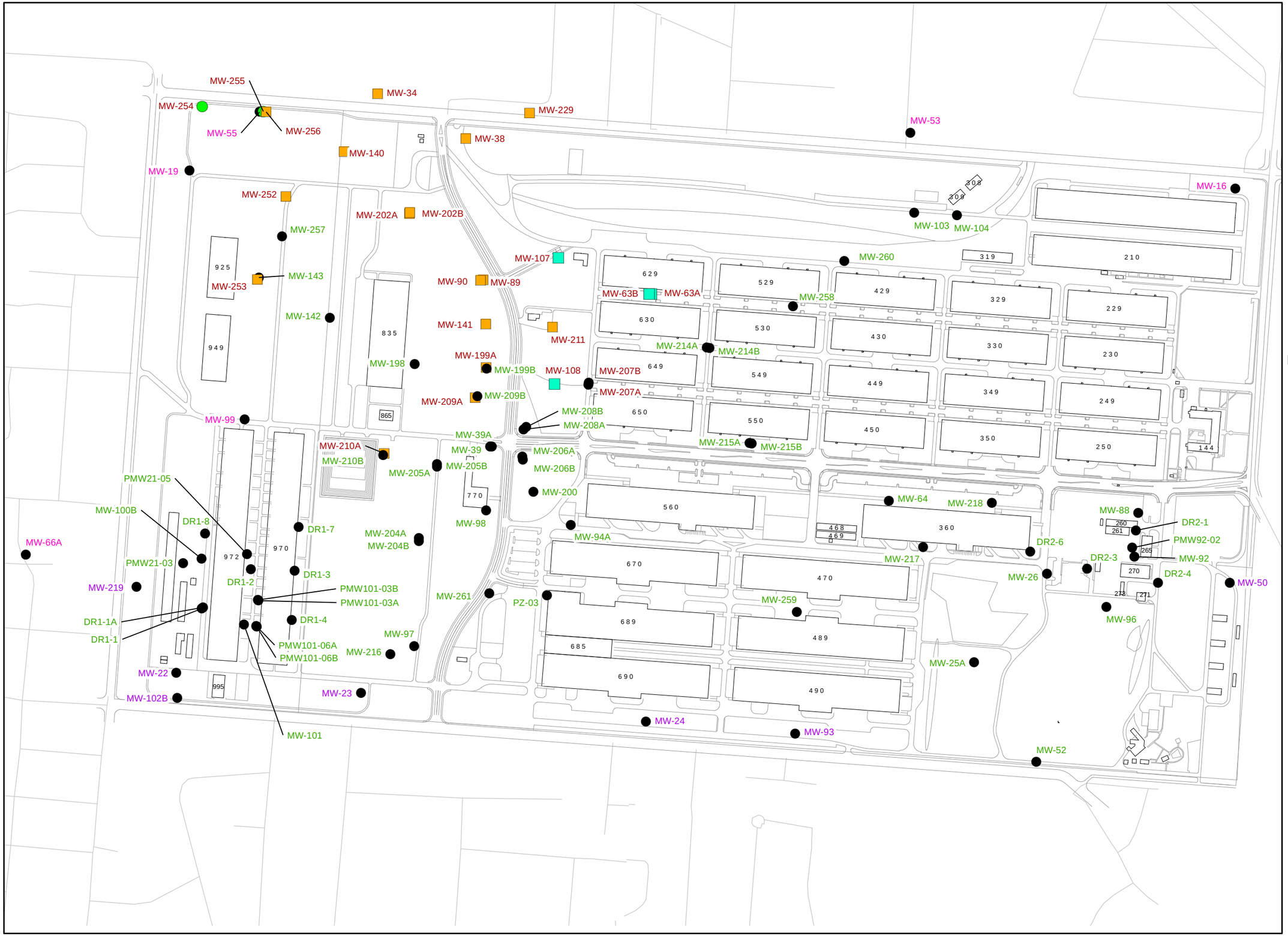


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Figure 13

MAIN INSTALLATION LTM WELLS

RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN

DEFENSE DEPOT MEMPHIS, TENNESSEE

Legend

- Monitoring Well Screened in the Fluvial Aquifer
- Monitoring Well Screened in the Intermediate Aquifer
- Monitoring Well Screened in the Transition Zone
- Monitoring Well Screened in the Memphis Aquifer

Well Classification

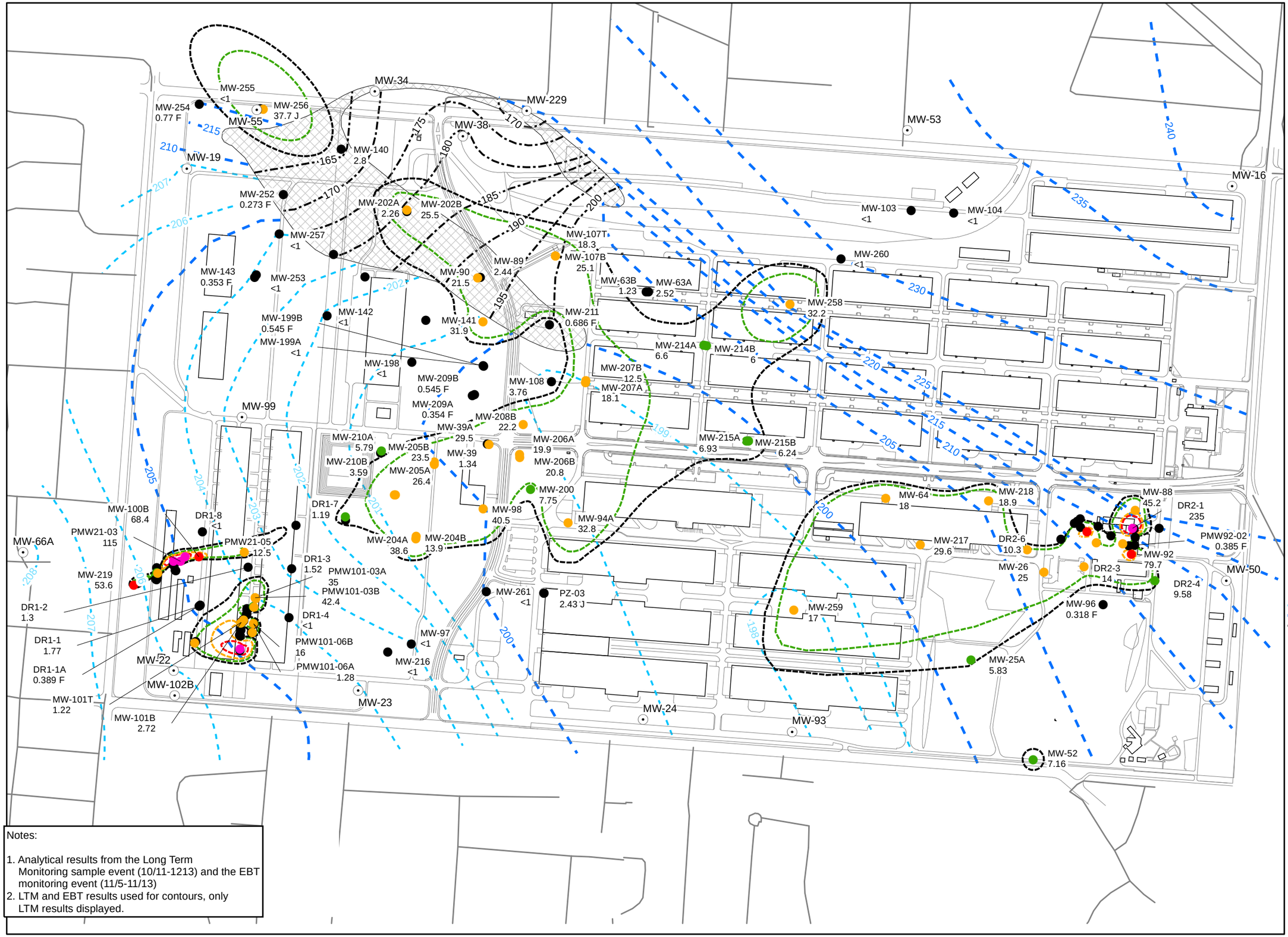
- MW-16 Background
- MW-34 Sentinel
- MW-88 Performance
- MW-219 Boundary

0 250 500 1,000

Feet

Installation Location
Memphis, Tennessee

Date: August 2014
Edition: Rev 1



Notes:

1. Analytical results from the Long Term Monitoring sample event (10/11-1213) and the EBT monitoring event (11/5-11/13)
2. LTM and EBT results used for contours, only LTM results displayed.

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Figure 14

MAIN INSTALLATION PCE CONCENTRATIONS

RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN

DEFENSE DEPOT MEMPHIS, TENNESSEE

Legend

PCE Ranges ug/L

- 0 - 5
- 5 - 10
- 10 - 50
- 50 - 100
- 100 - 300
- Not Sampled

PCE Isopleth ug/L

- 5
- 10
- 50
- 100
- Clay Elevation Exceeds Groundwater Elevation
- Potentiometric surface of the Fluvial Aquifer 1-ft. contour
- Potentiometric surface of the Fluvial Aquifer 5-ft. contour
- Potentiometric surface of the Intermediate Aquifer 5-ft. contour

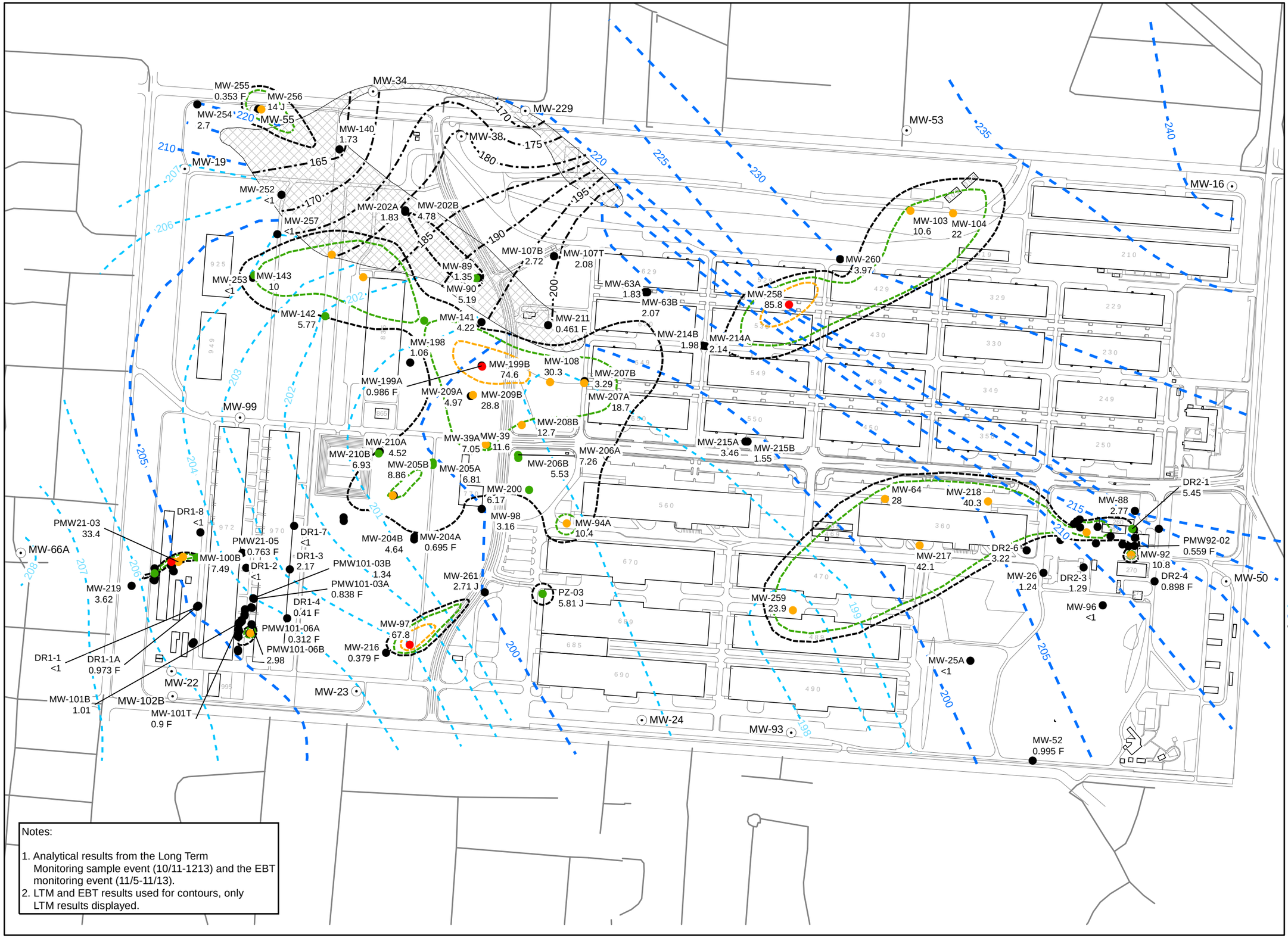
0 200 400 800

Feet

Installation Location
Memphis, Tennessee

Date: August 2014
Edition: Rev 1

HDR



Notes:

1. Analytical results from the Long Term Monitoring sample event (10/11-1213) and the EBT monitoring event (11/5-11/13).
2. LTM and EBT results used for contours, only LTM results displayed.

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Figure 15

MAIN INSTALLATION TCE CONCENTRATIONS

RA-O AND LTM QUALITY ASSURANCE PROJECT PLAN

DEFENSE DEPOT MEMPHIS, TENNESSEE

Legend

TCE Ranges ug/L

- 0 - 5
- 5 - 10
- 10 - 50
- 50 - 100
- 100 - 300
- Not Sampled

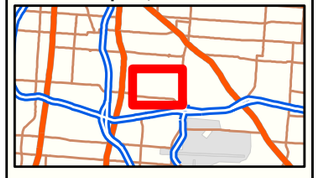
TCE Isopleth ug/L

- 5
- 10
- 50
- 100
- Clay Elevation Exceeds Groundwater Elevation
- Potentiometric surface of the Fluvial Aquifer 1-ft. contour
- Potentiometric surface of the Fluvial Aquifer 5-ft. contour
- Potentiometric surface of the Intermediate Aquifer 5-ft. contour

0 200 400 800

Feet

Installation Location
Memphis, Tennessee



Date: August 2014
Edition: Rev 1

HDR

APPENDIX A

UFP-QAPP WORKSHEETS

QAPP Worksheet #1 (UFP-QAPP Manual Section 2.1) -- Title and Approval Page

Remedial Action Operations and Long Term Monitoring Quality Assurance Project Plan, Defense Depot Memphis, Tennessee, Revision 1
Document Title

Department of the Army (DA)
Lead Organization

Thomas Holmes, HDR
Preparer's Name and Organizational Affiliation

9781 South Meridian Blvd., Suite 400, Englewood, CO 80112
404-295-3279, thomas.holmes@hdrinc.com
Preparer's Address, Telephone Number, and E-mail Address

24 November 2014
Preparation Date (Day/Month/Year)

Investigative Organization's Project Manager: Thomas Holmes
Signature
Thomas Holmes/ HDR/ 1 Dec 2014
Printed Name/Organization/Date

Investigative Organization's Project QA Officer: Lynn Lutz
Signature
Lynn Lutz / HDR / 1 Dec 2014
Printed Name/Organization/Date

Lead Organization's Program Manager: Carolyn Jones
Signature
Carolyn Jones/ ACSIM ODB/ 2 Dec 2014
Printed Name/Organization/Date

Approval Signatures:

Signature
Diedre Lloyd/USEPA Region 4/
Printed Name/Organization/Date

Signature
Jamie Woods/TDEC/
Printed Name/Organization/Date

QAPP Worksheet #2 (UFP-QAPP Manual Section 2.2.4) - QAPP Identifying Information

Site Number/Code: TN4210020570
Operable Unit: OUs 1, 2, 3 and 4
Contractor Name: HDR
Contract: W90FYQ-09-D-0005, Task Order DS01
Contract Title: Remedy Effectiveness Evaluation, Remedial Process Optimization, CERCLA Five-Year Review, and Environmental Restoration Program Support for Former Defense Depot Memphis, Tennessee
HDR Project Number: 192672

1. Identify guidance used to prepare QAPP: *Uniform Federal Policy for Quality Assurance Project Plans, Version 1*, March 2005 EPA-505-B-04-900A, DTIC ADA 427785

2. Identify regulatory program: CERCLA National Priorities List

3. Identify approval entities: USEPA Region 4, TDEC

4. Indicate whether the QAPP is a generic or a project-specific QAPP. (circle one)

5. List dates of scoping sessions that were held: Based on the status of environmental restoration and the approved work plans for DDMT, scoping sessions were not held.

6. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Approval Date
Remedial Action Sampling and Analysis Plan, Rev. 1 *Defense Depot Memphis Tennessee* *Volume 1, Field Sampling Plan* *Volume 2, Quality Assurance Project Plan* MACTEC Engineering and Consulting, November 2005	NA

7. List organizational partners (stakeholders) and connection with lead organization:
   USEPA Region 4, TDEC – oversight organizations

8. List data users: HDR, ACSIM-ODB, USEPA Region 4, TDEC

9. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below:
   All required information is provided in the QAPP text or the worksheets listed below.
   Worksheets 6 and 8 are not included; the required information is provided in QAPP Section 1.2.
   Worksheet 9 is not included. Scoping sessions were not conducted for development of this QAPP based on the status of environmental restoration and the approved RA work plans for DDMT.

Identify where each required QAPP element is located in the QAPP (provide section, worksheet, table, or figure number) or other project planning documents (provide complete document title, date, section number, page numbers, and location of the information in the document). Circle QAPP elements and required information that are not applicable to the project. Provide an explanation in the QAPP.

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Related Documents
Project Management and Objectives		
2.1 Title and Approval Page	- Title and Approval Page	WS 1
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	- Table of Contents - QAPP Identifying Information	WS 2
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	WS 3 WS 4
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table	WS 5 WS 7
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History, and Background	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)	 WS 10
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	- Site-Specific PQOs - Measurement Performance Criteria Table	WS 11 WS 12
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table	 WS 13
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	- Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table	WS 14 WS 15 WS 16

Measurement/Data Acquisition		
3.1 Sampling Tasks 3.1.1 Sampling Process Design and Rationale 3.1.2 Sampling Procedures and Requirements 3.1.2.1 Sampling Collection Procedures 3.1.2.2 Sample Containers, Volume, and Preservation 3.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures 3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures 3.1.2.5 Supply Inspection and Acceptance Procedures 3.1.2.6 Field Documentation Procedures	- Sampling Design and Rationale - Sample Location Map - Sampling Locations and Methods/ SOP Requirements Table - Analytical Methods/SOP Requirements Table - Field Quality Control Sample Summary Table - Sampling SOPs - Project Sampling SOP References Table - Field Equipment Calibration, Maintenance, Testing, and Inspection Table	WS 17 WS 18 WS 19 WS 20 WS 21 WS 22
3.2 Analytical Tasks 3.2.1 Analytical SOPs 3.2.2 Analytical Instrument Calibration Procedures 3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures 3.2.4 Analytical Supply Inspection and Acceptance Procedures	- Analytical SOPs - Analytical SOP References Table - Analytical Instrument Calibration Table - Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	 WS 23 WS 24 WS 25
3.3 Sample Collection Documentation, Handling, Tracking, and Custody Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample Handling and Tracking System 3.3.3 Sample Custody	- Sample Collection Documentation Handling, Tracking, and Custody SOPs - Sample Container Identification - Sample Handling Flow Diagram - Example Chain-of-Custody Form and Seal	 WS 26 WS 27
3.4 Quality Control Samples 3.4.1 Sampling Quality Control Samples 3.4.2 Analytical Quality Control Samples	- QC Samples Table - Screening/Confirmatory Analysis Decision Tree	WS 28
3.5 Data Management Tasks 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	- Project Documents and Records Table - Analytical Services Table - Data Management SOPs	WS 29 WS 30

Assessment/Oversight		
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses	- Assessments and Response Actions - Planned Project Assessments Table - Audit Checklists - Assessment Findings and Corrective Action Responses Table	 WS 31 WS 32
4.2 QA Management Reports	- QA Management Reports Table	WS 33
4.3 Final Project Report		
Data Review		
5.1 Overview		
5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	- Verification (Step I) Process Table - Validation (Steps IIa and IIb) Process Table - Validation (Steps IIa and IIb) Summary Table - Usability Assessment	WS 34 WS 35 WS 36 WS 37
5.3 Streamlining Data Review 5.3.1 Data Review Steps To Be Streamlined 5.3.2 Criteria for Streamlining Data Review 5.3.3 Amounts and Types of Data Appropriate for Streamlining		

QAPP Worksheet #3 (UFP-QAPP Manual Section 2.3.1) -- Distribution List

QAPP Recipients	Title	Organization	Telephone Number	E-mail Address	Control Number
Carolyn Jones	Program Manager	ACSIM ODB	(703) 545-2508	carolyn.a.jones28.civ@mail.mil	01
Joan Hutton	BRAC Environmental Coordinator	Calibre	(770) 317-4323	joan.hutton@calibresys.com	02
Tyler Jones	USACE Technical Manager	USACE, Tulsa	(918) 669-7072	tyler.p.jones@usace.army.mil	03
Laura Roebuck	USACE Technical Manager	USACE, Mobile	(251) 690-3480	laura.w.roebuck@usace.army.mil	04
Diedre Lloyd	USEPA Representative, Remedial Project Manager	USEPA Region 4	(404) 562-8855	lloyd.diedre@epa.gov	05
Jamie Woods	TDEC Representative, Remedial Project Manager	TDEC Division of Remediation	(901) 371-3041	jamie.woods@tn.gov	06
Tom Holmes	RA Project Manager	HDR	(404) 295-3279	thomas.holmes@hdrinc.com	07
Lynn Lutz	RA Project Chemist/ QA Manager	HDR	(303) 754-4200	lynn.lutz@hdrinc.com	08
Project File	--	HDR	(303) 754-4248	rebecca.miura@hdrinc.com	09

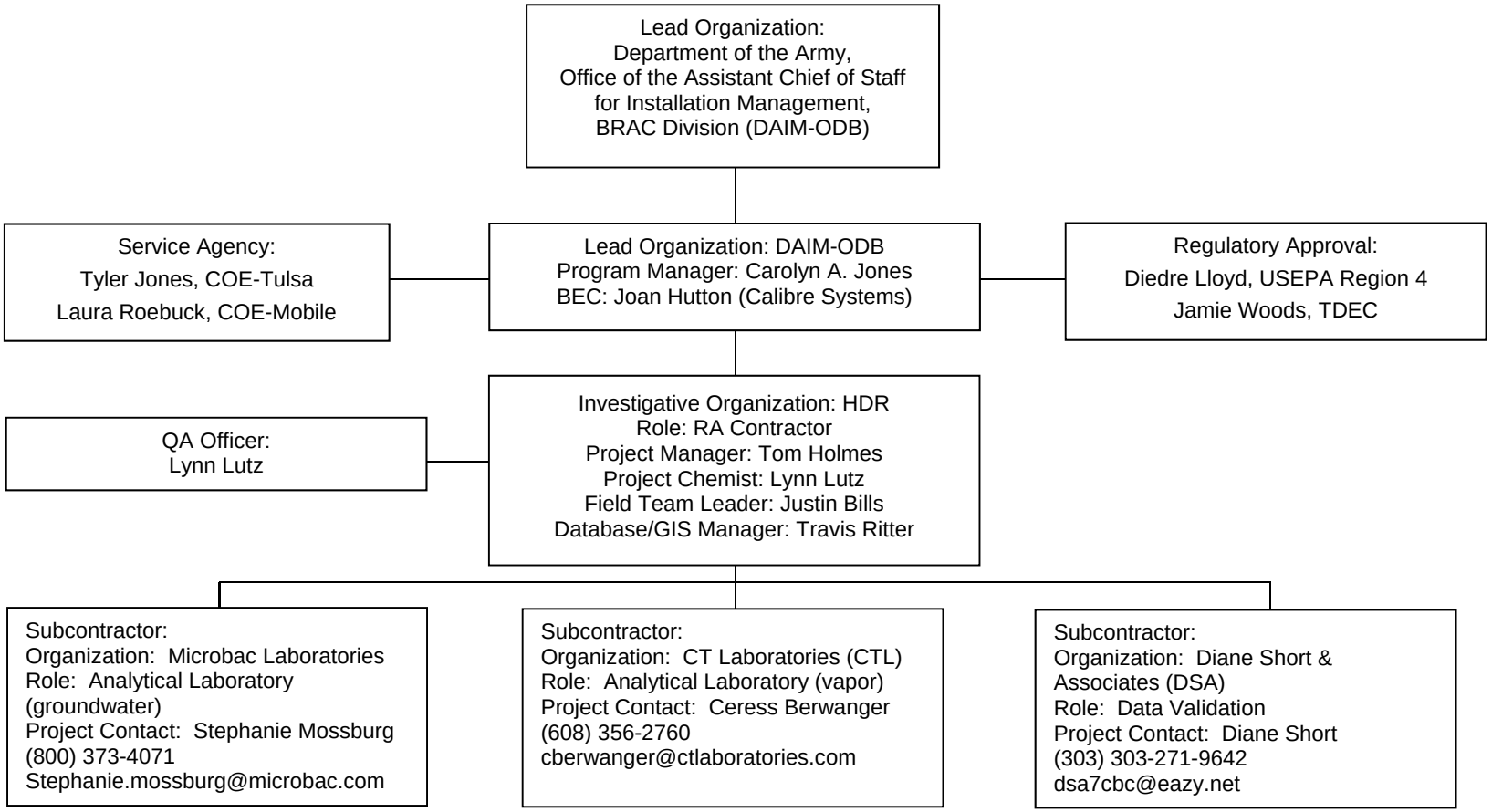
QAPP Worksheet #4 (UFP-QAPP Manual Section 2.3.2) – Project Personnel Sign-Off Sheet

Copies of this form to be signed by key project personnel from each organization to indicate that they have read the applicable sections of the QAPP and will perform the tasks as described.

Organization: _____

Project Personnel	Title	Telephone Number	QAPP Section(s)	Date QAPP Read	Signature

QAPP Worksheet #5 (UFP-QAPP Manual Section 2.4.1) -- Project Organizational Chart



QAPP Worksheet #7 (UFP-QAPP Manual Section 2.4.3) - Personnel Responsibilities and Qualifications Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Joan Hutton	BRAC Environmental Coordinator	CALIBRE	Oversees project and responds to EPA & TDEC	MS, Marine Science, 29 yrs. exp.
Glen Turney, PE	Program Manager	HDR	Manages restoration program; provides engineering and resource support	MBA, BS Chem Eng, 27 yrs. exp.
Tom Holmes, PG	Project Manager	HDR	Manages project and provides technical direction	MS Geophysics, 35 yrs. exp.
Lynn Lutz	Project Chemist/QA Manager	HDR	Coordinates analytical and data validation activities; oversees QA	BA Chemistry, 29 yrs. exp.
Justin Bills	Field Team Leader	HDR	Supervises field activities	BS Earth Science, 5 yrs. exp.
Travis Ritter	Database/GIS Manager	HDR	Maintains project database; prepares tables and figures	MS Env. Science, 15 yrs. exp.
Sylvia Fontes, CIH	Health & Safety Director	HDR	Oversees H&S for field activities	MS Occupational Health, 28 yrs. exp.
Stephanie Mossberg	Analytical Project Manager	Microbac Laboratories	Manages groundwater analyses	BS Biochemistry, 24 yrs. exp.
Patrick Letterer	Analytical Project Manager	CT Laboratories	Manages vapor analyses	BA Biology, 28 yrs. exp.
Diane Short	Validation Project Manager	Diane Short & Associates	Conducts independent analytical data validation	MS Chemistry and Molecular Genetics, 40 yrs. exp.

QAPP Worksheet #10 (UFP-QAPP Manual Section 2.5.2) - Problem Definition

Problem Definition:

USEPA added the Depot to the National Priorities List (NPL) in October 1992. USEPA, TDEC, and the Depot entered into a FFA in March 1995, which outlines the process for investigation and cleanup of the Depot sites under CERCLA.

The decision documents for DDMT are complete: *Record of Decision for Interim Remedial Action of the Groundwater at Dunn Field (OU-1)* (CH2M HILL, 1996); *Memphis Depot Main Installation Record of Decision* (CH2M HILL, 2001); *Memphis Depot Dunn Field Record of Decision* (CH2M HILL, 2004b); and the *Dunn Field Record of Decision Amendment* (e^2M, 2009).

The selected remedies have been implemented. The *Preliminary Close Out Report* (USEPA, 2010a) was approved in May 2010, and the DDMT NPL site status was revised to Construction Complete. Interim remedial action completion reports (IRACRs) have been approved for all actions. USEPA has concurred with operating properly and successfully (OPS) determinations for the remedies implemented on Federal property.

The *Third Five-Year Review, Revision 1* (HDR, 2012) was approved by USEPA in January 2013.

- The review noted that all selected remedies have been implemented; attainment of RGs has been documented in the subsurface soils at Dunn Field and attainment of cleanup goals in groundwater will be achieved through active treatment and natural attenuation. In the interim, exposure pathways that could result in unacceptable risks are being controlled and ICs are preventing exposure to, or the ingestion of, COCs. Long-term protectiveness will be verified by groundwater sampling performed during LTM and compliance monitoring at the MI and Dunn Field. Because the RAs at all OUs for DDMT are protective, the site is protective of human health and the environment.
- The review identified two issues for the MI: rebound in CVOC concentrations and the time required to achieve groundwater RAOs. Follow-up actions were recommended: restart EBT on the MI and re-evaluate the time required to achieve RAOs after the first year of EBT.

The estimated timeline for site closure includes the following:

- Main Installation
  - Final EBT sampling in November 2014.
  - SRI to be completed in November 2016.
  - FFS to be completed in January 2017.
  - ROD Amendment to be final in February 2018.
  - Additional remedial action June 2018 through June 2021.
  - MI LTM through 2021, with final quarterly compliance monitoring in 2022.
  - MI Remedial Action Completion Report to be completed in October 2023.
- Dunn Field
  - Off Depot AS/SVE operates through December 2016.
  - Off Depot LTM through 2018, with final quarterly compliance monitoring in 2019.

- Dunn Field Remedial Action Completion Report to be completed in October 2020.

- Final Closeout Report to be completed in April 2024

The selected remedy included EBT in the most contaminated areas and assumed that "untreated parts of the groundwater plume would degrade under natural attenuation" based on previous studies. EBT was re-started in five areas on the MI in November 2012 to address rebound and improve progress toward RAOs. However, 21 of 58 EBT wells exceeded MCLs in August 2014 and 48 of the 54 LTM wells sampled in April 2014 exceeded MCLs.

The annual EBT report, *Main Installation Year Three Enhanced Bioremediation Treatment Report* (HDR, 2014), determined that the timeline to meet RAOs in 2016 was not expected to be met. A revised schedule will require consideration of the CVOC concentration in the fluvial aquifer that requires active treatment versus natural attenuation, the need for remedial action in the intermediate aquifer, the continued use of EBT versus other remedial actions and the potential impact from off-site locations. A supplemental remedial investigation and focused feasibility study (SRI/FFS) will be performed to develop a remedial strategy to achieve RAOs throughout the MI.

The Dunn Field RAs are progressing well with continued reduction in groundwater concentrations. The treatment goal for the AS/SVE system is to reduce individual CVOC concentrations to 50 µg/L or less, and the system is to operate until concentrations in upgradient wells do not exceed 50 µg/L for individual CVOCs. Reduction in CVOC concentrations below MCLs is to be achieved through natural attenuation. In April 2014, individual CVOC concentrations were above 50 µg/L in only one LTM performance well and exceeded MCLs in 12 LTM performance wells and 5 LTM background wells impacted by an off-site plume. In comments on the 2013 LTM report, USEPA requested that the timeline to meet RAOs at Dunn Field also be reviewed.

Project Description:
Project activities covered by this QAPP are remedial action operations for EBT on the MI and AS/SVE in the Off Depot area near Dunn Field; and groundwater monitoring for EBT performance monitoring at the MI and LTM at the MI and Dunn Field. The project tasks are summarized on WS #14.

Project Decision Conditions:
The primary parameter for environmental restoration at DDMT is progress toward RAOs at the MI and Dunn Field demonstrated by decreasing CVOC concentrations in groundwater. A review of the project schedule and remedial alternatives will be performed as part of the SRI/FFS. This WS will be revised to incorporate project decision conditions following completion of the review.

QAPP Worksheet #11 (UFP-QAPP Manual Section 2.6.1) – Project Quality Objectives/Systematic Planning Process Statements

Who will use the data? HDR, U.S Army, U.S. EPA, TDEC.

What will the data be used for? HDR will use the data to evaluate RA performance and progress toward RAOs, and to recommend changes to optimize RA operations and LTM.

What type of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques) Data requirements are summarized for each activity below. The complete requirements are described in the RAWPs and LTM plans listed on WS #17.
EBT: field records for mixing each tank of sodium lactate solution and injections at each well; performance monitoring at each injection and monitoring well including field measurements (groundwater depth, specific conductivity, temperature, pH, dissolved oxygen, oxygen reduction potential, turbidity) and groundwater samples analyses (VOCs, dissolved gases, metabolic fatty acids and total organic carbon). Groundwater samples collected by low-flow purging with bladder pumps or with bailers where recharge is limited.
AS/SVE: records for system operating hours; air injection and extraction rates; vacuum and photoionization detector readings at SVE wells, system effluent and vapor monitoring points; effluent samples collected in SUMMA canisters and analyzed for VOCs.
LTM: Groundwater samples are collected with passive diffusion bags at most wells; low flow purging with bladder pumps or bailers are used where saturated thickness is insufficient for PDBs. Groundwater levels are collected at all wells; field parameters (specific conductivity, temperature, pH, dissolved oxygen, oxygen reduction potential, turbidity) are collected where bailers or bladder pumps are used for sampling. LTM groundwater samples are analyzed for VOCs only.
All sample analyses are conducted at off-site laboratories.

How "good" do the data need to be in order to support environmental decisions? Field measurements during groundwater monitoring provide screening level data that are sufficient to determine that water quality has stabilized for sample collection and to evaluate aquifer conditions as a factor in adjusting lactate solution concentrations for EBT injections. Field PID measurements at the AS/SVE system also provide screening level data for use in determining approximate VOC concentrations in the treatment area. Flow rate and vacuum measurements at the AS/SVE system are sufficient to determine that negative pressure is maintained throughout the treatment area.
The primary decisions on progress toward RAOs are based on definitive data from laboratory analyses of groundwater samples collected for EBT monitoring and LTM. EBT injections cause interferences in analytical results in many of the EBT wells, raising reporting limits by 2 to 50 times; the results are sufficient to evaluate progress toward RAOs. The interference should disappear after injections are completed and the final decision on meeting RAOs will be determined by analytical results that fully meet QA requirements.

How much data are needed? (number of samples for each analytical group, matrix, and concentration) The numbers of sample locations are provided in RAWPs and LTM plans. Groundwater samples are currently collected at 45 IWs and 13 PMWs for EBT on the MI, at 99 wells for MI LTM and 86 wells for Dunn Field LTM.

Where, when, and how should the data be collected/generated? Sample locations and frequencies for sample collections are provided in RAWPs and LTM plans. LTM samples are collected semiannually and individual wells are sampled at semiannual, annual and biennial frequencies. The current EBT implementation requires quarterly samples collected from February 2013 through November 2014.

Who will collect and generate the data? HDR and subcontract laboratories, Microbac (groundwater samples) and CT Laboratories (vapor samples).

How will the data be reported? Laboratories provide Level 4 reports following each sample event; complete analytical results with final DQE flags are provided in annual reports with data validation reports.

How will the data be archived? All project deliverables, including analytical reports, are electronically archived for 10 years following project completion. Annual reports for RA and LTM are included in the DDMT Administrative Record and will be stored permanently through the National Archives and Records Administration.

QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

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

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP4 (PDB, dedicated or portable pump, or bailer)	L-1 (SW-846 5030B/8260B)	Precision-overall	≤ 30% RPD	Field duplicate RPDs	S&A
		Precision-overall	≤ 30% RPD	MS/MSD RPDs	S&A
		Precision-lab	±30% RPD	LCS/LCSD RPDs	A
		Accuracy/bias	QSM v.5 App. C Table 24 (or lab if not in QSM) control limits	Surrogate spike recoveries	S&A
		Accuracy/bias	QSM v.5 App. C Table 24 (or lab if not in QSM) control limits	MS/MSD recoveries	S&A
		Accuracy/bias	QSM v.5 App. C Table 24 (or lab if not in QSM) control limits	LCS/LCSD recoveries	A
		Accuracy/bias-contamination	No analyte detected at ≥1/2 RL or > 10% sample concentration or regulatory limit	Method blanks	A
		Accuracy/bias-contamination	No analyte detected at ≥ RL	Equipment blanks, ambient blanks, trip blanks	S&A

QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group[1]	TOC
Concentration Level	Low/medium

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP4 (PDB, dedicated or portable pump, or bailer)	L-2 (SW-846 9060)	Precision-overall	RPD <20% for samples >5X CRDL; ± RL for samples <5X RL	Field duplicate RPDs	S&A
		Precision-lab	RPD <20% for samples >5X CRDL; ± RL for samples <5X RL	Lab duplicate RPDs	A
		Accuracy/bias	Method control limits: ± 25% from expected value	MS/MSD recoveries	S&A
		Accuracy/bias	Method control limits: ± 20% from expected concentration	LCS/LCSD recoveries	A
		Accuracy/bias-contamination	< RL	Method blanks	A
		Accuracy/bias-contamination	No analyte detected at ≥ RL	Equipment blanks	S&A

QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group[1]	MBA
Concentration Level	Low/medium

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP4 (PDB, dedicated or portable pump, or bailer)	L-3 (MBA-830)	Precision-overall	≤ 30% RPD	Field duplicate RPDs	S&A
		Precision-overall	≤ 30% RPD	MS/MSD RPDs	S&A
		Precision-lab	≤ 30% RPD	LCS/LCSD RPDs	A
		Precision-lab	≤ 30% RPD	Lab duplicate RPDs	A
		Precision	≤ 40% RPD	RPD between primary and confirmation columns	A
		Accuracy/bias	Lab control limits: Propionic acid 80-115% All others 90-110%	MS/MSD recoveries	S&A
		Accuracy/bias	Lab control limits: Propionic acid 80-115% All others 90-110%	LCS/LCSD recoveries	A
		Accuracy/bias-contamination	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Method blanks	A
		Accuracy/bias-contamination	<RL	Equipment blanks	S&A

QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group[1]	Dissolved Gases
Concentration Level	Low/medium

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP4 (PDB, dedicated or portable pump, or bailer)	L-4 (RSK-175)	Precision-overall	≤ 30% RPD	Field duplicate RPDs	S&A
		Precision-overall	≤ 30% RPD	MS/MSD RPDs	S&A
		Precision-lab	≤ 30% RPD	LCS/LCSD RPDs	A
		Precision-lab	≤ 30% RPD	Lab duplicate RPDs	A
		Precision	≤ 40% RPD	RPD between primary and confirmation columns	A
		Accuracy/bias	QSM v.5 App. C Table 42 control limits: Carbon dioxide 80-122% Ethane 74-131% Ethene 72-133% Methane 73-125%	MS/MSD recoveries	S&A
		Accuracy/bias	QSM v.5 App. C Table 42 control limits: Carbon dioxide 80-122% Ethane 74-131% Ethene 72-133% Methane 73-125%	LCS/LCSD recoveries	A
		Accuracy/bias-contamination	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Method blanks	A
		Accuracy/bias-contamination	No analytes detected > ½ LOQ or > 1/10 the amount measured in ny sample or 1/10 the regulatory limit, whichever is greater.	Equipment blanks	S&A

QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group[1]	Metals (Cu, Zn)
Concentration Level	Low/medium

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP4 (PDB, dedicated or portable pump, or bailer)	L-5 (SW-846 6010)	Precision-overall	≤ 20% RPD	Field duplicate RPDs	S&A
		Precision-overall	≤ 20% RPD	MS/MSD RPDs	S&A
		Precision-lab	≤ 20% RPD	LCS/LCSD RPDs	A
		Precision-lab	≤ 20% RPD	Lab duplicate RPDs	A
		Accuracy/bias	QSM v.5 App. C Table 4 control limits: Copper 86-114% Zinc 87-115%	MS/MSD recoveries	S&A
		Accuracy/bias	QSM v.5 App. C Table 4 control limits: Copper 86-114% Zinc 87-115%	LCS/LCSD recoveries	A
		Accuracy/bias	Five-fold dilution must agree within ± 10% of the original measurement.	Dilution	S&A
		Accuracy/bias	Recovery within 80-120%.	Post-digestion spike (PDA)	S&A
		Accuracy/bias-contamination	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Method blanks	A
		Accuracy/bias-contamination	No analytes detected > ½ LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Equipment blanks	S&A
		Accuracy/bias-contamination	No analytes detected > LOD.	ICB / CCB	A

QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2) -- Measurement Performance Criteria Table

Matrix	Air (soil vapor)
Analytical Group[1]	VOCs
Concentration Level	Low/medium

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
SOP5 (6-L Summa™ canister)	L-6 (TO-15)	Precision-overall	<25% RPD	Field duplicate RPDs	S&A
		Precision-lab	<25% RPD	LCS/LCSD RPDs	A
		Accuracy/bias	70-130% recovery	Surrogate spike recoveries	S&A
		Accuracy/bias	QSM v.5 App. C Table 43 control limits	LCS/LCSD recoveries	A
		Accuracy/bias-contamination	No analyte detected at ≥1/2 RL or > 10% sample concentration or regulatory limit	Method blanks	A

[1]If information varies within an analytical group, separate by individual analyte.
[2]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[3]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #13 (UFP-QAPP Manual Section 2.7) – Secondary Data Criteria and Limitations Table

Secondary Data Source (Originating Organization, Report Title, and Date)	Data Types	How Data Will Be Used
U.S. Army Environmental Hygiene Agency, *Geohydrologic Study, Defense Depot Memphis Tennessee*. January 1983.	Site history, soil borings and sampling activities.	Background information, cross-sections.
O.H. Materials Company. *Summary Report, On-Site Remedial Activities at the Defense Depot Memphis*. February 1986.	Remedial action summary and confirmation sampling	Background information.
A.T. Kearney, Inc. *RCRA Facilities Assessment Report*. 1990	Environmental site descriptions	Background information.
Law Environmental. *Defense Depot Memphis Tennessee Remedial Investigation Final Report*. August 1990.	Site history, soil borings and sampling activities.	Background information, cross-sections.
Woodward-Clyde. *Environmental Baseline Survey Report, Defense Depot Memphis Tennessee*. November 1996.	Site history, soil borings and sampling activities.	Background information, cross-sections.
OHM/IT Remediation Services, Inc. *Post Removal Report: Contaminated Soil Remediation Family Housing Area, Memphis Depot, Tennessee, Volumes I and II.* March 1999.	Remedial action summary and confirmation sampling	Background information.
CH2M HILL. *Memphis Depot Main Installation Remedial Investigation Report - Volumes I through IV.* January 2000.	Site history, soil borings and sampling activities.	Background information, cross-sections.
CH2M HILL. *Memphis Depot – Main Installation Groundwater Feasibility Study Report - Final*. July 2000.	Groundwater modeling and attenuation study	Background information.
Jacobs-Sverdrup Inc. *Remediation Report, Removal Action in Parcels 35 and 28 (Old Paint Shop and Maintenance Area), Former Defense Distribution Depot, Memphis.* September 2000.	Remedial action summary and confirmation sampling	Background information.
Jacobs Engineering Group. *Decontamination Report and Certification for Closure of Permitted Container Storage Facility (Building T-308).* November 2001.	Remedial action summary and confirmation sampling	Background information.
UXB International. *Final Chemical Warfare Material Investigations/ Removal Report*. 2001.	Dunn Field CWM investigation and removal	Background information.
CH2M HILL. *Memphis Depot Dunn Field Remedial Investigation Report* - Volumes I through III. July 2002.	Site history, soil borings and sampling activities.	Background information, cross-sections.
Jacobs Federal Programs. *Remediation Report, Removal Action at Building 949, Former Defense Distribution Depot, Memphis*. February 2002	Remedial action summary and confirmation sampling	Background information.

MACTEC. *Early Implementation of Selected Remedy Interim Remedial Action Completion Report, Revision 1.* September 2005	Remedial action and monitoring results in Off Depot area	Background information.
HDR|e²M. *Main Installation Source Area Evaluation, Revision 0*. March, 2008.	Summary of site history and sampling activities.	Background information.
HDR|e²M. *Main Installation Source Area Investigation, Revision 0*. February, 2009.	Site assessment and sampling activities.	Background information.

QAPP Worksheet #14 (UFP-QAPP Manual Section 2.8.1) – Summary of Project Tasks

Remedial Action Tasks:

Enhanced Bioremediation Treatment
- Mix sodium lactate solution for injection in designated wells; use water quality data to adjust lactate volume
- Inject solution measuring total volume injected and average flow rate at each well

AS/SVE Operations
- Make periodic inspection of system operations and perform maintenance per schedule and observations
- Record system operational data and collect PID and vacuum measurements at SVE wells, system effluent and vapor monitoring points
- Record air flow rates for sparge wells and system

Sampling Tasks:

Groundwater Sampling for LTM
- Well assessments and water level measurement sweep prior to sampling
- Collect samples using passive diffusion bags (PDBs) at most wells; use low-flow sampling/ bailers where necessary
- Collect water quality measurements for well stabilization when using low flow sampling/ bailers

Groundwater Sampling for EBT
- Perform well assessment and collect water level prior to sampling at each well
- Collect samples using low-flow sampling or bailers where recharge is not sufficient
- Collect water quality measurements for well stabilization prior to sampling

AS/SVE Vapor Sampling
- Take PID measurements prior to sample; collect vapor sample in Summa canister

Analysis Tasks:

Analyze LTM groundwater and vapor samples for volatile organic compounds (VOCs).
Analyze EBT groundwater samples for VOCs, total organic carbon, dissolved gases and metabolic fatty acids

Quality Control Tasks:

Follow SOPs for sample collection, sample packaging and shipping, and analysis.

Secondary Data:

Not applicable.

Data Management Tasks:

Analytical data will be added to DDMT database after validation.

Documentation and Records:

All sample locations have GPS locations documented, field measurements and sample data noted in field records and maintained in project files.

Sample results and data validation presented in annual reports.

Data Packages:

Microbac and CT Laboratories LLC (CTL) to 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).

Assessment/Audit Tasks:

Field sampling procedures reviewed in annual audits by QA officer.

Annual laboratory audits performed through NELAP.

Data Review Tasks:

Laboratories will verify that all data are complete for samples received. All data package deliverables requirements will be met. Data will be reviewed by Diane Short & Associates 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. Final validation qualifiers will be applied as described in section 4.2.2 and detailed in Table 7. Achievement of all project-specific measurement performance criteria (MPC) specified in the QAPP and data validation criteria (DVC) 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 all 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 and required Action Limits. Corrective action is initiated, as necessary. Final data are placed in a database, with any necessary qualifiers, and tables, charts, and graphs are generated.

QAPP Worksheet #15 (UFP-QAPP Manual Section 2.8.1) -- Reference Limits and Evaluation Table

Matrix: Groundwater
Analytical Group: VOCs
Concentration Level: Low/medium

Analyte	CAS Number	Project Action Level (MCL or TC) (µg/L)	Project Quantitation Limit Goal (µg/L)	Achievable Laboratory Limits[2] MDLs (µg/L)	Achievable Laboratory Limits[2] RLs (µg/L)
1,1,2,2-Tetrachloroethane	79-34-5	2.2	1	0.2	0.5
1,1,2-Trichloroethane	79-00-5	1.9	1	0.25	1
1,1-Dichloroethene	75-35-4	7	1	0.5	1
1,2-Dichloroethane	107-06-2	5	1	0.25	0.5
Carbon tetrachloride	56-23-5	3	1	0.25	1
Chloroform	67-66-3	12	1	0.125	0.3
Tetrachloroethene	127-18-4	2.5	1	0.25	1
Trichloroethene	79-01-6	5	1	0.25	1
Vinyl chloride	75-01-4	2	1	0.25	1
cis-1,2-Dichloroethene	156-59-2	35	1	0.25	1
trans-1,2-Dichloroethene	156-60-5	50	1	0.25	1

[1]Analytical MDLs and QLs are those documented in validated methods.

[2]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

Action Levels for water are the lowest of the USEPA Maximum Contaminant Levels (2014) or the Target Concentrations from Dunn Field Record of Decision (March 2004).

Matrix: Groundwater
Analytical Group: TOC
Concentration Level: Low/medium

Analyte	CAS Number	Screening Level (MCL or PRG) (mg/L)	Project Quantitation Limit Goal (mg/L)	Achievable Laboratory Limits[2] MDLs (mg/L)	Achievable Laboratory Limits[2] RLs (mg/L)
TOC	7440-44-0		1	0.5	1.0

Matrix: Groundwater
Analytical Group: MBAs
Concentration Level: Low/medium

Analyte	CAS Number	Screening Level (MCL or PRG) (mg/L)	Project Quantitation Limit Goal (mg/L)	Achievable Laboratory Limits[2] MDLs (mg/L)	Achievable Laboratory Limits[2] RLs (mg/L)
Acetic acid	64-19-7	-	1	0.5	1.0
Butyric acid	107-92-6	-	1	0.5	1.0
Lactic acid	50-21-5	-	1	0.5	1.0
Propionic acid	79-09-4	-	10	5	10
Pyruvic acid	127-17-3	-	1	0.05	0.1

Matrix: Groundwater
Analytical Group: Dissolved Gases
Concentration Level: Low/medium

Analyte	CAS Number	Screening Level (MCL or PRG) (µg/L)	Project Quantitation Limit Goal (µg/L)	Achievable Laboratory Limits[2] MDLs (µg/L)	Achievable Laboratory Limits[2] RLs (µg/L)
Carbon dioxide	124-38-9	-	10,000	2,500	10,000
Ethane	74-84-0	-	5	1	5
Ethene	74-85-1	-	5	1	5
Methane	74-82-8	-	5	1	5

Matrix: Groundwater
Analytical Group: Metals
Concentration Level: Low/medium

Analyte	CAS Number	Screening Level (MCL or PRG) (mg/L)	Project Quantitation Limit Goal (mg/L)	Achievable Laboratory Limits[2] MDLs (mg/L)	Achievable Laboratory Limits[2] RLs (mg/L)
Copper	7440-50-8	0.032	0.02	0.005	0.02
Zinc	7440-66-6	0.521	0.02	0.005	0.02

Matrix: Air (soil vapor)
Analytical Group: VOCs
Concentration Level: Low/medium

Analyte	CAS Number	Project Action Limit (Vapor RG) (ppbv)	Project Quantitation Limit Goal (ppbv)	Analytical Method[1] MDLs (ppbv)	Analytical Method[1] Method QLs (ppbv)	Achievable Laboratory Limits[2] MDLs (ppbv)	Achievable Laboratory Limits[2] RDLs (ppbv)
1,1,2,2-Tetrachloroethane	79-34-5	0.55	0.4	0.28		0.023	0.15
1,1,2-Trichloroethane	79-00-5	2.03	0.5	0.50		0.029	0.18
1,1-Dichloroethene	75-35-4	29.03	0.5			0.043	0.25
1,2-Dichloroethane	107-06-2	0.64	0.4	0.24		0.047	0.25
Carbon Tetrachloride	56-23-5	14.22	0.5	0.42		0.025	0.16
Chloroform	67-66-3	32.63	0.5	0.25		0.039	0.20
cis-1,2-Dichloroethene	156-59-2	39.52	0.5			0.045	0.25
Methylene Chloride	75-09-2	2.85	0.5	1.38		0.043	0.29
Tetrachloroethene	127-18-4	0.99	0.5	0.75		0.021	0.15
trans-1,2-Dichloroethene	156-60-5	133.5	0.5			0.043	0.25
Trichloroethene	79-01-6	2.06	0.5	0.45		0.028	0.19
Vinyl Chloride	75-01-4	14.77	0.5	0.33		0.074	0.39

[1]Analytical MDLs and QLs are those documented in validated methods.

[2]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

Action levels are fluvial vapor remediation goals (RGs) from the Dunn Field Record of Decision (March 2004).

QAPP Worksheet #16 (UFP-QAPP Manual Section 2.8.2) - Project Schedule/Timeline Table

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Semiannual LTM	HDR	10/6/14	10/16/14	Samples to laboratory, field reports to file	10/20/14
LTM Sample Analysis	Microbac-CTL	10/13/14	11/13/14	Level 4 report and EDD	11/13/14
Data Validation	DSA	11/17/14	11/28/14	Data narrative report	11/28/14
Report Preparation	HDR	11/17/14	1/8/15	2014 LTM annual report	1/8/15
EBT Monitoring	HDR	11/3/14	11/13/14	Samples to laboratory, field reports to file	11/17/14
EBT Sample Analysis	Microbac-CTL	11/10/14	12/11/14	Level 4 report and EDD	12/11/14
Data Validation	DSA	12/15/14	1/7/15	Data narrative report	1/9/15
Annual Report Preparation	HDR	12/15/14	3/2/15	EBT Y4 annual report	3/6/15
AS/SVE Y5-2	HDR	7/1/14	9/30/14	Vapor sample to laboratory, field reports to file	10/3/14
Effluent Sample Analysis	Microbac-CTL	9/30/14	10/21/14	Level 4 report and EDD	10/21/14
Data Validation	DSA	10/23/14	10/31/14	Data narrative report	10/31/14
Report Preparation	HDR	10/23/14	11/17/14	Quarterly summary report	11/17/14
AS/SVE Y5-3	HDR	7/1/14	9/30/14	Vapor sample to laboratory, field reports to file	10/3/14
Effluent Sample Analysis	Microbac-CTL	9/30/14	10/21/14	Level 4 report and EDD	10/21/14
Data Validation	DSA	10/23/14	10/31/14	Data narrative report	10/31/14
Report Preparation	HDR	10/23/14	11/17/14	Quarterly summary report	11/17/14
AS/SVE Y5-4	HDR	10/1/14	12/31/14	Vapor sample to laboratory, field reports to file	1/6/15
Effluent Sample Analysis	Microbac-CTL	12/30/14	1/20/15	Level 4 report and EDD	1/20/15
Data Validation	DSA	1/22/15	1/30/15	Data narrative report	1/30/15
Annual Report Preparation	HDR	1/22/15	2/27/15	Y5 annual report	3/1/15

QAPP Worksheet #17 (UFP-QAPP Manual Section 3.1.1) - Sampling Design and Rationale

Rationale for sampling approach:

- Groundwater Sampling for LTM is conducted based on the MI LTM Plan in Appendix B of the *Main Installation Final Remedial Design, Rev.1* (MI RD) (CH2M HILL 2004a) and the Dunn Field LTM Plan in Appendix C of *Off Depot Groundwater Final Remedial Design, Revision 1* (Off Depot RD) (CH2M HILL 2008). LTM is conducted to evaluate progress toward remedial action objectives (RAOs).
  - MI LTM includes 99 wells classified as Background (6), Boundary (7), Performance (62) and Sentinel (24); the wells have the following sample frequency: biennial (15), annual (30) and semiannual (54). The MI LTM wells are listed on Table 4 and the locations are shown on Figure 13. Groundwater samples from MI LTM wells are analyzed for VOCs only, with concentrations at low-moderate levels. The highest concentration in the most recent annual or biennial MI sample event (October 2013) was PCE at 235 micrograms per liter (µg/L); eight wells contained individual VOCs at concentrations above 50 µg/L.
  - Dunn Field LTM includes 86 wells classified as Background (8), Background-NE (5), Performance (50), Performance-FSVE (14) or Sentinel (9); the wells have the following sample frequency: semiannual (38), annual (32) or biennial (16). The Dunn Field LTM wells are listed on Table 3 and the locations are shown on Figure 9. Groundwater samples from Dunn Field LTM wells are analyzed for VOCs only, with concentrations at low-moderate levels. The highest concentration in the most recent annual or biennial MI sample event (April-May 2013) was TCE at 181 micrograms per liter (µg/L); no other wells contained individual VOCs at concentrations above 50 µg/L.
  - The LTM well classifications and sample frequencies are evaluated annually and changes are recommended in the annual LTM report.
- EBT activities including performance monitoring are conducted based on the *Remedial Action Work Plan Addendum, Main Installation* (HDR 2012a). Monitoring is conducted to evaluate success in expanding anaerobic conditions and decreasing CVOC concentrations through reductive dechlorination. Overall progress toward RAOs is evaluated through LTM.
  - Quarterly groundwater samples for performance monitoring are collected from 45 injection wells and 13 monitoring wells. The EBT wells are listed on Table 2 and the locations are shown on Figure 8. Quarterly monitoring is performed prior to injections and includes water level measurements, field water quality measurements and groundwater sampling for laboratory analyses.
  - Field water quality parameters are DO, ORP, pH, temperature, and conductivity. Laboratory analyses are made for VOCs, TOC, metabolic fatty acids (MFAs) and dissolved gases. The highest VOC concentrations in the November 2013 sample event were cDCE at 211 µg/L and PCE at 149 µg/L; sixteen wells contained individual VOCs at concentrations above 50 µg/L.
- AS/SVE Vapor Sampling – a vapor sample is collected quarterly *Dunn Field Off Depot Groundwater Remedial Action Work Plan, Revision 2* (e^2M, 2009). AS/SVE system monitoring consists of vacuum measurements at VMPs; PID readings at the system effluent, SVE wells and VMPs; and vapor samples from the system effluent. Vapor samples collected to monitor system performance and to confirm treatment system compliance with permitted discharge limits; samples are analyzed for VOCs. Total VOCs in the December 2013 sample were 53.2 parts per billion, volume (ppbv) and the main constituent was TCE at 21 ppbv.

QAPP Worksheet #18 (UFP-QAPP Manual Section 3.1.1) - Sampling Locations and Methods/SOP Requirements Table

Sampling Location	Number of Locations	Matrix	Depth (feet)	Analytical Group	Concentration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]	Rationale for Sampling Location
MI EBT Injection	45	GW	77-143	VOCs, Dissolved Gases, MFAs, TOC	Low-moderate	58 field, 6 duplicate	SOP 4	Treatment area
MI EBT Performance	13	GW	85-162		Low-moderate		SOP 4	Near downgradient
MI LTM Background	6	GW	58-92	VOCs	Low	101 field, 10 duplicate	SOP 4	Background
MI LTM Boundary	7	GW	92-121		Low		SOP 4	Offsite sources
MI LTM Sentinel	24	GW	108-286		Low-moderate		SOP 4	Vertical migration
MI LTM Performance	64	GW	68-148		Low-moderate		SOP 4	Within plumes
DF LTM Background	8	GW	16-76	VOCs	Low	86 field, 9 duplicate	SOP 4	Background
DF LTM Background-NE	5	GW	57-67		Low-moderate		SOP 4	Offsite Plume
DF LTM Sentinel	9	GW	51-260		Low		SOP 4	Vertical migration
DF LTM Performance	50	GW	45-91		Low-moderate		SOP 4	Within plumes
DF LTM Performance-FSVE	14	GW	51-75		Low		SOP 4	FSVE rebound
AS/SVE Vapor	1	V	35-76	VOCs	Low	1 field	SOP 5	Effluent

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

QAPP Worksheet #19 (UFP-QAPP Manual Section 3.1.1) -- Analytical SOP Requirements Table

Matrix	**Analytical Group**	**Concentration Level**	**Analytical and Preparation Method / SOP Reference**[1]	**Sample Size**	**Containers** (number, size, and type)	**Preservation Requirements** (chemical, temperature, light protected)	**Maximum Holding Time** (preparation / analysis)
Groundwater	VOCs	Low/Medium	SW-846 5030B/8260B (L-1)	5-25 mL	40-mL VOA w/ Teflon®-Lined septum (3) No headspace	<6°C; HCl to pH<2	14 days if preserved or 7 days if unpreserved
Groundwater	TOC	Low/medium	EPA 415.1 / SW846 9060A / SM5310C 2000 (L-2)	40 mL	250-mL glass bottle	<6°C; H_2SO_4 to pH<2	28 days
Groundwater	Metabolic Acids (MBA)	Low/medium	Metabolic Acids Method 830-MBA (L-3)	400 µL	250-mL amber glass bottle	<6° C; 5 mL of 5% H_3PO_4 for pH < 2.	28 days
Groundwater	Dissolved Gases	Low/medium	EPA RSK-175 (L-4)	20 mL	Pre-cleaned 20-mL glass headspace screw top vials with Teflon-faced silicon septa	<6° C; HCl to pH ≤ 2	14 days if preserved (pH ≤ 2) or 7 days if unpreserved (pH >2)
IDW Water	VOCs	Low/medium	SW-846 5030B/8260B (L-1)	5-25 mL	40-mL VOA w/ Teflon®-Lined septum (3) No headspace	<6°C; HCl to pH<2	14 days if preserved or 7 days if unpreserved
IDW Water	Metals	Low/medium	SW-846 6010 (L-5)	50 mL	250-1000 mL plastic bottle	HNO_3 to pH < 2	180 days
Air (soil vapor)	VOCs	Low/Medium	TO-15 (L-6)	100 mL	6-liter Summa™ canister (1)	None	30 days

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

QAPP Worksheet #20 (UFP-QAPP Manual Section 3.1.1) -- Field Quality Control Sample Summary Table

Summarize by matrix, analytical group, and concentration level the number of field QC samples that will be collected and sent to the laboratory.

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations[2]	No. of Field Duplicate Pairs	No. of MS/MSD sets	No. of Field Blanks	No. of Equip. Blanks	No. of PT Samples	Total No. of Samples to Lab
Groundwater	VOCs	Low/medium	SW-846 5030B/8260B (L-1)	Per SAP	10%	5%	1 Trip Blank per day; Ambient Blanks as needed	1 per day as appropriate	None	Per SAP
Groundwater	TOC	Low/medium	SW-846 9060 Mod (L-2)	Per SAP	10%	5%	Ambient Blanks as needed	1 per day as appropriate	None	Per SAP
Groundwater	Metabolic Acids (MBA)	Low/medium	830-MBA (L-3)	Per SAP	10%	5%	Ambient Blanks as needed	1 per day as appropriate	None	Per SAP
Groundwater	Dissolved Gases	Low/medium	RSK-175 (L-4)	Per SAP	10%	5%	Ambient Blanks as needed	1 per day as appropriate	None	Per SAP
IDW Water	VOCs	Low	SW-846 5030B/8260B (L-1)	One (IDW collection site)	None	None	None	None	None	1 per year
IDW Water	Metals	Low	SW-846 6010B (L-5)	One (IDW collection site)	None	None	None	None	None	1 per year
Air (soil vapor)	VOCs	Low	TO-15 (L-6)	Per SAP	10%	None	None	None	None	Per SAP

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).
[2]If samples will be collected at different depths at the same location, count each discrete sampling depth as a separate sampling location or station.

QAPP Worksheet #21 (UFP-QAPP Manual Section 3.1.2) -- Project Sampling SOP References Table

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
SOP1	General Procedures for Field Personnel	HDR	Log books, first aid kit, PPE	N	
SOP2	Drilling and Soil Sampling	HDR	Drilling equipment, sample jars, disposable scoops	N	
SOP3	Well Installation, Development and Abandonment	HDR	Well construction materials, cement/bentonite grout, concrete	N	
SOP4	Groundwater Sample Collection	HDR	YSI6920 or similar multi-probe device with flow-through cell, non-dedicated bladder pumps, disposable Teflon bailers, passive diffusion bags	N	
SOP5	Vapor Sample Collection	HDR	Summa canisters, flow controllers	N	
SOP6	EBT Mixing and Injections	HDR	EBT solution, injection equipment	N	
SOP7	Sample Control and Documentation	HDR	Sampling log book, DQCR forms, digital camera, chain of custody forms	N	
SOP8	Sample Packing and Shipping	HDR	Sample bottles, bubble wrap, ice, zip lock bags, coolers, tape, custody seals	N	
SOP9	Sampling Equipment Decontamination	HDR	ASTM Type II water (supplied by lab) or distilled water, pesticide-grade methanol, Alconox detergent, brushes	N	
L-8	Standard Operating Procedure Sample Receiving and Login, Revision 14, 12 November 2013 (LOGIN01)	Microbac	Thermometers, hood, pH strips, IR temperature guns, disposable pipets, Geiger counter, disposable gloves, PDA, laptop or notebook computer equipped for bar code reading	N	Water samples
L-10	Sample Receiving, Acceptance and Log-In (SMO-SMPL_REC)	ALS	COCs, scanner with PDF function, computer with LIMS software	N	VOCs in air

QAPP Worksheet #22 (UFP-QAPP Manual Section 3.1.2.4) -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maint. Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference[1]
YSI 650MDS or similar	pH, ORP, DO, conductivity, temperature		pH, ORP, DO, conductivity, temperature	pH, ORP, DO, conductivity, temperature	Daily during sampling event			Sampler	SOP4
Horiba U-2000	pH, ORP, DO, conductivity, temperature, turbidity		pH, ORP, DO, conductivity, temperature, turbidity	pH, ORP, DO, conductivity, temperature, turbidity	Daily during sampling event			Sampler	SOP4
Lamotte 2020e	Turbidity		Turbidity	Turbidity	Daily during sampling event			Sampler	SOP4
RAE Systems PGM-7600	PID		PID	PID	Daily during sampling event			Sampler	SOP4
Heron Dipper-T			Water level	Water level	Daily during sampling event			Sampler	SOP4
Solinst 101			Water level	Water level	Daily during sampling event			Sampler	SOP4
Geotech PRO pumpbox			Pumpbox					Sampler	SOP4
Geotech Geocontrol 2			Pump controller box					Sampler	SOP4
Thomas TG-180HST			Air Compressor					Sampler	SOP5
Thomas 107CDC20			Air Pump					Sampler	SOP5

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

QAPP Worksheet #23 (UFP-QAPP Manual Section 3.2.1) -- Analytical SOP References Table

Reference Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
SOP4	Groundwater Sampling	Screening	pH	Field multimeter (YSI)	HDR	N
SOP4	Groundwater Sampling	Screening	Conductivity	Field multimeter (YSI)	HDR	N
SOP4	Groundwater Sampling	Screening	Dissolved oxygen (DO)	Field multimeter (YSI)	HDR	N
SOP4	Groundwater Sampling	Screening	Oxidation-reduction potential (ORP)	Field multimeter (YSI)	HDR	N
SOP4	Groundwater Sampling	Screening	Temperature	Field multimeter (YSI)	HDR	N
SOP4	Groundwater Sampling	Screening	Turbidity	Field multimeter (YSI)	HDR	N
L-1	Analysis of Volatile Organic Analytes by Methods 8260A and 8260B, Revision 14 September 2010 (MSV01)	Definitive	VOCs in Water	Hewlett-Packard [HP] 6890 GC equipped with HP 5973 mass spectrometer HP Enviroquant software	Microbac	N
L-2	Organic Carbon, Total (Oxidation), EPA 415.1 / SW846 9060A / SM5310C-2000 (2011 Editorial Revision), 20 March 2014 (K4151)	Definitive	TOC in Water	Shimadzu TOC-VWP with Autosampler Computer, and printer Software version 1.06.00	Microbac	N

Reference Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
L-3	Organic Analysis of Metabolic Acids, Method 830MBA, 23 July 2014 (HPLC03)	Definitive	Metabolic Fatty Acids in Water	Hewlett Packard 1050 HPLC System or equivalent HPLC pump Autosampler unit UV Detector Vacuum Degasser HP3365 Series 2 Chem Station; Supelco Supelcogel H Column, 25 cm x 4.6 mm ID	Microbac	N
L-4	Analysis of Dissolved Gases in Groundwater, EPA RSKSOP-175, 26 June 2014 (RSK01)	Definitive	Dissolved Gases in Water	Agilent 6890N GC equipped with an Agilent FID/TCD Teledyne Tekmar HT3 Headspace Autosampler Varian Archon autosampler for sample preparation (i.e.: create headspace) Front column with FID: Restek Rt-Q BOND column 30 m .53 mm ID Back column with TCD: Restek Rt-Q BOND column 15 m .53 mm ID 20 mL glass headspace screw top vials with Teflon-lined screw caps (SUN-SRi) Enviroquant Chemstation C.00.00	Microbac	N

Reference Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
L-5	Perkin Elmer Optima 4300 Inductively Coupled Plasma Atomic Emission Spectroscopy SW846 6010 / EPA 200.7, 15 April 2014 (ME600E)	Definitive	Metals in Water	Perkin Elmer Optima 4300 equipped with a CETAC ASXpress-520 Autosampler Argon gas supply (liquid) Dell Pentium 4 computer with Microsoft Windows 2000 Professional and Perkin Elmer WinLab32 ICP Continuous Software Version 4.0.0.0303 ESI Microflow PFA-ST3-84 Nebulizer	Microbac	N
L-6	Standard Operating Procedure for Determination of Volatile Organic Compounds in air Samples Collected in Specially Prepared Canisters and Gas Collection Bags by Gas Chromatography/Mass Spectrometry (GC/MS), 22 December 2010 (VOA-TO15 _Rev.18)	Definitive	VOCs in Air	GC: HP 5890 Series II Plus, HP 6890 Series, HP 6890A Series, Agilent 6890N Series MS: HP 5972 Series, HP 5973 Series, Agilent 5973, Agilent 5973N, Agilent 5973*inert*, Agilent 5975B*inert*, Agilent 5975C*inert* Data System: IBM-compatible PC with Windows 95/98/NT/XP (Microsoft Office EXCEL version 2003 or newer) and Hewlett Packard Chemstation software including EnviroQuant with Extracted Ion Current Profile (EICP), National Institute of Standards and Technology (NIST) library (2002 version or newer) or equivalent	ALS	N

Reference Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
L-7	Microwave Digestion – Aqueous, SW846 3015A, 15 December 2013 (ME407)	Definitive	Metals in Water	Mars Xpress microwave unit, must provide programmable power with a minimum of 574W and can be programmed to within ± 10W of required power Beckman GS-6 or equivalent centrifuge Analytical balance, 600 g capacity	Microbac	N
L-8	Sample Receiving and Login, 12 November 2013 (LOGIN01)	Not Applicable	All	PDA, Laptop or notebook computer (equipped for bar coding) IR Temperature Guns Thermometers pH paper	Microbac	N

QAPP Worksheet #24 (UFP-QAPP Manual Section 3.2.2) -- Analytical Instrument Calibration Table

Identify all analytical instrumentation that requires calibration and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/MS for VOCs in Water SW-846 8260B	Mass spectral ion intensities with 4-bromofluoro-benzene (BFB)	Every 12 hours prior to ICAL, ICV or CCV	Mass – Ion Abundance Criteria 50 – 15-40% of mass 95 75 – 30-60% of mass 95 95 – base peak, 100% relative abundance 96 – 5-9% of mass 95 173 – <2% of mass 174 174 – >50% of mass 95 175 – 5-9% of mass 174 176 - >95% and <101% of mass 174 177 - 5-9% of mass 176 (Established criteria in Table 4 of SW-846 8260B)	Retune instrument and repeat BFB check. Flagging criteria are not appropriate.	Analyst	L-1
	Initial multipoint calibration for all analytes (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 SPCCs: ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. RSD for RFs for CCCs: ≤ 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 (COD) r2 ≥ 0.99 (6 points shall be used for second order, 7 points shall be used for third order).	Evaluate cause; repeat calibration; or qualify data and discuss in narrative. See SOP section 13.7 for additional corrective action. Flagging criteria are not appropriate.	Analyst	L-1

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC/MS for VOCs in Water SW-846 8260B (continued)	Continuing Calibration verification (CCV)	Once per each 12 hours, prior to sample analysis (criteria for these checks must be met prior to sample analysis)	1. Average RRF for SPCCs : ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. %Difference/Drift for all target compounds and surrogates: VOCs and SVOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	1) Evaluate system and take corrective action. 2) Rerun calibration check. 3) If still out, prepare new calibration curve for any analyte not meeting criteria. 4) Reinject any samples analyzed after criteria were exceeded. 5) Qualify the data. Criteria for these checks must be met prior to sample analysis. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Analyst	L-1
	Retention time window position establishment for each analyte and surrogate	Once per ICAL	Position shall be set using the midpoint standard of the initial calibration curve.	NA	Analyst	L-1
TOC analyzer for TOC in water SW-846 9060	Initial Calibration (minimum blank + 5 points) (ICAL)	Daily	r ≥ 0.995	1. Terminate analysis 2. Recalibrate and verify before sample analysis	Analyst	L-2
	Initial Calibration Verification (ICV) (Separate source from ICAL standards)	Daily, prior to sample analysis, immediately following ICAL	± 10% from expected concentration	1. Reprep ICV and, reanalyze all associated samples 2. Identify and document problem 3. Recalibrate and reanalyze reprepped ICV and all associated samples, if necessary	Analyst	L-2

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
TOC analyzer for TOC in water SW-846 9060 (cont'd.)	Continuing Calibration Verification (CCV)	Before sample analysis; after every 10 samples and end of run	± 10% from expected concentration	1. Recalibrate and verify 2. Reanalyze samples back to last good CCV	Analyst	L-2
	CRDL Verification Standard (< 2X CRDL)	After initial CCV	± 20% from expected concentration	1. Reprep and reanalyze standard 2. Recalibrate and verify	Analyst	L-2
HPLC for MBA in Water MBA830	Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression for each analyte: r2 ≥ 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99.	Correct problem then repeat ICAL. No samples shall be analyzed until ICAL has passed. Minimum 5 levels for linear and 6 levels for quadratic.	Analyst	L-3
	Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used. Calculated for each analyte and surrogate.	NA	Analyst	L-3
	Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study. Calculated for each analyte and surrogate.	NA	Analyst	L-3

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
HPLC for MBA in Water MBA830 (cont'd.)	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 15% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL. No samples shall be analyzed until calibration has been verified with a second source.	Analyst	L-3
	Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ± 15% true value. Retention time windows are updated per the method.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.	Analyst	L-3

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC for Dissolved Gases in Water RSK-175	Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	ICAL must meet one of the three options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression for each analyte: r2 ≥ 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99. Minimum 5 levels for linear and 6 levels for quadratic. Results may not be quantitated using a single point. No samples shall be analyzed until ICAL has passed.	Correct problem then repeat ICAL.	Analyst	L-4
	Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used. Calculated for each analyte and surrogate.	NA	Analyst	L-4
	Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study. Calculated for each analyte and surrogate.	NA	Analyst	L-4
	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 20% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL. No samples shall be analyzed until calibration has been verified with a second source.	Analyst	L-4

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
GC for Dissolved Gases in Water RSK-175 (cont'd.)	Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ± 20% of true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; -or- Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Results may not be reported without a valid CCV.	Analyst	L-4
ICP for Metals in Water SW-846 6010	Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r2 ≥ 0.99. Minimum one high standard and a calibration blank.	Correct problem, then repeat ICAL. No samples shall be analyzed until ICAL has passed.	Analyst	L-5
	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL. No samples shall be analyzed until calibration has been verified with a second source.	Analyst	L-5
	Low-level Calibration Check Standard (Low-level ICV)	Daily.	All reported analytes within ± 20% of true value. Low level calibration check standard should be less than or equal to the LOQ.	Correct problem and repeat ICAL. No samples shall be analyzed without a valid low-level calibration check standard (LLICV).	Analyst	L-5

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
ICP for Metals in Water SW-846 6010 (cont'd.)	Continuing Calibration Verification (CCV)	After every 10 field samples, and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; -or- Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV. Results may not be reported without a valid CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.	Analyst	L-5
GC/MS for VOCs in Air TO-15	Mass spectral ion intensities with 4-bromofluoro-benzene (BFB)	Every 24 hours prior to ICAL, ICV or CCV	<u>Mass – Ion Abundance Criteria</u> 50 – 8-40% of mass 95 75 – 30-66% of mass 95 95 – base peak, 100% relative abundance 96 – 5-9% of mass 95 173 – <2% of mass 174 174 – 50-120% of mass 95 175 – 4-9% of mass 174 176 – 93-101% of mass 174 177 - 5-9% of mass 176 (Established criteria in Table 3 of TO-15)	Retune instrument and repeat BFB check. Flagging criteria are not appropriate.	Analyst	L-6

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

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

QAPP Worksheet #25 (UFP-QAPP Manual Section 3.2.3) -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Identify all analytical instrumentation that requires maintenance, testing, or inspection and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
GC/MS for VOCs in Water	Clean mass spectrometer, change/check helium, change trap, clip column, change purge and trap ferrules, bake out column	GC/MS	Purge lines, purge flow, trap, ion source, column	Prior to calibration check and/or as necessary	Acceptable ICAL or CCV	Correct problem and repeat ICAL or CCV	Analyst / Supervisor	MSV01 (L-1)
TOC analyzer	Verify dilution water is sufficient; Verify sufficient reagents for analyses ; Verify drain vessel is full; Verify waste container is not full; Verify there is sufficient gas; Check for leaks.	TOC Analyzer	Check all six noted items	Daily	Acceptable performance	Fill or empty vessels as needed	Analyst	K4151 (L-2)
	Replace CO2 absorber; Replace halogen scrubber; Replace syringe plunger tip; Wash the TC reactor; Wash the IC reactor		Check noted items	As required	Acceptable performance	Replace or wash as needed	Analyst	

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
HPLC for Metabolic Acids in Water	Monitor system pressures daily.	HPLC	System pressures are monitored daily to indicate any changes in the HPLC which can include leaks, restrictions, temperature and solvent fluctuations, and gas bubbles.	Daily	Acceptable performance	Fix issues as necessary.	Analyst	HPLC03 (L-3)
	Other preventive maintenance indicated for each HPLC module.		Note decreased performance	As required	Acceptable performance	Follow the vendor's instruction manual for other preventive maintenance indicated for each HPLC module.	Analyst	
GC for Dissolved Gases in Water	Gas pressures are checked daily and other maintenance (e.g. clip column, injector port maintenance, clean detector) performed as needed.	GC	Gas pressures are checked daily and other maintenance (e.g. clip column, injector port maintenance, clean detector) performed as needed.	Daily/as required	Acceptable performance	Fix issues as necessary.	Analyst	RSK01 (L-4)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
	Trouble-shooting involves, but is not limited to, monitoring chromatography, contamination, standards recoveries, injection port maintenance, and pressurized checks.		Trouble-shooting involves, but is not limited to, monitoring chromatography, contamination, standards recoveries, injection port maintenance, and pressurized checks.	As required	Acceptable performance	Fix issues as necessary.	Analyst	
ICP for metals in water	Clean torch and nebulizer when needed.	ICP	Check torch and nebulizer every day.	Daily	Acceptable instrument performance	Fix issues as necessary.	Analyst	ME600E (L-5)
	Change tubing.		Inspect tubing	When it loses pliability and is worn.	Acceptable performance	Fix issues as necessary.	Analyst	
	Drain compressor daily.		Drain compressor daily.	Daily	Acceptable performance	Fix issues as necessary.	Analyst	
	The instruments are under service contracts so that every year a service representative will perform a systems check.		The instruments are under service contracts so that every year a service representative will perform a systems check.	Yearly	Acceptable performance	Fix issues as necessary.	Service represent-ative	
	The water in the recirculator/cooler must be changed yearly.		The water in the recirculator/cooler must be changed yearly.	Yearly	Acceptable performance	Fix issues as necessary.	Analyst	

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

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

QAPP Worksheet #26 (UFP-QAPP Manual Appendix A) -- Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): HDR FTL and field team
Sample Packaging (Personnel/Organization): HDR FTL and field team
Coordination of Shipment (Personnel/Organization): HDR FTL, HDR PC, Laboratory (Microbac, ALS) PM
Type of Shipment/Carrier: Cooler (groundwater) or cardboard carton (air samples in Summa™ canisters), shipped via Federal Express (FedEx), next day delivery
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Laboratory (Microbac, ALS) sample custodian
Sample Custody and Storage (Personnel/Organization): Laboratory (Microbac, ALS) sample custodian
Sample Preparation (Personnel/Organization): Laboratory (Microbac, ALS) sample preparation chemist or analyst
Sample Determinative Analysis (Personnel/Organization): Laboratory (Microbac, ALS) sample preparation chemist or analyst
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 60 days from data package report
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 60 days from data package report
Biological Sample Storage (No. of days from sample collection): Not Applicable
SAMPLE DISPOSAL
Personnel/Organization: Laboratory (Microbac, ALS) sample custodian
Number of Days from Analysis: 60 days from data package report

QAPP Worksheet #27 (UFP-QAPP Manual Section 3.3.3) -- Sample Custody Requirements

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory):
Field sample custody procedures are described in detail in the HDR sampling SOP number SOP7, *Sample Control and Documentation*.

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal):
Laboratory sample custody procedures are described in detail in the Microbac SOP number L-8, *Standard Operating Procedure Sample Receiving and Login, Revision 14, 12 November 2013 (LOGIN01)* and the ALS SOP number L-10, *Sample Receiving, Acceptance and Log-In (SMO-SMPL_REC)*.

Sample Identification Procedures:
Field sample ID procedures are described in detail in the HDR sampling SOP numbers SOP1, *General Procedures for Field Personnel*; SOP4, *Groundwater Sample Collection*; SOP5, *Vapor Sample Collection*; and SOP7, *Sample Control and Documentation* and the appropriate Sampling and Analysis Plan (SAP).

Chain-of-custody Procedures:
Field chain of custody procedures are described in detail in the HDR field SOP numbers SOP7, *Sample Control and Documentation*; and SOP8, *Sample Packing and Shipping*.

Laboratory chain of custody procedures are described in detail in the Microbac SOP number L-8, *Standard Operating Procedure Sample Receiving and Login, Revision 14, 12 November 2013 (LOGIN01)* and the ALS SOP number L-10, *Sample Receiving, Acceptance and Log-In (SMO-SMPL_REC)*.

QAPP Worksheet #28 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Matrix	Groundwater
Analytical Group	VOCs by GC/MS
Concentration Level	Low/medium
Sampling SOP	SOP4
Analytical Method / SOP Reference	SW-846 8260B / L-1
Sampler's Name	HDR
Field Sampling Organization	HDR
Analytical Organization	Microbac
Number of Sample Locations	Per SAP

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	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 initial calibration.	Analyst	Accuracy/ Bias	Compounds within ± 20% expected value. Flagging criteria are not appropriate.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Evaluation of relative retention times (RRT)	Each sample.	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of ICAL	Correct problem then reanalyze all samples analyzed since the last retention time check. Lab may update retention times 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 corrective actions as required by the method and rerun the ICAL to reestablish the retention times.	Analyst	--	Relative retention time (RRT) of the analyte within ± 0.06 RRT units of ICAL. Flagging criteria are not appropriate.
Internal Standards (IS) – Retention Time (RT) and area response checked from daily calibration check	Every field sample, standard, and QC sample.	RT ± 30 seconds and EICP area within -50% to +100% of *the mid-point standard in the initial calibration* for each IS compound.	1) Inspect MS and GC for malfunctions. 2) Take appropriate corrective actions. 3) Reanalyze samples analyzed while system was malfunctioning. 4) If sample exceeds criteria, reanalyze sample. If still out, report both analyses and document corrective action.	Analyst	Accuracy	RT ± 30 seconds and EICP area within -50% to +100% of *the mid-point standard in the initial calibration* for each IS compound. Apply Q-flag to analytes associated with the non-compliant IS.
Demonstrate ability to generate acceptable accuracy and precision using four replicate analyses of a second source QC check sample	Once per analyst	Analyte-specific limits as per laboratory historical limits	1) Recalculate results. 2) Locate and fix the source of the problem. 3) Rerun demonstration for those analytes that did not meet criteria.	Analyst	Precision, Accuracy	Analyte-specific limits as per laboratory historical limits

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MDL Study	Once per year, upon any major system change, or quarterly MDL check.	Method detection limits established as described in 40 CFR Part 136, App. B shall not exceed one-half the RL	Method detection limits that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples.	Analyst	Sensitivity	Method detection limits 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%	1) Review lab QC data to determine if they are in control. 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action.	Analyst	Precision-overall	RPD ≤ 30%
MS/MSD	Sampling: 1 pair per every 20 samples Lab: same	In-house control limits (or, if not established, control limits in DoD QSM 4.2 [10/25/2010] 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 4.2 [10/25/2010] Table G-4. RPD ≤ 30 % For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.
Laboratory Control Sample (LCS)	One per preparatory batch	In-house control limits (or, if not established, control limits in DoD QSM 4.2 [10/25/2010] Table G-4. Up to 3 marginal exceedances if full list of 66 analytes is run.	1) The analytical batch must be reprocessed. 2) Reprep and analyze LCS and affected samples. 3) Qualify the data if corrective action was unsuccessful or was not performed	Analyst	Precision-lab	In-house control limits (or, if not established, control limits in DoD QSM 4.2 [10/25/2010] 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.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Surrogate spike recoveries	Every sample, spike, standard, and reagent blank	In-house control limits (or, if not established, control limits in DoD QSM 4.2 [10/25/2010] Table G-3.	1) Recalculate result, and reanalyze sample if still out. 2) Re-extract and reanalyze sample, if still out. 3) Report both analyses and document in report that steps 1 and 2 were performed. 4) Qualify the data.	Analyst	Accuracy/ bias	In-house control limits (or, if not established, control limits in DoD QSM 4.2 [10/25/2010] Table G-3. Apply Q-flag to all associated analytes if acceptance criteria are not met.
Method blanks	One per preparatory batch	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL.	1) Take and document appropriate corrective action 2) Reanalyze all samples processed with a contaminated blank. 3) Qualify the data if the corrective action was not successful or was not performed.	Analyst	Accuracy/ bias-contamin-ation	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.
Trip blank	Sampling: 1 for each batch of samples shipped to laboratory Lab: NA	No analytes detected at > RL	1) Review lab QC data to determine if there is a laboratory problem. 2) If same compounds are found in field samples at similar concentrations, qualify the data. OR 3) Resample the batch	Analyst	Accuracy/ bias-contamin-ation	No analytes detected at > RL Apply B-flag to all results for the specific analyte(s) in all samples associated with the trip blank.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Ambient blank	Sampling: Collected when samples are collected downwind of possible volatile sources. Lab: NA	No analytes detected at > RL	1) Review lab QC data to determine if there is a laboratory problem. 2) If same compounds are found in field samples at similar concentrations, qualify the data. OR 3) Resample the batch.	Analyst	Accuracy/ bias-contamin-ation	No analytes detected at > RL Apply B-flag to all results for the specific analyte(s) in all samples associated with the ambient blank.
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 #28 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Matrix	Groundwater
Analytical Group	TOC
Concentration Level	Low/medium
Sampling SOP	SOP4
Analytical Method / SOP Reference	EPA 415.1 / SW846 9060A / SM5310C 2000 / L-2
Sampler's Name	HDR
Field Sampling Organization	HDR
Analytical Organization	Microbac
Number of Sample Locations	Per SAP

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration Verification (ICV) (Separate source from ICAL standards)	Daily, prior to sample analysis, immediately following ICAL	± 10% from expected concentration	1. Reprep ICV and, reanalyze all associated samples 2. Identify and document problem 3. Recalibrate and reanalyze reprepped ICV and all associated samples, if necessary	Analyst	Accuracy/ Bias	Compounds within ± 10% expected value.
Carbonate-bicarbonate (CO3-HCO3) Standard	Daily, Following ICV and ICB	a ± 10% from expected concentration; b < CRDL	1. Reprep and reanalyze CO3-HCO3 standard 2. Identify and document problem 3. Recalibrate and reanalyze samples if necessary	Analyst	Accuracy/ Bias	Compounds within ± 10% expected value.
Calibration Blank Verification (ICB, CCB)	After ICV and CCVs	< CRDL	1. Terminate analysis 2. Identify and document the problem 3. Recalibrate, verify and reanalyze all associated samples	Analyst	Accuracy/ Bias	< CRDL
CRDL Verification Standard (< 2X CRDL)	After initial CCV	± 20% from expected concentration	1. Reprep and reanalyze standard 2. Recalibrate and verify	Analyst	Accuracy/ Bias	± 20% from expected concentration

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory duplicate	1 for every 10 samples	RPD <20% for samples >5X CRDL; ± CRDL for samples <5X CRDL	RPD <20% for samples >5X CRDL; ± CRDL for samples <5X CRDL	Analyst	Precision	Flag associated data
Field duplicate	Sampling: 1 for every 10 field samples Lab: NA	RPD ≤ 30%	1) Review lab QC data to determine if they are in control. 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action.	Analyst	Precision-overall	RPD ≤ 30%
Quadruplicate Sample Analysis	Lab: Every sample	NA	All samples for method 9060 are to be analyzed in quadruplicate,	Analyst	Precision-overall	NA
Matrix spike (MS)	Sampling: 1 per 20 samples Lab: same	± 25% from expected value	NA	Analyst	Accuracy/ bias	± 25% from expected value Flag associated data
Laboratory Control Sample (LCS)	One per batch or SDG (1 per 20 samples minimum)	± 20% from expected concentration	1. Terminate analysis 2. Identify and document the problem 3. Reanalyze all associated samples	Analyst	Accuracy-lab	± 20% from expected concentration
Method blanks	One per Batch (1 per 20 samples minimum)	< CRDL	1. If lowest sample concentration is more than 10X the blank concentration, no action 2. If samples are non-detected, no action 3. If detected sample concentrations are less than 10X blank conc., all associated samples must be prepared again with another method blank and reanalyzed	Analyst	Accuracy/ bias-contamin-ation	< CRDL

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	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 #28 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Matrix	Groundwater
Analytical Group	Metabolic Acids (MBA) by HPLC
Concentration Level	Low/medium
Sampling SOP	SOP4
Analytical Method / SOP Reference	Metabolic Acids Method 830-MBA / L-3
Sampler's Name	HDR
Field Sampling Organization	HDR
Analytical Organization	Microbac
Number of Sample Locations	Per SAP

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 15% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst	Accuracy/ Bias	Flagging is not appropriate. No samples shall be analyzed until calibration has been verified with a second source.
Evaluation of relative retention time (RRT)	Every sample	RRT of each reported analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst	--	RRTs may be updated based on the daily CCV. RRTs shall be compared with the most recently updated RRTs.
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 corrective action.	Analyst	Precision-overall	RPD ≤ 30%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike (MS)	Sampling: One per 20 field samples Lab: One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst	Accuracy / bias	If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	Sampling: One per 20 samples (if MSD) Lab: One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst	Precision	The data shall be evaluated to determine the source of difference. For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative.
Surrogate spike recoveries	All field and QC samples.	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst	Accuracy/ bias	Alternative surrogates are recommended when there is obvious chromatographic interference. Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blanks	One per preparatory batch	No analytes detected >1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst	Accuracy/ bias-contamin-ation	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.
LCS	One per preparatory batch	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for the failed reported analytes, if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.	Analyst	Accuracy/ bias	Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Rinsate blank	Sampling: 1 per day per sampling team per matrix if using non-dedicated equipment Lab: None	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.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Confirmation of positive results (second column)	All positive results must be confirmed.	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column/detector RPD ≤ 40%.	NA	Analyst	Identification / Accuracy	Spectral match confirmation of a UV detector with a UV diode array detector (or vice versa) is not considered an acceptable confirmation technique. A second column confirmation is required. Use project-specific reporting requirements if available; otherwise, use method requirements, if available; otherwise, report the result from the primary column. Apply J-flag if RPD >40%. Discuss in the case narrative.
Results reported between MDL and RL.	--	--	--	--	--	Apply J-flag to all results between MDL and RL.

QAPP Worksheet #28 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Matrix	Groundwater
Analytical Group	Dissolved Gases by GC
Concentration Level	Low/medium
Sampling SOP	SOP4
Analytical Method / SOP Reference	EPA RSK-175 / L-4
Sampler's Name	HDR
Field Sampling Organization	HDR
Analytical Organization	Microbac
Number of Sample Locations	Per SAP

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Second-source calibration verification	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 20% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst	Accuracy/ Bias	No samples shall be analyzed until calibration has been verified with a second source. Flagging is not appropriate.
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 corrective action.	Analyst	Precision-overall	RPD ≤ 30%

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike (MS)	Sampling: 1 per every 20 samples Lab: same	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst	Accuracy	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference, i.e., matrix effect or analytical error.
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	Sampling: 1 per 20 samples (if MSD) Lab: 1 pair per every 20 samples	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. RPD ≤ 30% (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	Analyst	Precision-overall and accuracy/ bias	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the case narrative. The data shall be evaluated to determine the source of difference.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample (LCS)	One per preparatory batch.	A laboratory must use the QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst	Precision-lab	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Surrogate spike recoveries	All field and QC samples	QC acceptance criteria specified by the project, if available; otherwise use QSM Appendix C limits or in-house LCS limits if analyte(s) are not listed.	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst	Accuracy/ bias	Alternative surrogates are recommended when there is obvious chromatographic interference. Apply Q-flag to all associated analytes if acceptance criteria are not met and explain in the case narrative.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method blanks	One per preparatory batch	No analytes detected >1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	Analyst	Accuracy/ bias-contamin-ation	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Confirmation of positive results (second column)	All positive results must be confirmed.	Calibration and QC criteria for second column are the same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%.	NA	Analyst	Identification /accuracy	Use project-specific reporting requirements if available; otherwise, use method requirements if available; otherwise report the result from the primary column.
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 #28 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Matrix	Groundwater
Analytical Group	Metals by ICP
Concentration Level	Low/medium
Sampling SOP	SOP4
Analytical Method / SOP Reference	SW-846 6010 / L-5
Sampler's Name	HDR
Field Sampling Organization	HDR
Analytical Organization	Microbac
Number of Sample Locations	Per SAP

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Second-source calibration verification	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Analyst	Accuracy/ Bias	No samples shall be analyzed until calibration has been verified with a second source. Flagging is not appropriate.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Low-level Calibration Check Standard (Low-level ICV)	Daily	All reported analytes within ± 20% of true value.	Correct problem and repeat ICAL.	Analyst	Accuracy/ Bias/ Sensitivity	No samples shall be analyzed without a valid low-level calibration check standard (LLICV). Low level calibration check standard should be less than or equal to the LOQ. Flagging is not appropriate.
Linear Dynamic Range (LDR) or high-level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range.	Within ± 10% of true value.	Dilute samples to within the calibration range, or re-establish/ verify the LDR.	Analyst	Accuracy/ Bias/ Sensitivity	Data cannot be reported above the high calibration range without an established/passing high level check standard. Flagging is not appropriate.
Field duplicate	Sampling: 1 for every 10 field samples Lab: NA	RPD ≤ 30%	1) Review lab QC data to determine if they are in control. 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action.	Analyst	Precision-overall	RPD ≤ 30%

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

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

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

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	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 #28 (UFP-QAPP Manual Section 3.4) -- QC Samples Table

Matrix	Air (soil vapor)
Analytical Group	VOCs
Concentration Level	Low/medium
Sampling SOP	SOP5
Analytical Method / SOP Reference	TO-15 / L-6
Sampler's Name	HDR
Field Sampling Organization	HDR
Analytical Organization	CAS
Number of Sample Locations	Per SAP

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Second-source calibration verification	Once per ICAL	All analytes within ± 30% of expected value	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Problem must be corrected. Samples may not be analyzed until the calibration has been verified.	Analyst	Accuracy/ Bias	All analytes within ± 30% of expected value Flagging criteria are not appropriate.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Internal Standards (ISs)	Every field sample, standard, and QC sample.	Area response within ± 40% of the area response for each internal standard in the most recent valid calibration (CCV or mid-point from the initial calibration, whichever is most current). Retention time within ± 0.33 minutes of the retention time for each internal standard in the most recent valid calibration (CCV or mid-point from the initial calibration, whichever is most current).	1) Inspect MS and GC for malfunctions. 2) Take appropriate corrective actions. 3) Reanalyze samples analyzed while system was malfunctioning. 4) If sample exceeds criteria, reanalyze sample. If still out, report both analyses and document corrective action.	Analyst	Accuracy	Area response within ± 40% of the area response for each internal standard in the most recent valid calibration (CCV or mid-point from the initial calibration, whichever is most current). Retention time within ± 0.33 minutes of the retention time for each internal standard in the most recent valid calibration (CCV or mid-point from the initial calibration, whichever is most current). Apply Q-flag to analytes associated with the non-compliant IS.
Method blank (humid zero air)	Immediately after ICV or daily CCV, and whenever a high concentration sample is encountered and carryover is suspected	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected > RL. Blank result must not otherwise affect sample results.	The source of the contamination must be investigated and appropriate corrective measures need to be taken and documented before further sample analysis proceeds. If an analyte in the blank is found to be out of control (i.e., contaminated) and the analyte is also found in associated samples, those sample results should be "flagged" by the lab as possibly contaminated.	Analyst	Accuracy/bias-contamination	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For common laboratory contaminants, no analytes detected > RL. Blank result must not otherwise affect sample results. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
LCS for all analytes	One LCS per analytical batch	All analytes within ± 30% of expected value	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Problem must be corrected. Samples may not be analyzed until the calibration has been verified.	Analyst	Precision-lab	All analytes within ± 30% of expected value Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.
Surrogate spike recoveries	Every sample, spike, standard, and reagent blank	70-130% recovery	1) Recalculate result, and reanalyze sample if still out. 2) Re-extract and reanalyze sample, if still out. 3) Report both analyses and document in report that steps 1 and 2 were performed. 4) Qualify the data.	Analyst	Accuracy/bias	70-130% recovery Apply Q-flag to all associated analytes if acceptance criteria are not met.
Field duplicate	Sampling: 1 for every 10 field samples Lab: NA	RPD ≤ 25%	1) Review lab QC data to determine if they are in control. 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action.	Analyst	Precision-overall	RPD ≤ 25%
Laboratory duplicate	Daily	RPD ≤ 25%	1) Review lab QC data to determine if they are in control. 2) Qualify data. Use data to evaluate whether proper collection procedures were followed. 3) Determine further corrective action.	Analyst	Precision-laboratory	RPD ≤ 25%
MDL study	Once per year, upon any major system change, or quarterly MDL check.	Method detection limits established as described in 40 CFR Part 136, App. B shall not exceed one-half the RL	Method detection limits that exceed established criteria shall be submitted to the USACE for approval prior to the analysis of any project samples.	Analyst	Sensitivity	Method detection limits established as described in 40 CFR Part 136, App. B shall not exceed one-half the RL

QAPP Worksheet #29 (UFP-QAPP Manual Section 3.5.1) -- Project Documents and Records Table

Sample Collection Documents and Records	On-Site Analysis Documents and Records	Off-Site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field notebook	Sample Receipt, Custody, and Tracking Records	Sample Receipt, Custody, and Tracking Records	Field Sampling audit Checklists	
COC	Standards Traceability Logs	Standards Traceability Logs	Field Analysis Audit Checklists	
Sampling sheets	Equipment Calibration Logs	Equipment Calibration Logs	Fixed Laboratory Audit Checklists	
Air Bills	Sample Prep Logs	Sample Prep Logs	Data Validation Reports	
Custody Seals	Instrument Run Logs	Instrument Run Logs	Corrective Action Forms	
Telephone Logs	Equipment Maintenance, Testing, and Inspection Logs	Equipment Maintenance, Testing, and Inspection Logs	Reported Results for Standards, QC Checks, and QC Samples	
Corrective Action Forms	Corrective Action Forms	Corrective Action Forms	Reported Field Sample Results	
DQCR Forms	Reported Field Sample Results	Reported Field Sample Results	Telephone Logs	
SOPs	Sample Disposal Records	Reported Results for Standards, QC Checks, and QC Samples	Instrument Printouts (raw data) for Field Samples, Standards, QC Checks and QC Samples	
Field Sampling Plan	Telephone Logs	Instrument Printouts (raw data) for Field Samples, Standards, QC Checks and QC Samples	Data Package Completeness Checks	
QAPP	Field Sampling Plan	Data Package Completeness Checks	Field Sampling Plan	
Health and Safety Plan	QAPP	Sample Disposal Records	QAPP	
	Health and Safety Plan	Telephone Logs		
	Well Logs	Extraction/Cleanup Records		
		Raw Data (stored on disk or DC-R)		
		Field Sampling Plan		
		QAPP		
		Health and Safety Plan		

QAPP Worksheet #30 (UFP-QAPP Manual Section 3.5.2.3) -- Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Locations/ID Number	Analytical SOP	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Water	VOCs	Low/medium	Per SAP	L-1	15 working days for Level 3 report and EDDs data, 20 working days for Level 4 report	Microbac Laboratories, Inc. 158 Starlite Drive Marietta, Ohio 45750 Stephanie Mossburg (800) 373-4071 Stephanie.mossburg@microbac.com	CT Laboratories, LLC 1230 Lange Court Baraboo, Wisconsin 53913 608.356.2760 cberwanger@ctlaboratories.com
Water	TOC	Low/medium	Per SAP	L-2			
Water	Metabolic Fatty Acids	Low/medium	Per SAP	L-3			
Water	Dissolved Gases	Low/medium	Per SAP	L-4			
Water	Metals	Low/medium	Per SAP	L-5			
Air (soil vapor)	VOCs	Low/medium	Per SAP	L-6	15 working days for Level 3 report and EDDs data, 20 working days for Level 4 report	ALS Global (ALS) 2655 Park Center Drive, Suite A Simi Valley, California 93065 Kate Aguilera (805) 526-7161 kaguilera@caslab.com	NA

QAPP Worksheet #31 (UFP-QAPP Manual Section 4.1.1) -- Planned Project Assessments Table

Assessment Type	**Frequency**	**Internal or External**	**Organization Performing Assessment**	**Person(s) Responsible for Performing Assessment** (title and organizational affiliation)	**Person(s) Responsible for Responding to Assessment Findings** (title and organizational affiliation)	**Person(s) Responsible for Identifying and Implementing Corrective Actions (CA)** (title and organizational affiliation)	**Person(s) Responsible for Monitoring Effectiveness of CA** (title and organizational affiliation)
Field Sampling Technical Systems Audit (TSA)	Biennial	I	HDR	Project Chemist/QA Officer, HDR	PM and Field Sampling Lead, HDR	PM and Field Sampling Lead, HDR	PM, QA Officer/ Project Chemist, HDR
On-Site Analytical TSA	Biennial (concurrent with Field Sampling TSA)	I	HDR	Project Chemist/QA Officer, HDR	PM and Field Sampling Lead, HDR	PM and Field Sampling Lead, HDR	PM, QA Officer/ Project Chemist, HDR
Off-Site Laboratory TSA	Annual	E	ELAP	DoD Environmental Laboratory Accreditation Program (ELAP) personnel or contractor	Laboratory Representative	Laboratory PM, Analysts, Technicians	Laboratory PM, and PM and Project Chemist/QA Officer, HDR
Lab Performance Audit	Ongoing with data package data validation	E / I	Diane Short & Associates (DSA) and HDR	Data reviewer/validator, DSA and Project Chemist/QA Officer, HDR	Laboratory PM, Analysts, Technicians	Laboratory PM, Analysts, Technicians	Laboratory PM, and PM and Project Chemist/QA Officer, HDR
Data Review TSA	Ongoing with review of data validation reports and qualifications	I	HDR	Project Chemist/QA Officer, HDR	Data reviewer/validator, DSA	Data reviewer/validator, DSA	PM and Project Chemist/QA Officer, HDR

QAPP Worksheet #32 (UFP-QAPP Manual Section 4.1.2) -- Assessment Findings and Corrective Action Responses

Assessment Type	**Nature of Deficiencies Documentation**	**Individual(s) Notified of Findings** (name, title, organization)	**Timeframe of Notification**	**Nature of Corrective Action Response Documentation**	**Individual(s) Receiving Corrective Action Response** (name, title, organization)	**Timeframe for Response**
Field Sampling Technical Systems Audit (TSA)	Verbal, written in audit notebook, typed copy	PM and Field Sampling Lead, HDR	Immediately upon encountering action requiring correction	Update/addition to SOP; notification to sampling personnel of changes	PM, QA Officer/ Project Chemist, HDR	Prior to next sampling event
On-Site Analytical TSA	Verbal, written in audit notebook, typed copy	PM and Field Sampling Lead, HDR	Immediately upon encountering action requiring correction	Update/addition to SOP; notification to sampling personnel	PM, QA Officer/ Project Chemist, HDR	Prior to next on-site analytical event (sampling event)
Off-Site Laboratory TSA	In ELAP annual certification audit	Laboratory Representative	Unknown	Per ELAP	Laboratory PM, and PM and Project Chemist/QA Officer, HDR	Per ELAP
Lab Performance Audit	Phone call and/or email from DSA to HDR QA Officer/Project Chemist, followed by phone call or email from HDR to lab PM	Project Chemist/QA Officer, HDR; and Laboratory PM, Analysts, Technicians	Immediately upon encountering issue requiring correction or clarification	Documented in data package if it required edits to the data package	Laboratory PM, and PM and Project Chemist/QA Officer, HDR	Corrections are to be made before final data package is issued, and included in final data package
Data Review TSA	Phone call and/or email from HDR QA Officer/Project Chemist to DSA; Data Review Report Summary Report	Data reviewer/validator, DSA; PM, HDR	Immediately upon encountering issue requiring correction or clarification; Summary Report prepared for each sampling event	Summary Report prepared for each sampling event, to summarize major issues with the data and to explain changes made to the Data Review Reports prepared by DSA	PM and Project Chemist/QA Officer, HDR	Within one week from receipt of final Data Review Report for a sampling event

QAPP Worksheet #33 (UFP QAPP Manual Section 4.2) -- QA Management Reports Table

Type of Report	**Frequency** (daily, weekly monthly, quarterly, annually, etc.)	**Projected Delivery Date(s)**	**Person(s) Responsible for Report Preparation** (title and organizational affiliation)	**Report Recipient(s)** (title and organizational affiliation)
Field Daily Quality Control Report (DQCR)	Daily during sampling events	Daily	FTL, HDR	PM, HDR
Field Sampling TSA Report	Biennialllly	One month after audit	Project Chemist/QA Officer, HDR	PM and FTL, HDR
Lab Performance Audit Report	Annually	As required by previous audit report	DoD Environmental Laboratory Accreditation Program (ELAP) personnel or contractor	Laboratory Representative
Data Review Report (validation)	Every data package	One week after receipt of data package	Data validator, DSA	PM and Project Chemist, HDR
Data Usability Assessment Report (as part of Data Review Report)	Every data package	One week after receipt of data package	Data validator, DSA	PM and Project Chemist, HDR
Data Review Report Summary and Usability Report	One report for every sampling event, possibly comprising several data packages	After all Data Review Reports for a project have been completed	Project Chemist, HDR	PM, HDR
DQE (as section in QA reports)	As needed for QA reports	TBD	Project Chemist, HDR	PM, HDR
Final Project Report	As needed	TBD	PM and Project Chemist, HDR	Client

QAPP Worksheet #34 (UFP-QAPP Manual Section 5.2.1) -- Verification (Step I) Process Table

Verification Input	**Description**	**Internal/ External**	**Responsible for Verification** (name, organization)
Chain of custody	- Chain-of-custody (COC) forms will be reviewed and verified against the packed sample coolers, field notes and Sampling Plan Detail (SPD). - The shipper will sign the COC form and retain a copy of the form in the site file. -Original and remaining copies of the COC will be sealed in a plastic bag and included in the sample cooler. - See SOPs #3, #5 and #6 for further details.	Internal	FTL, HDR Data validator, DSA
Field Notes	- Field notes will be scanned and stored on the network with the project. These notes will be referred to when there is an apparent discrepancy in sample ID, sampling time, field conditions, etc. noted during sample receipt at the laboratory or during data verification. - Field notes will contain information on field duplicate IDs.	Internal / External	Sampler, HDR Project Chemist, HDR for Data validator, DSA
Analytical data package	-All analytical data packages will be verified internally by the laboratory performing the work for completeness prior to submittal, according to procedures in SOPs L-1, L-2, L-3, L-4 and L-5.	Internal	Project Chemist at Lab
Analytical data package	-The data packages and summaries of all QC sample results will be verified for completeness (presence/absence). -See following data verification table for a list of inputs.	External	Data validator, DSA

Step I Verification Item	Responsible Person, Organization
Planning Documents	
Evidence of required approval of plan (QAPP)	Project Chemist, HDR
Identification of personnel (those involved in the project and those conducting verification steps)	Data validator, DSA
Laboratory name	Data validator, DSA
Methods (sampling and analysis)	Data validator, DSA
Performance requirements (including QC criteria) for all inputs	Data validator, DSA
Project quality objectives	Project Chemist, HDR
Reporting forms	Data validator, DSA
Sampling plans, location, maps, grids, and sample ID numbers	Project Chemist, HDR
Site identification	Data validator, DSA
SOPs (sampling and analytical)	Project Chemist, HDR
Analytical Data Package	
Case narrative	Data validator, DSA
Internal laboratory chain of custody	Data validator, DSA
Sample condition upon receipt, and storage records	Data validator, DSA
Sample chronology (time of receipt, extraction, and analysis)	Data validator, DSA
Identification of QC samples (sampling or lab, temporal, and spatial)	Data validator, DSA
Communication logs	Data validator, DSA
Copies of laboratory notebook, records, prep sheets	Data validator, DSA
Corrective action reports	Project Chemist, HDR
Documentation of corrective action results	Project Chemist, HDR
Definitions of laboratory qualifiers	Data validator, DSA
Documentation of individual QC results (e.g., spike, duplicate, LCS)	Data validator, DSA
Documentation of laboratory method deviations	Data validator, DSA
Electronic data deliverables	Project Chemist, HDR and Data validator, DSA
Instrument calibration reports	Data validator, DSA
Laboratory name	Data validator, DSA
Laboratory sample identification numbers	Data validator, DSA
QC sample raw data	Project Chemist, HDR
QC summary report	Data validator, DSA
Raw data	Project Chemist, HDR
Reporting forms, completed with actual results	Data validator, DSA
Signatures for laboratory sign-off (e.g., laboratory QA manager)	Data validator, DSA
Sampling Documents	
Chain of custody	Data validator, DSA
Communication logs	Data validator, DSA
Corrective action reports	Project Chemist, HDR
Documentation of corrective action results	Project Chemist, HDR
Documentation of deviation from methods	Data validator, DSA
Documentation of internal QA review	Data validator, DSA
Identification of QC samples	Project Chemist, HDR and Data validator, DSA
Sampling location and plan	Project Chemist, HDR
Sampling notes and drilling logs	Project Chemist, HDR
External Reports	
External audit report	Project Chemist, HDR
Laboratory QA plan	Project Chemist, HDR
NELAP accreditation	Project Chemist, HDR

QAPP Worksheet #35 (UFP-QAPP Manual Section 5.2.2) -- Validation (Steps IIa and IIb) Process Table

Step IIa or IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	**Data Deliverables and QAPP**	-Ensure that all required information on sampling and analysis from Step I was provided (including planning documents).	Data validator, DSA
IIb	**Data Deliverables and QAPP**	-Ensure that the data report from Step IIa was provided.	Data validator, DSA
IIb	**Deviations**	-Determine the impacts of any deviations from sampling or analytical methods and SOPs. For example, confirm that the methods given in the QAPP were used and, if they were not, determine if data still meet goals. -Consider the effectiveness and appropriateness of any corrective action.	Data validator, DSA
IIa	**Analytes**	-Ensure that the required lists of analytes were reported.	Data validator, DSA
IIb	**Analytes**	-Ensure that the required RLs and MDLs were reported as specified in the QAPP or SAP.	Data validator, DSA
IIa	**Chain-of-Custody**	-Ensure that COC records were present.	Data validator, DSA
IIb	**Chain-of-Custody**	-Examine the traceability of the data from time of sample collection until reporting of data. -Examine chain-of-custody records against contract, method, or procedural requirements.	Data validator, DSA
IIa	**Holding Times**	-Ensure that actual and required holding times were reported.	Data validator, DSA
IIb	**Holding Times**	-Identify holding time criteria, and either confirm that they were met or document any deviations. -If holding times were not met, confirm that deviations were documented, that appropriate notifications were made, and that approval to proceed was received prior to analysis.	Data validator, DSA Project Chemist, HDR
IIa	**Sample Handling**	-Ensure that sample handling, receipt, and storage procedures were reported.	Data validator, DSA
IIb	**Sample Handling**	-Ensure that required sample handling, receipt, and storage procedures were followed, and that any deviations were documented, and determine the effect of deviations on data quality/usability.	Data validator, DSA
IIa	**Sampling Methods and Procedures**	-Establish that required sampling methods and procedures and field measurements were reported.	Data validator, DSA
IIb	**Sampling Methods and Procedures**	-Establish that required sampling methods and procedures and field measurements were used and that any deviations were noted, and determine the effect of deviations on data quality/usability.	Data validator, DSA

Step IIa or IIb	Validation Input	Description	**Responsible for Validation** (name, organization)
IIb	**Sampling Plan**	-Determine whether the sampling plan was executed as specified (i.e., the number, location, and type of field samples were collected and analyzed as specified in the QAPP).	Project Chemist, HDR
IIa	**Analytical Methods and Procedures**	-Establish that required analytical methods (off-site laboratory) were specified.	Data validator, DSA
IIb	**Analytical Methods and Procedures**	-Establish that required analytical methods (off-site laboratory) were used and that any deviations were noted.	Data validator, DSA
IIa	**QC Sample Summaries**	-Ensure that the QC sample result summaries listed control limits.	Data validator, DSA
IIb	**QC Sample Summaries**	-Ensure that the QC samples met performance criteria and that any deviations were documented.	Data validator, DSA
IIa	**Data Qualifiers**	-Determine that the laboratory data qualifiers were defined.	Data validator, DSA
IIb	**Data Qualifiers**	-Determine that the laboratory data qualifiers were defined and applied as specified in methods, procedures, or contracts.	Data validator, DSA
IIb	**Laboratory Transcription**	-Authenticate accuracy of the transcription of analytical data to the EDD.	Data validator, DSA Project Chemist, HDR
IIb	**Standards**	-Determine that standards are traceable and meet contract, method, or procedural requirements.	Data validator, DSA
IIb	**Audits**	-Review field and laboratory audit reports and accreditation and certification records for the laboratory's performance on specific methods.	Project Chemist, HDR
IIb	**Co-located Field Duplicates**	-Compare results of collocated field duplicates with criteria established in the QAPP.	Data validator, DSA
IIb	**Project Quantitation Limits**	-Determine that quantitation limits were achieved, as outlined in the QAPP and that the laboratory successfully analyzed a standard at the QL.	Data validator, DSA
IIa	**Step IIa Validation Report**	-Summarize missing information.	Data validator, DSA
IIb	**Step IIb Validation Report**	-Summarize deviations from methods, procedures, or contracts. Include qualified data and explanation of all data qualifiers.	Data validator, DSA
IIb	**EDD Qualifiers**	-Add validation data qualifiers to the EDD.	Data validator, DSA
IIb	**EDD Qualifiers**	-Verify appropriate validation qualifiers were used, and add final qualifiers to the EDD.	Project Chemist, HDR

QAPP Worksheet #36 (UFP-QAPP Manual Section 5.2.2) -- Validation (Steps IIa and IIb) Summary Table

Step IIa / IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	**Data Validator** (title and organizational affiliation)
IIa / IIb	Groundwater	VOCs	Low/medium	Worksheet #28 and SOP10	Data validator, DSA and Project Chemist, HDR
IIa / IIb	Groundwater	TOC	Low/medium		
IIa / IIb	Groundwater	Metabolic Fatty Acids	Low/medium		
IIa / IIb	Groundwater	Dissolved Gases	Low/medium		
IIa / IIb	Groundwater	Metals	Low/medium		
IIa / IIb	Air (soil vapor)	VOCs	Low/medium		

QAPP Worksheet #37 (UFP-QAPP Manual Section 5.2.3) -- Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:
The PARCCS parameters (Precision, Accuracy, Representativeness, Completeness, Comparability and Sensitivity) will be assessed.

Describe the evaluative procedures used to assess overall measurement error associated with the project:
Measurement error will be deemed within acceptable limits when project DQOs as assessed by PARCCS parameters are met.

Identify the personnel responsible for performing the usability assessment:
The HDR Project Chemist will assess the PARCCS parameters and determine overall usability of the data. In general, non-rejected data will be considered usable.

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:
The PC will write a brief assessment of data usability for each data package. Trends, relationships and anomalies will be presented in the project reports.

APPENDIX B

HDR 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 5 – Vapor Sample Collection

SOP 6 – EBT Mixing and Injections

SOP 7 – Sample Control and Documentation

SOP 8 – Sample Packing and Shipping

SOP 9 – Equipment Decontamination

SOP 10 – Data Verification, Validation, Qualification and Usability Assessment

SOP 11 –Field Sampling Technical Systems Audit

STANDARD OPERATING PROCEDURE 1 - GENERAL PROCEDURES FOR FIELD PERSONNEL

Lead Organization: Department of the Army (DA)

Preparing Organization: HDR

SOP Approved by: Field Team Leader: Justin Bills
Project QA Officer: Lynn Lutz
Project Manager: Tom Holmes

1.0 PURPOSE AND SUMMARY

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.

2.0 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 Health and Safety Plan (HASP) for the specific project activity. Upon arrival at the site, each person shall sign the acknowledgement sheet confirming their review of the HASP. Personal protective equipment (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. In particular, evaluation and repair of remediation systems (air sparge and soil vapor extraction) will only be performed by personnel familiar with the systems. 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).

3.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

Field activities will be directed by the FTL, a mid- or senior level 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.

4.0 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.

5.0 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;
    - Review project work plan, HASP, and QAPP.
    - Work with PM to properly staff the field activity.
    - Coordinate sampling activities with the project chemist and analytical laboratory.
    - Confirm availability and condition of DDMT-owned equipment and order additional equipment/supplies for delivery prior to the start of each event.
    - Prepare field forms and other documentation for the planned event.

- If work is to be subcontracted, review the subcontract agreement, work plan, and HASP.
- 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 HASP.
- 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:
   - The identification number and calibration results for each field instrument
   - The numerical value and units of each measurement
   - 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:

   - Decontaminate and check condition of field equipment.
   - Provide log books and other field documentation to FTL for review and scanning.
   - Properly dispose of trash, debris and used PPE.
   - Safely store purge and decontamination water, or transfer to large storage tanks at Dunn Field.
   - Make arrangements for shipment of samples (if applicable) and follow-up with the analytical laboratory to confirm samples arrived in good condition.
   - Complete activity-specific field reports as required by applicable SOPs.
   - Complete the Daily Field Report and submit to PM (Attachment 1-2).

5.3 Field Log Books and Documentation

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

   - The first page of each log book will list the following information:
     - Site Name: Former Defense Depot Memphis Tennessee
     - EPA ID (TN4210020570)
     - Project Location: 2241 Truitt Street, Memphis, TN 38114
   - The first entry for each field event will list the following information: log books:
     - Project Name and Number
     - FTL (full name) and initials
     - Sample team leader and members (full names) and initials
   - At minimum, the log book will describe general activities performed, date and time, personnel and weather conditions. All field equipment calibration and maintenance records will be documented in the logbook. Communications with the FTL, PM or project chemist regarding field activities will be documented. Additional field data will be recorded in the log book if other field records are not used.

- Any deviations from the QAPP or workplan 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).

6.0 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.

7.0 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.

8.0 REFERENCES

MACTEC *RA SAP Volume I: Field Sampling Plan, Defense Depot Memphis, Tennessee, Revision 1*, *WTP-1 General Procedures for Field Personnel*. November, 2005.

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

Attachment 1-1

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.)

Attachment 1-1

Rental Equipment

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

Lab-provided Sampling Supplies

Sample Type	Number	Supplies

Additional Tools/Supplies

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

Attachment 1-1

Final Check

1. All required equipment/tools received and condition checked

Yes ___ No ___ Comment:

2. Initial equipment calibration completed

Yes ___ No ___ Comment:

3. Vehicles inspected

Yes ___ No ___ Comment:

4. Field locations reviewed

Yes ___ No ___ Comment:

5. Weather forecast checked

Yes ___ No ___ Comment:

6. Staff documents (OSHA, DL) checked

Yes ___ No ___ Comment:

7. Review of project plans confirmed and activities discussed

Yes ___ No ___ Comment:

8. Initial Safety Meeting held and HASP signed

Yes ___ No ___ Comment:

Attachment 1-2

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**

Attachment 1-3

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:

Attachment 1-3

Close-out Checklist

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

Yes ___ No ___ Comment:

2. Equipment/tools decontaminated

Yes ___ No ___ Comment:

3. Rental equipment shipped to supplier

Yes ___ No ___ Comment:

4. Rental vehicles returned

Yes ___ No ___ Comment:

5. DDMT equipment and tools properly stored

Yes ___ No ___ Comment:

6. List damaged equipment

Yes ___ No ___ Comment:

7. Replacement supplies ordered

Yes ___ No ___ Comment:

8. Field locations inspected and trash/debris removed

Yes ___ No ___ Comment:

9. Field shop/office cleaned

Yes ___ No ___ Comment:

STANDARD OPERATING PROCEDURE 2 – DRILLING AND SOIL SAMPLING

Lead Organization: Department of the Army (DA)

Preparing Organization: HDR

SOP Approved by: Field Team Leader: Justin Bills
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 at DDMT; it has proven to be the most effective method for boring advancement and well installation based on the depth to water (i.e. 75-105 feet below ground surface [ft bgs]) and geologic characteristics of the fluvial aquifer (i.e. tight sands mixed with gravel up to cobble size).

Drilling activities that require the use of a truck-mounted drill rig will be conducted by a Tennessee-licensed subcontractor with experience on similar projects. The drilling subcontractor will advance boring to the target depth using the selected drilling technology and provide equipment sufficient to carry out the work as specified. Drilling and soil sampling will be overseen by the field team leader (FTL), a qualified geologist/engineer, 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 site-specific Health and Safety Plan (HASP).

3 HEALTH AND SAFETY

Proper safety precautions must be observed during drilling activities and when collecting soil samples in accordance with the HASP. 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 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 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, a mid- or senior level 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 HASP. 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, 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 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:
    - Review project-specific work plan, HASP, QAPP, and for subcontracted work, review of the subcontract agreement.

- Work with PM to properly staff the field activity.
- Arrange site access with the property manager (Colliers International-Memphis Depot Industrial Park), tenants and/or property owners.
- Have a surveyor locate all of the proposed drilling locations, and mark each location with a wooden stake and white flagging or white paint.
- Notify the Tennessee One Call underground utility location and, if necessary, a private utility location service.
- Provide drilling subcontractor with proposed boring location and depth for well permits from Shelby County Health Department (SCHD); confirm receipt of permits.
- Coordinate sampling activities and supplies with the project chemist and analytical laboratory.
- Confirm availability and condition of DDMT-owned equipment and order additional equipment/supplies for delivery prior to the start of each event.
- Prepare field forms and other documentation for the planned event.
- Provide all HDR and subcontracted field personnel with time and location for personnel to meet prior to beginning field activities.
- 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 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 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, 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 HASP. 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

6.2.1.1 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.

6.2.1.2 Headspace Sampling

At five-foot intervals within the soil cores, the headspace will be screened with a flame ionization detector (FID) or 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°C (68°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/FID and recording the highest reading.
- If the reading is > 10 ppm, 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.

6.2.1.3 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. 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 chain-of-custody (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.

6.2.1.3.1 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.

6.2.1.3.2 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.

6.3 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

MACTEC *RA SAP Volume I: Field Sampling Plan, Defense Depot Memphis, Tennessee, Revision 1*, *WTP-2 Drilling Operations*, and *WTP-11 Soil 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.

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

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

STANDARD OPERATING PROCEDURE 3 – WELL INSTALLATION, DEVELOPMENT AND ABANDONMENT

Lead Organization: Department of the Army (DA)

Preparing Organization: HDR

SOP Approved by: Field Team Leader: Justin Bills

Project QA Officer: Lynn Lutz

Project Manager: Tom Holmes

1.0 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.

2.0 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.

3.0 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 (HASP). 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).

4.0 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.

5.0 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.

6.0 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;

    - Review project project-specific work plan, HASP, and QAPP and for subcontracted work, review of the subcontract agreement.
    - Work with PM to properly staff the field activity.
    - Arrange site access with the Memphis Depot Associates, tenants and/or property owners.
    - Confirm availability and condition of DDMT-owned equipment and order additional equipment/supplies for delivery prior to the start of each event.
    - Prepare field forms and other documentation for the planned event.
    - Prepare the required Shelby County Health Department (SCHD) well installation and abandonment forms.
    - Provide all HDR and subcontracted field personnel with time and location for personnel to meet prior to beginning field activities.
    - Confirm that field staff have a valid Driver's License (or other picture identification) and current OSHA Certification in their possession prior to leaving the office.

6.1.2 Field

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

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

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

6.2 Field Operations

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

6.2.1 Monitoring Well Installation

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

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

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

- The length of risers, screens, and end caps for each monitoring well including adjustment for riser sections cut off during installation.

- Record the type, manufacturer, and gradation of the filter sand, and the volume used for each well.
- The type and manufacturer of the Portland cement and bentonite and the volume used for the bentonite seal and grout at each well.
- Record surface completion details including: completion type, number of bollards installed, and a description of surface completion materials.

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

- If the boring was drilled deeper than the total depth of the well, backfill the boring to approximately 1 foot below the planned well depth in accordance with the work plan, either with bentonite or by allowing the formation material to collapse as the casing is raised.
- Remove the new polyvinyl chloride (PVC) or stainless steel screen and riser from manufacturer packaging and decontaminate as described in SOP9 *Equipment Decontamination*.
- Install a 10 to 20-foot section of minimum 2-inch (I.D.), threaded, flush jointed, pre-manufactured PVC or stainless steel screen inside the steel drill casing.
- Install solid riser to ground surface, plus stick-up (if required).
- Install the filter pack using the gravity method through the annular opening between drill casing and well screen as the drill casing is removed. Continue removing drill casing and installing filter pack until at least 4 feet above the top of the well screen. Use the sonic drilling head to vibrate the steel casing as it is slowly withdrawn to distribute and compact the filter pack around the screen and to prevent bridging. Measure the thickness of the filter pack as it is placed.
- Install a minimum 5-foot bentonite seal. If bentonite is gravity fed in dry form, the seal will be hydrated with potable water. Allow the bentonite seal a minimum of 4 hours of hydration time before grouting the annulus. If the seal is in the saturated section of if potential for bridging is an issue, a bentonite slurry can be installed using a side-discharge tremie pipe.
- Remove remaining drill casing and grout boring annulus to ground surface with grout/bentonite mixture.
- Develop the well at least 24 hours after grout installation.

6.2.1.1 Well Construction Materials

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

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

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

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

6.2.1.2 Well Design

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

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

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

6.2.1.2.1 Screen Location

The screened intervals will be selected for each proposed well, based on visual observations of aquifer materials encountered, as recorded on the drilling log, and objectives in the project work plan. There are several water bearing units of interest at DDMT (fluvial, intermediate, and Memphis aquifer). Both the fluvial and intermediate aquifers can be found in unconfined conditions, with significant saturated thickness (>50 feet). In many areas, the saturated thickness of the fluvial aquifer is 20 feet or less. For most wells at DDMT the screen will start from the top of clay upward, for a maximum of 20 feet of screen per well. If the saturated thickness is substantially greater than 20 feet, cluster (or nested) wells may be installed to screen the entire saturated interval.

6.2.1.2.2 Filter Pack

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

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

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

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

placed. As the steel casing is slowly withdrawn between lifts, it will be vibrated with the sonic drilling head to compact the sand filter pack.

6.2.1.2.3 Bentonite Seal

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

6.2.1.2.4 Grout Seal

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

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

6.2.1.2.5 Surface Completion

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

If an aboveground surface completion is used, the well casing will be extended 2 or 3 feet above ground surface and secured with a water-tight cap. The protective casing will be a steel sleeve placed over the casing and cap; the steel sleeve diameter will be at least 4 inches greater than the casing diameter. The

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

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

6.2.1.2.6 Location Survey

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

6.2.1.3 Well Installation Diagrams

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

- Well identification.
- Drilling method.
- Installation date(s).
- Total boring depth.
- Lengths and descriptions of the screen and riser.
- Thickness and descriptions of filter pack, bentonite seal, annular grout, and any backfilled material.

- Quantities of all well construction materials used.

6.2.2 Well Development

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

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

The monitoring well development protocol is as follows:

- Measure the static water level (SWL) and the depth to the top of sediment in the well.
- Record the total depth of the well (from the Well Installation Diagram).
- Calculate the volume of water in the well and saturated annulus.
- Begin developing the well using a combination of surging and pumping. Continue pumping and periodically surging until each the following criteria have been met:
    a. Fluids lost to the formation during drilling and well installation have been removed (this is a minimum requirement where conditions permit).
    b. 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.
    c. 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.
    d. 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.

7.0 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.

8.0 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.

9.0 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: Justin Bills

Project QA Officer: Lynn Lutz

Project Manager: Tom Holmes

1.0 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.

2.0 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.

3.0 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.

4.0 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.

5.0 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 3-1.
- 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 dedicated pump or 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.
4. Turn the water level indicator on and slowly lower it into the well until it alerts to the water level.
5. 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 from an area marked on top of the casing; if no mark is present, use the north side of the casing.
6. Put the cap and lock back on the well casing and then close the well box. At this time assess the well condition record any cracks in the pad, missing bolts, missing caps, etc.
7. 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 a YSI 650 MDS or similar multi-probe device with flow-through cell. Flow cells add efficiency to low flow purging and field sampling applications. Calibration procedures for the YSI 650 MDS are provided in the operations manual.

Groundwater samples will be collected when water quality indicators of dissolved oxygen (DO), redox potential (ORP), pH, specific conductivity, and turbidity stabilize. Readings will be taken every 5 to 10 minutes and recorded on the Sample Collection Data sheet (Attachment 3-2). Stabilization is achieved after three successive readings are within ± 0.1 for pH, 10 millivolts (mV) for ORP, ± 5% for specific conductance, 10% for DO, and <10 nephelometric turbidity units (NTU) for turbidity. Temperature will also 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 may be collected from monitoring and injection wells, or piezometers. In most cases, dedicated bladder pumps and passive diffusion bags (PDBs) are used for sampling. In some wells a portable bladder or a disposable bailer will be used. Decontamination of portable pumps is required prior to each use in accordance with SOP 9.

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 3-2)
4. 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. The water level probe should be carefully lowered down the well to minimize disturbance.
5. Decontaminate the water-level indicator and tape prior to each use. 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; measuring to the bottom of the well casing may cause re-suspension of settled solids.

5.2.3.1 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. New disposable bailers will be used for sampling. Purging and sampling will be conducted in a manner that minimizes the agitation of sediments in the well and formation; the bailer will not be allowed to free fall into a well.

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

1. Measure the static water level prior to purging using a decontaminated electronic water level indicator. The probe of the water level indicator will be lowered into the well bore and the water level will be recorded.

2. Attach the Teflon coated stainless steel leader rope to the bailer and polypropylene (or nylon) rope to the Teflon coated rope. Lower the bailer into the well, until it contacts the water surface. Allow the bailer to sink and fill with a minimum of water surface disturbance. Slowly withdraw the bailer from the well, preventing the bailer and bailing line from touching the ground.

3. The well should be purged until a minimum of three well volumes is removed from the well, and the water quality indicators of DO, ORP, 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, 10 mV for ORP, 5% for specific conductance, 10% for DO, and <10 NTU for turbidity. Temperature will also be measured and recorded, but will not be used as a stabilization parameter. Sampling may begin once the well has stabilized. If, after three well volumes have been removed, stabilization does not occur or turbidity cannot be reduced below 10 NTU, additional purging (up to five well volumes), should be performed. If the parameters have not stabilized within five volumes, the field team leader should be contacted for direction.

4. If the well is purged dry, a sample will be collected as soon as sufficient recharge has occurred and within 24 hours. Temperature, specific conductance, turbidity, ORP, 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. 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).

7. After samples have been collected, replace the well cap and lock the security casing.

8. 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).

9. 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).

5.2.3.2 Sampling Using a Bladder Pump

The sampling protocol will be as follows for the collection of groundwater samples using a stainless steel/Teflon bladder pump:

1. For wells requiring portable bladder pumps, 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. Many wells have dedicated bladder pumps in the well where the pump has been placed near the middle of the saturated screen.
2. 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.
3. Allow at least 1-foot of water above the inlet so there is little risk of entrainment or air in the sample.
4. 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. Drawdown should not exceed 0.1 meter (4 inches).
5. The discharge during purging and sampling should flow with minimal turbulence or agitation.
6. 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.
7. Record groundwater level frequently until water level stabilization occurs. After stabilization, measure water levels at regular intervals.
8. 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).
9. 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, ORP, 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.

10. Stabilization is achieved after three successive readings are within ± 0.1 for pH, ± 10 mV for ORP, ± 5% for specific conductance, ± 10% for DO, and <10 NTU for turbidity. Temperature will also be measured and recorded, but will not be used as a stabilization parameter. Sampling may begin once the well has stabilized.
11. 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.
12. The pump will not be turned off between the purging and sampling processes.
13. If stabilization does not occur or turbidity is >10 NTU after two hours of purging, the FTL should be contacted for direction.
14. 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 Passive Diffusion Bag Sampler

Select groundwater samples will be collected for VOC analyses using passive diffusion bag (PDB) sampling. 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 (at least two weeks) before retrieval. The sampler water is then decanted into 40 mL volatile organic analysis (VOA) vials and sent to the lab for analysis. Detailed procedures for using PDB samplers in wells can be found in “User’s Guide for Polyethylene-Based Passive Diffusion Bag Samplers to Obtain Volatile Organic Compound Concentrations in Wells” (USGS, 2001). The following is a generalized summary of PDB sampling:

1. The top and bottom of the PDB sampler will be attached to 3/16” polyester or similar non-buoyant rope strong enough to support the weight of the sampler and subject to minimal stretch. The PDB will be suspended within the well screen at selected depths based on the measured total depth and location of the screen. 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.

5.2.3.4 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.

6.0 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.

7.0 QUALITY CONTROL AND QUALITY ASSURANCE

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

8.0 REFERENCES

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

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

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

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

Attachment 4-1

Water Level Measurement and Well Assessment Record

Sample Event: _____

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			

Attachment 4-2

Water Sample Collection Sheet

Site Name:_____ Project No.: _____

Sample No.:_____ Well ID.:_____

Date/Time Collected:_____ Personnel:_____

Sample Method: _____

Sample QC: Duplicate Yes No Duplicate Sample ID: _____

MS/MSD: Yes No

Well Purging Data (Fill In All Blanks)

Depth Of Sample Collection (ft, btoc) _____

Date: _____ Depth To Water (ft, btoc) _____

Time Completed _____ Total Purge _____ Units_____

Field Measurements: Fill In All Blanks

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

Flow Rate_____

General Comments:

_____

_____

STANDARD OPERATING PROCEDURE 5 – VAPOR SAMPLE COLLECTION

Lead Organization: Department of the Army (DA)

Preparing Organization: HDR

SOP Approved by: Field Team Leader: Justin Bills

Project QA Officer: Lynn Lutz

Project Manager: Tom Holmes

1.0 PURPOSE AND SUMMARY

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

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

3.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

Vapor 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.

4.0 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 a vapor sampling will include: a photoionization

detector (PID), a vacuum pump, a set of tedlar bags, 6-liter Summa canisters and Summa canisters wrenches.

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

5.0 PROCEDURE

5.1 Start-Up Activities

5.1.1 Office

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

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

5.1.2 Field

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

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

5.2 Field Operations

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

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

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

5.2.1 Sample Locations

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

5.2.2 Sample Containers

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

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

5.2.3 SVE Wells and System Effluent

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

5.2.3.1 Field Measurements

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

- Connect sampling pump inlet hose to SVE well sample port located on SVE manifold.
- Open appropriate well sample port ball valve.
- Turn on sampling pump and allow it to run for five seconds to purge the pump and tubing.
- Connect tedlar bag to discharge of sampling pump by inserting nipple of bag into pump discharge tube.
- Allow tedlar bag to fill (approximately 20 seconds).
- Once filled, disconnect tedlar bag from sampling pump.
- Close SVE well sample port ball valve.
- Connect calibrated PID Meter to tedlar bag.
- Allow PID Meter to measure VOC concentration. Ensure reading on PID meter stabilizes before recording VOC concentration. This usually takes 10 to 15 seconds.
- Record peak VOC concentration and time.

5.2.3.2 Laboratory Samples

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

- Fill out Summa canister tag with sampling information using a pen with blue or black waterproof ink.
- Remove the Summa canister valve cap.
- Run dedicated tubing from SVE manifold to canister by connecting swagelock.

- Open appropriate SVE well/effluent sample port ball valve.
- Record starting Summa canister pressure on chain-of-custody (COC). The starting Summa canister pressure should be at least -25 inches of mercury (in. Hg) or greater. If not, the canister has leaked and should not be used for sampling.
- Open Summa canister valve located at top of sampling canister.
- Record sampling start time on COC.
- Allow Summa canister to fill until pressure gauge reads -5 in. Hg. (approximately 2 minutes with a 6-liter canister).
- Close sampling port ball valve at SVE well.
- Disconnect Summa canister from regulator.
- Record time of sample collection, date, and Summa canister serial and regulator numbers on COC form.

5.2.4 VMPs

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

Purging:

- Unlock VMP well casing (secured by padlock).
- Attach regulator to "quick connect" on well cap, run line to a "T" connection.
- Run one line out from the "T" to the pump.
- Attach second line to the Summa canister via swagelock.
- Turn on sampling pump and allow lines to purge for approximately five minutes. Purge time is based on tubing diameter and length and is intended to remove three tubing volumes.

Field (PID) Measurements:

- Attach tedlar bag to discharge of sampling pump by inserting nipple of bag into pump discharge tube.
- Allow tedlar bag to fill (approximately 20 seconds).

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

5.2.4.1 Laboratory Samples

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

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

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

5.3 Closeout

5.3.1 Field

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

- Decontaminate all field equipment.
- Ensure all field documentation is completely filled out. This includes the COC and Summa sampling tag. Unless revised by the project manager, standard turn-around time (15 days) will be used. Retain copy of COC for the project file.
- Package Summa canisters in sturdy cardboard boxes with packing material to prevent any potential puncture of the canister. In most cases, the boxes and packing material used by the laboratory to ship the Summa canisters to the site can be reused.
- Ship Summa canisters to laboratory for analysis via Federal Express or other overnight service. Ensure copy of COC is included in shipment.
- Complete logbook, making notations as to site conditions, anomalous readings, etc.
- Ensure that equipment and associated supplies have been shipped back to the office or supplier.
- Ensure that all IDW/trash has been disposed in accordance with the Remedial Action Work Plan (RAWP).

5.3.2 Office

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

- Submit logbook and any original forms to Project/Task Manager for review
- Completion of the Field Event Closeout Report (Attachment 1-2) is not required.

6.0 DATA AND RECORDS MANAGEMENT

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

7.0 QUALITY CONTROL AND QUALITY ASSURANCE

All work will be performed in accordance with the Quality Assurance Project Plan (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.

8.0 REFERENCES

e2M. *Dunn Field Off Depot Groundwater Remedial Action Work Plan, Defense Depot Memphis, Tennessee, Revision 2, WTP-18 Vapor Sample Collection*. April, 2009.

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STANDARD OPERATING PROCEDURE 6 – EBT MIXING AND INJECTING

Lead Organization: Department of the Army (DA)

Preparing Organization: HDR

SOP Approved by: Field Team Leader: Justin Bills

Project QA Officer: Lynn Lutz

Project Manager: Tom Holmes

1.0 PURPOSE AND SUMMARY

This Standard Operation Procedure (SOP) provides guidance for proper mixing and injecting of sodium lactate for enhanced bioremediation treatment (EBT) at Defense Depot Memphis, Tennessee (DDMT). The project work plan must be reviewed for specific requirements.

2.0 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 equipment decontamination include exposure to sodium lactate, hazardous noise, and fluids under pressure. Safety gloves, safety glasses, and hearing protection should be used during mixing and injection procedures.

3.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

Sodium lactate mixing and injections will be directed by the Field Team Leader (FTL), a mid- or senior level environmental professional (engineer, geologist or scientist) with experience in groundwater bioremediation. Injections will be performed by a two-person team consisting of environmental professionals or technicians. The field staff are responsible for following these procedures and seeking direction from the FTL when questions or problems arise.

4.0 EQUIPMENT AND SUPPLIES

The required equipment and supplies will consist of injection trailers, transfer pumps, batch controllers, static water level meters, road cones, and personal protective equipment (PPE).

5.0 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:

- Review injection plan and coordinate injection activities with the PM.
- Confirm availability and condition of DDMT-owned equipment and order additional equipment/supplies for delivery prior to the start of injections.
- Review EBT well location maps and tables showing water level measurements and injection results from previous event.
- 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 sampling activities and assignments with field staff.

5.2 Field Operations

5.2.1 Mixing Procedures

The FTL will be responsible for creating the sodium lactate mixtures in accordance with the injection plan, created by the PM. The injection solution used at DDMT will consist of WILCLEAR PLUS® (a sodium lactate concentrate with a proprietary nutrient blend), water, and Biogreen (aloe vera). To ensure

the batches are made with the correct volumes of lactate and water, the FTL will use the batch controllers in the containment area of the shop.

WILCLEAR PLUS® is stored in 260-gallon totes; any totes being used for active sodium lactate transfer will be kept within the containment area, all other totes will be stored in the adjacent bay. The totes will be moved between bays by forklift, operated by a certified forklift operator.

The injection trailers will be backed into the mixing bay; a spotter will be used while backing up each trailer. The truck's emergency brake will be engaged, and trailer's wheels chocked any time the trailer is parked. Transfer hoses with cam fittings will connect the trailer to the batch controller. A drum pump will be lowered into the tote of WILCLEAR PLUS®.

The FTL will program the batch controller to ensure the target volumes of water and sodium lactate are delivered to each trailer. The target volumes are programed into the controller by first clearing stored information in the controller. This is done by pressing the "Total" key, followed by the "Reset" key. Next, the "Final" key should be pushed, followed by the "Clear" key. Once stored information has been cleared from the controller, the final target volume should be entered into the system. This is done by entering the volume (in gallons) on the key pad, followed by the "Enter" key.

Prior to beginning pumping, the FTL will check to make sure all valves are open and connections are secure. To begin the volume transfer, the "Start" key should be pressed. The pumps will automatically turn off once the target volume is reached. Care should be taken to ensure each sodium lactate tote has at least enough material to reach the target volume for each batch. Additionally, the drum pump should be monitored for signs of overheating. To stop the transfer of either water of sodium lactate, the red "STOP" button should be pressed on the batch controllers.

After the target water and sodium lactate has been delivered to each trailer, 130mL of Biogreen aloe vera will be added to each batch.

The tank, trailer, and injection well numbers will be recorded on the dry erase board attached to each trailer. The FTL will record the mixing volumes, times, and any problems or notes for each tank on the EBT tank mixing form (Attachment 6-1).

5.2.2 Injection Procedures

The field team will conduct the injection procedures per direction from the FTL. Prior to leaving the shop, the generator on the injection trailer will be started and the tank mixer will be started by activating the

mixer switch on the trailer control panel. The tank contents will continue to mix while in transit to the injection well; to ensure a homogenous mixture, each tank should be mixed for a minimum of 10 minutes prior to injections.

Upon arrival at the injection well location, the team will engage the truck's emergency brake and chock the trailer's wheels. Road cones will be placed around the truck and trailer to ensure a safe work area. Additionally, the field team will wear safety glasses, hearing protection, gloves, and high-visibility vests while performing injection activities. A pre-injection static water level reading will be taken at each injection location; this information will be recorded on the EBT injection form. To begin injections, any tubing and pumps will need to be removed from the well and placed on a poly sheet. Once the tubing is removed, a fitting, equipped with a pressure gauge and camlocks, will be attached the well. The injection hose will then be connected to the open end of the pressure gauge fitting. For 2-inch injection wells, a threaded male cam-lock adapter will need to be attached to the top of the casing before attaching the pressure gauge fitting and injection hose; the 4-inch injection wells have the cam-lock fitting installed. Next, prior to activating the injection pump, the valves along the injection hose will need to be opened. Once all fittings are secure and valves open, the pump should be engaged by turning the pump switch on the trailer control box to "On".

The field team is responsible for continuously monitoring injection pressures and flow rates. Injection pressures should not exceed 30 pounds per square inch (psi), as indicated on the pressure gauge fitting. In the event that high injection pressures are observed, the flow rate should be throttled back by slowly closing off the valve beneath the control panel. Once a stable injection pressure is reached, the valve should remain in the throttled back position. The injections will continue until the entire volume of each trailer is emptied into the well. If tubing was removed from the well, the tubing should be replaced prior to leaving the injection location. Injection volume, time, flow rate (gallons per minute), and any changes in injection pressure will be recorded on the EBT injection form (Attachment 6-2). An injection summary, including well ID, start and end times, and total volume injected, will be noted in the field log book.

Occasionally, some injection wells experience high sustained injection pressures (~30psi), despite low flow rates. In these cases, efforts should be made to ensure the target injection volume is delivered to each well; varying flow rates, pausing injections and allowing injection pressures to stabilize are techniques that can help deliver target volumes. If, after two hours, the well is no longer able to receive additional injection mixture, the remaining lactate solution will be injected in an adjacent well to be determined by the FTL.

5.3 CLOSEOUT

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

- Clean and check condition of the injection trailer.
- Provide log books and injection forms to FTL for review.
- Properly dispose of trash, debris and used PPE.
- Clean up spills of sodium lactate concentrate and add recovered liquids to the purge water storage tanks at Dunn Field.
- Update the injection summary spreadsheet and complete the Daily Field Report (SOP 1, Attachment 1-2) and submit to PM.

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

6.0 DATA AND RECORDS MANAGEMENT

All field forms and log book entries will be scanned and copied to 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.

7.0 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.

8.0 REFERENCES

Main Installation Remedial Action Enhanced Bioremediation Treatment Operations and Maintenance Manual (e2M, 2007)

*Remedial Action Work Plan Addendum, Main Installation Defense Depot Memphis, Tennessee (*HDR, 2012).

Attachment 6-1

TABLE C-1
EBT TANK MIXING FORM
RAWP ADDENDUM
Main Installation - Defense Depot Memphis, Tennessee

Site Name: _____ Injection Event: _____ Project No.: _____

Date: _____

Tank No: _____ for Area: _____ Injection Wells: _____

Target Lactate Concentration (by vol): _____

Lactate Type (circle): Wilclear™ or Wilclear Plus™ Other: _____

Concentrate/Water Addition Time: Start: _____ Finish: _____

Mixing Fluid Volumes (gal) - Concentrate: _____ Water: _____

Fluid Mixing Time: Start: _____ Finish: _____

Mixing Personnel: _____

Comments: _____

_____

_____

Form Completed by: _____ _____
Signature **Date**

Attachment 6-2

TABLE C-2
EBT INJECTION FORM
RAWP ADDENDUM
Main Installation - Defense Depot Memphis, Tennessee

Site Name: _____ Injection Event: _____ Project No.: _____

Well ID: _____ Area: _____ Tank No: _____

Target Injection Volume: _____ (gal) Lactate Concentration: _____

Lactate Type (circle): Wilclear™ or Wilclear Plus™ Other: _____

Pre-Injection Groundwater Depth: _____ (ft, btoc) Date/Time: _____

Injection Measurements Date: _____

Time: Start: _____ Mid-point: _____ Finish: _____

Duration (min): _____

Injection Flow Rate (gpm): Start: _____ Mid-point _____ Finish _____

Injection Pressure (psi): Start: _____ Mid-point _____ Finish: _____

Note: Take initial reading about 2 minutes after start, mid-point when half volume injected, and final reading prior to shutting down pump.

Variations in Flow Rate, Pressure or Amperage: _____

Total Volume of Injection Fluid Delivered to Well (gal): _____ (Note below if not total volume planned)

Injection Personnel: _____

Comments: _____

Average Flow Rate _____ (gpm)

Form Completed by: _____ _____
Signature **Date**

STANDARD OPERATING PROCEDURE 7 – SAMPLE CONTROL AND DOCUMENTATION

Lead Organization: Department of the Army (DA)

Preparing Organization: HDR

SOP Approved by: Field Team Leader: Justin Bills

Project QA Officer: Lynn Lutz

Project Manager: Tom Holmes

1.0 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.

2.0 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 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.

3.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

Sample control 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. The field staff, environmental professionals or technicians, are responsible for proper sample handling and documentation of the sample collection.

4.0 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.

5.0 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 the samples are being collected and a hand written COC will be used, all information should be recorded in a log book as to the type of sample, date and time collected and number of sample containers. The COC can then be filled out back at the field office in a quiet environment with out disturbances to avoid errors. 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 Section 2.3.2 of 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 observing field activities.

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 Z-Drive.

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, but the "relinquished to" box will not be filled in.

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

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

Custody Seals: Custody seals will be preprinted, adhesive-backed seals designed to break if disturbed. Sample shipping containers (coolers, cardboard boxes, etc., as appropriate) will be sealed in as many places as necessary to ensure security. 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.

6.0 DATA AND RECORDS MANAGEMENT

All field forms, COCs, and log book entries will be scanned and copied project folder on the "Z" drive 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.

7.0 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.

8.0 REFERENCES

MACTEC *RA SAP Volume I: Field Sampling Plan, Defense Depot Memphis, Tennessee, Revision 1*, *WTP-7 Sample Control and Documentation*. November, 2005.

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

Attachment 7-1

EXAMPLE SAMPLE PLAN DETAIL

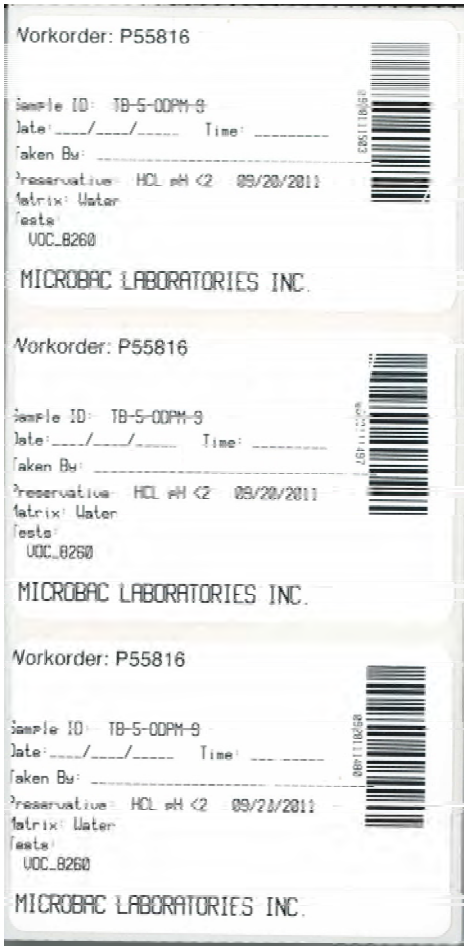
SAMPLING PLAN DETAIL (OFF DEPOT PM WELLS September 2011) - ODPM-9

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

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

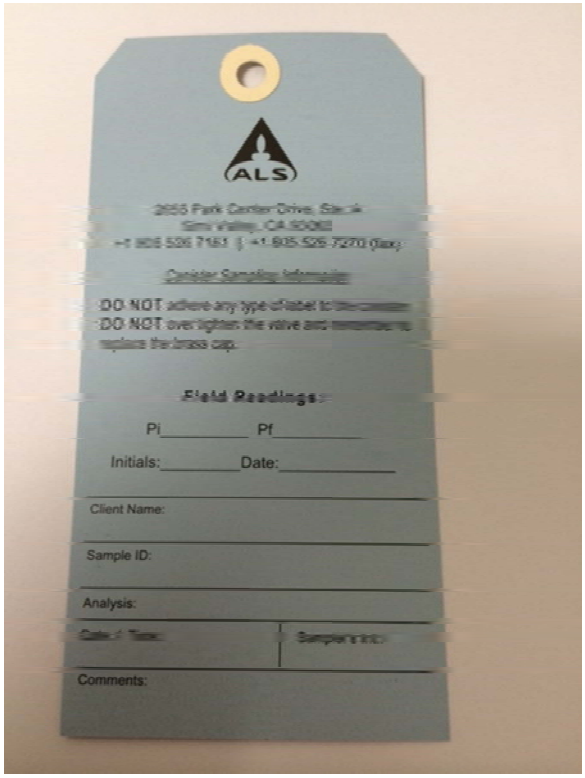
Attachment 7-2

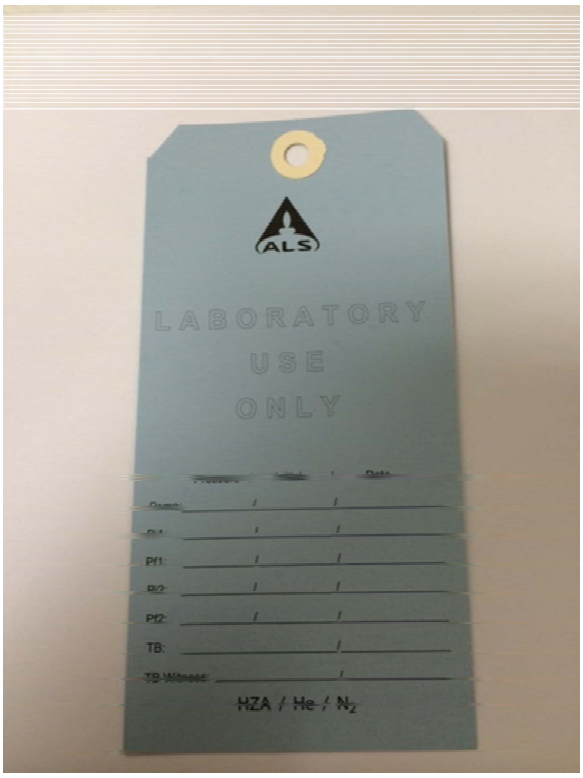
EXAMPLE SAMPLE LABELS FOR GROUNDWATER SAMPLES



Attachment 7-3

EXAMPLE SAMPLE LABELS FOR AIR SAMPLES





Attachment 7-4

EXAMPLE MICROBAC CHAIN OF CUSTODY FORM (COMPUTER)

Microbac

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

Barcode	Client ID	Tests	Collect Date	Beg. Depth	End. Depth	Notes
~~0420111~~	~~MW-91-ODLB-3~~	~~VOC_8260~~	~~04/25/2011 10:09~~	9J 4/26		
~~0420112~~	~~MW-91-ODLB-3~~	~~VOC_8260~~	~~04/25/2011 10:09~~	9J 4/26		
~~0420113~~	~~MW-91-ODLB-3~~	~~VOC_8260~~	~~04/25/2011 10:09~~	9J 4/26		
0420111	04/25/11-TB-1-ODPM-8	VOC_8260	04/25/2011 10:09			
0420112	04/25/11-TB-1-ODPM-8	VOC_8260	04/25/2011 10:09			
0420113	04/25/11-TB-1-ODPM-8	VOC_8260	04/25/2011 10:09			
0420114	DUP-1-ODPM-8	VOC_8260	04/25/2011 11:32			
0420115	DUP-1-ODPM-8	VOC_8260	04/25/2011 11:32			
0420116	DUP-1-ODPM-8	VOC_8260	04/25/2011 11:32			
0420117	MW-250-ODPM-8	VOC_8260	04/25/2011 10:30			
0420118	MW-250-ODPM-8	VOC_8260	04/25/2011 10:30			
0420119	MW-250-ODPM-8	VOC_8260	04/25/2011 10:30			
04201110	MW-251-ODPM-8	VOC_8260	04/25/2011 10:42			
04201111	MW-251-ODPM-8	VOC_8260	04/25/2011 10:42			
04201112	MW-251-ODPM-8	VOC_8260	04/25/2011 10:42			
04201113	MW-54-ODPM-8	VOC_8260	04/25/2011 11:32			
04201114	MW-54-ODPM-8	VOC_8260	04/25/2011 11:32			
04201115	MW-54-ODPM-8	VOC_8260	04/25/2011 11:32			
04201116	MW-70-ODPM-8	VOC_8260	04/25/2011 13:23			
04201117	MW-70-ODPM-8	VOC_8260	04/25/2011 13:23			
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			
~~04201123~~	~~MW-70-ODPM-8-MSD~~	~~VOC_8260~~	~~04/25/2011 13:23~~	KS 4/26		
04201125	MW-76-ODPM-8	VOC_8260	04/25/2011 13:07			
04201126	MW-76-ODPM-8	VOC_8260	04/25/2011 13:07			
04201127	MW-76-ODPM-8	VOC_8260	04/25/2011 13:07			
04201128	MW-77-ODPM-8	VOC_8260	04/25/2011 13:14			
04201129	MW-77-ODPM-8	VOC_8260	04/25/2011 13:14			
04201130	MW-77-ODPM-8	VOC_8260	04/25/2011 13:14			
04201131	MW-79-ODPM-8	VOC_8260	04/25/2011 11:17			
04201132	MW-79-ODPM-8	VOC_8260	04/25/2011 11:17			

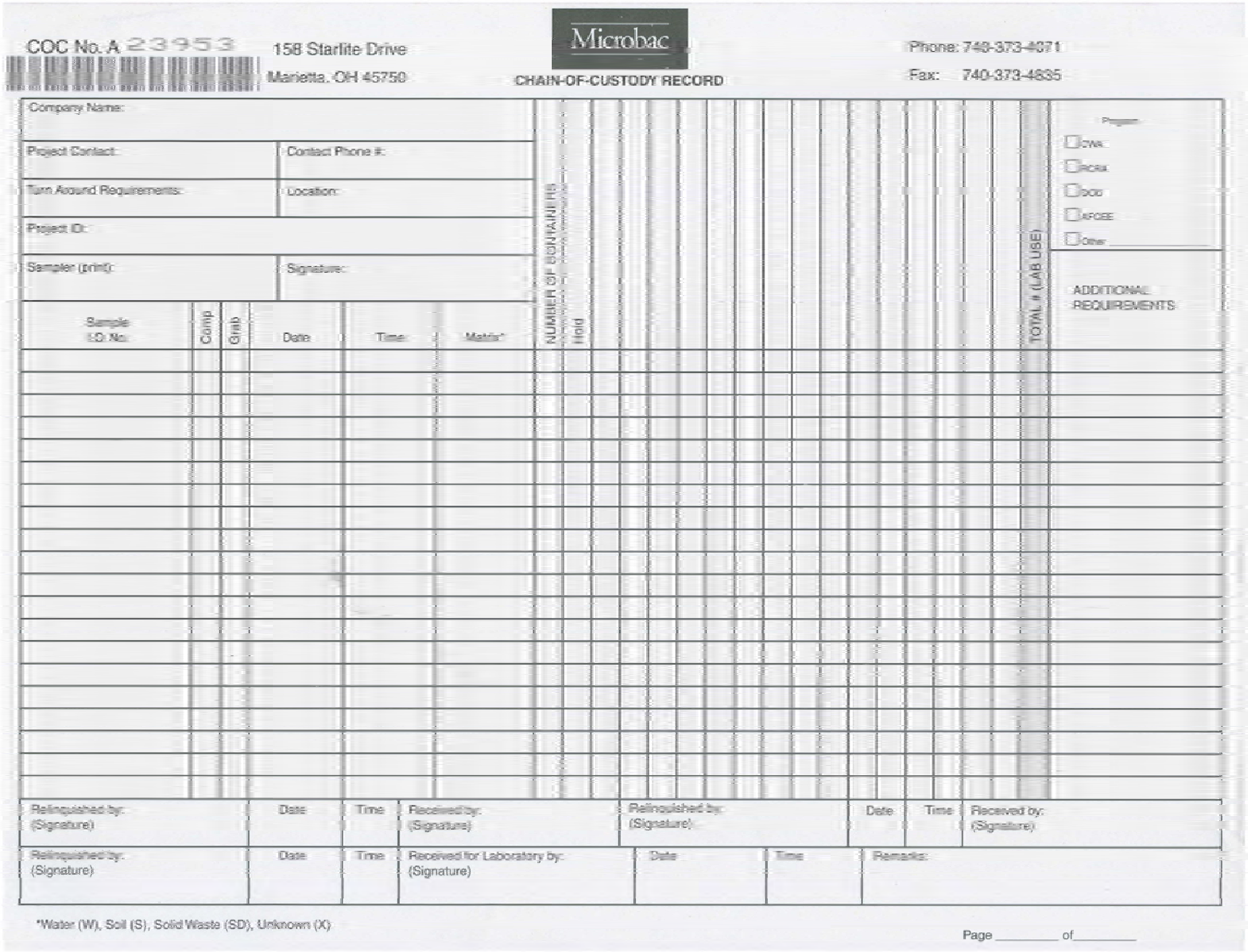
Microbac OVD
Received: 04/27/2011 12:55
By: BRENDA GREENWALT
(signed)
221000014398

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

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

Attachment 7-4

EXAMPLE MICROBAC CHAIN OF CUSTODY FORM (HAND)



COC No. A 23953

158 Starlite Drive
Marietta, OH 45750

Microbac

CHAIN-OF-CUSTODY RECORD

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

Company Name:

Project Contact: | Contact Phone #:

Turn Around Requirements: | Location:

Project ID:

Sampler (print): | Signature:

Program: ☐ CWA ☐ RCRA ☐ DOD ☐ NPDES ☐ Other

ADDITIONAL REQUIRMENTS

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

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

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

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

Page _____ of _____

Attachment 7-5

EXAMPLE ALS CHAIN OF CUSTODY FORM

Air - Chain of Custody Record & Analytical Service Request Page _____ of _____

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

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

ALS Project No.

Company Name & Address (Reporting Information)

Project Name

Project Number

ALS Contact:

Analysis Method

Project Manager

P.O. # / Billing Information

Phone | Fax

Email Address for Result Reporting

Sampler (Print & Sign)

Comments e.g. Actual Preservative or specific instructions

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

Report Tier Levels - please select

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

EDD required Yes / No
Type: _____ Units: _____

Chain of Custody Seal: (Circle)
INTACT BROKEN ABSENT

Project Requirements (MRLs, QAPP)

Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:	
Relinquished by: (Signature)	Date:	Time:	Received by: (Signature)	Date:	Time:	Cooler / Blank Temperature ____°C

STANDARD OPERATING PROCEDURE 8 – SAMPLE PACKING AND SHIPPING

Lead Organization: Department of the Army (DA)

Preparing Organization: HDR

SOP Approved by: Field Team Leader: Justin Bills

Project QA Officer: Lynn Lutz

Project Manager: Tom Holmes

1.0 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.

2.0 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 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.

3.0 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.

4.0 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.

5.0 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 6-1).

6.0 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.

7.0 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.

8.0 REFERENCES

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

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

Attachment 8-1

SAMPLE HANDLING, PACKING & SHIPPING CHECKLIST

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

Sample Event: _____

Date: _____

PROJECT SAMPLES

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

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

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

DOCUMENTS

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

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

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

□ **Custody seals** placed on the front and back of each cooler.

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: Justin Bills

Project QA Officer: Lynn Lutz

Project Manager: Tom Holmes

1.0 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.

2.0 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 Health and Safety Plan (HASP) for the specific project activity 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.

3.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

Sampling equipment decontamination and rinsate sample collection will be directed by the Field Team Leader (FTL), a mid- or senior level 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.

4.0 EQUIPMENT AND SUPPLIES

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

5.0 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 Remedial Action Operations (RA-O) and Long-Term Monitoring (LTM) activities.

Decontamination procedures will be evaluated by the collection of equipment rinsates. 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

Tap water from the municipal water treatment 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 shop water hose 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 workplan.

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

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

5.2.4 Equipment Rinsate Collection

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

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

The equipment rinsate protocol will be as follows:

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

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

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

5.3 Closeout

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

- Confirm all equipment is decontaminated and properly stored all equipment.

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

6.0 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.

7.0 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.

8.0 REFERENCES

MACTEC *RA SAP Volume I: Field Sampling Plan, Defense Depot Memphis, Tennessee, Revision 1*, *WTP-10 Sampling Equipment Decontamination*. November, 2005.

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

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

Lead Organization: Department of the Army (DA)

Preparing Organization: HDR

SOP Approved by: Project QA Officer: Lynn Lutz

Project Manager: Tom Holmes

1.0 PURPOSE AND SUMMARY

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

2.0 HEALTH AND SAFETY

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

3.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

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

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

4.0 EQUIPMENT AND SUPPLIES

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

5.0 PROCEDURE

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

To maintain comparability among data sets for the entire DDMT project, the data validation guidelines in Appendix E, Data Quality Evaluation SOPs, of the previous version of the DDMT Quality Assurance Project Plan (QAPP) (MACTEC, 2005) and the United States Environmental Protection Agency (USEPA) Functional Guidelines (USEPA, 2008 [for organics] and 2010 [for inorganics]) have been incorporated herein.

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

Final qualifiers will be:

- No qualification
- Non-detect (U)
- Detected and estimated (J)
- Non-detect and estimated (UJ)
- Blank contamination (B)
- Rejected (R)

5.1 Chain-of-Custody

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

5.2 Sample Receipt

5.2.1 Water Samples

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

5.2.2 Air Samples

Air samples have no temperature requirements.

5.3 Holding Times and Preservation

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

5.4 Method Identification, Analyte List, and RLs/MDLs

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

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

5.5 Gas Chromatography/Mass SpectrometryTuning and Analytical Sequence

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

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

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

5.6 Initial Calibration

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

5.6.1 Water Samples

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

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

Poor Responders		
Acetone	Chloroethane	1,2-Dibromoethane (EDB)
2-Butanone	Chloromethane	1,2-Dibromo-3-chloropropane
2-Hexanone	Dichlorodifluoromethane	cis-1,2-Dichloroethene
4-Methyl-2-pentanone	Trichlorofluoromethane	trans-1,2-Dichloroethene
Carbon disulfide	Methyl tert-butyl ether (MTBE)	1,2-Dichloropropane
	Isopropylbenzene	Methylene chloride

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

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

5.6.2 Air Samples

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

5.7 Initial Calibration Verification (Second Source Standard)

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

5.7.1 Water Samples

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

5.7.2 Air Samples

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

5.8 Continuing Calibrations

5.8.1 Water Samples

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

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

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

5.8.2 Air Samples

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

5.9 Blanks

5.9.1 Method Blank

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

Analytes detected in the samples as well as the method blank will be qualified with a B if the sample concentration was less than five times the blank concentration (10 times for the common lab contaminants acetone, 2-butanone and methylene chloride). Blank results will not be subtracted from sample results. If the sample concentration was greater than five times (or 10 times) the blank concentration, the blank is not considered to have greatly affected sample concentration, and sample results will not be qualified.

5.9.2 Trip Blank

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

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

5.9.3 Rinsate Blank

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

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

5.10 Laboratory Control Sample and Duplicate (LCS/LCSD)

There must be an LCS associated with each sample. There may also be an LCSD, although this is not required. LCSs must be spiked with all COCs.

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

5.11 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

MS/MSD samples will be indicated on the COC. MS/MSD samples must be spiked with all COCs.

5.11.1 Water Samples

One set of MS/MSD samples will be collected for every 20 field samples. Recovery limits are the lab's in-house control limits. Analytes with higher recoveries may be biased high and will be qualified as estimated (J) when detected; non-detect results will not be qualified. Analytes with lower recoveries may be biased low and will be qualified as estimated (J) or non-detect estimated (UJ). Analytes with RPD values greater than 30% will be qualified as estimated (J) when detected; non-detect results will not be qualified. Only the parent sample will be qualified.

5.11.2 Air Samples

MS/MSD samples are not collected for air samples.

5.12 Field Duplicates

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

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

5.13 Laboratory Duplicates

5.13.1 Water Samples

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

5.13.2 Air Samples

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

5.14 Surrogates

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

Surrogates with recoveries above the control limits may indicate a high bias in detected sample results; all detected analytes in the sample will be qualified as estimated (J). Surrogates with recoveries below control limits may indicate a low bias in sample results; all analytes in the sample will be qualified as estimated (J) or non-detect estimated (UJ).

5.15 Internal Standards

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

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

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

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

5.16 Usability Assessment

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

6.0 DATA AND RECORDS MANAGEMENT

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

6.1 Data Files from Laboratories

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

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

6.2 Data Files from HDR

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

6.3 Data Files at DSA

DSA will perform data review (the three steps described in section 3.0 of this SOP) and write a report that summarizes what was reviewed, what decisions were made, and what qualifiers were applied to the data. DSA's report format (redacted for confidentiality and to omit sections related to inorganic samples) also serves as their SOP and is located in Attachment 9-1 of this SOP.

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

6.4 Data Files from DSA to HDR

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

6.5 Data Files at HDR

The PC will save the report and EDD file to the appropriate folders on the HDR network Z: drive. The PC will make a copy of the EDD file, with the same name plus "-final". The PC will open the "final" file, add a field named "Final_Qualifier" at the end of the fields, and record final qualifiers. Qualifiers for undetected results (U) do not all need to be copied to this field, unless the final qualifier is UJ or B or R, which will be entered into this field.

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

7.0 QUALITY CONTROL AND QUALITY ASSURANCE

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

8.0 REFERENCES

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

United States Environmental Protection Agency (USEPA), 2008. *USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review*. June 2008.

United States Environmental Protection Agency (USEPA), 2008. *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review*. January 2010.

Attachment 10-1

EXAMPLE DSA ORGANIC DATA QUALITY REVIEW REPORT – WATER SAMPLES

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

SDG _____

PROJECT Memphis Defense Depot, LS-13 Sampling for HDR, Denver

LABORATORY _____

SAMPLE MATRIX Water SAMPLING DATE _____

NUMBER OF SAMPLES 77, including 6 trip blanks, 1 rinse blank, and 7 field duplicates

ANALYSES REQUESTED SW-846 8260B (VOA by GC/MS)

SAMPLE NUMBER See result forms attached and associated EDD

DATA REVIEWER: _____

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

Telephone Logs included Yes____ No __X_

Contractual Violations Yes____ No __X_

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

Attachment 10-1

I. DELIVERABLES

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

Yes __X__ No _____

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

B. Chain of Custody documentation was complete and accurate.

Yes __X__ No _____

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

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

Yes __X__ No _____

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

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

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

II. ANALYTICAL REPORT FORMS

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

Yes __X__ No _____

B. Holding Times

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

Yes __X__ No _____

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

Yes __X__ No _____

III. INSTRUMENT CALIBRATION – GC/MS

A. Initial Calibration

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

Yes __X__ No _____ NA _____

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

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

Attachment 10-1

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

Yes __X__ No _____ NA _____

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

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

Yes __X__ No _____ NA _____

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

Yes __X__ No _____ NA _____

B. Continuing Calibrations

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

Yes __X__ No _____ NA _____

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

Yes _____ No __X__ NA _____

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

The QAPP indicates that compounds in a run should be rejected if the % D is > 40%. We interpret this to mean that non-detects should be rejected and that detected compounds should be "J" qualified, which is the normal validation process for rejection. Professional judgment is that high bias CCVs with a % D greater than

40% should not be rejected for non-detects if the response factors are sufficient to ensure verification of the non-detect. Non-detects with % D values > 40% are qualified "RC#", where # is the % D.

The table below shows the outliers observed in CCVs for this report.

SDG	Batch	Analyte	% D	Bias	Qualifier
L110xxxxx	WGxxx	Bromomethane	27.5	high	None, ND
	WGyyy	Bromomethane	33.6	high	None, ND

IV. GC/MS INSTRUMENT PERFORMANCE CHECK

The BFB (VOA) performance check was injected once at the beginning of each 12-hour period and relative abundance criteria for the ions were met.

Yes __X__ No _____ NA _____

V. INTERNAL STANDARDS

The Internal Standards met the 100% upper and -50% lower limits criteria, and the Retention times were within the required windows.

Yes __X__ No _____ NA _____

VI. SURROGATE STANDARDS

Surrogate spikes were analyzed with every sample.

Yes __X__ No _____

And met the recovery limits defined in the QAPP of 70 – 130% for VOA water or 75 – 125% for soil samples.

Yes __X__ No _____

VII. MATRIX SPIKE/MATRIX SPIKE DUPLICATE

A. Matrix Spike (MS) and Matrix Spike Duplicate (MSD) were analyzed for every analysis performed and for every 20 samples or for every matrix whichever is more frequent.

Yes __X__ No _____

There are eight MS/MSD samples associated with the 77 non-QC client samples. This meets the recommendation of 1 per 20 field samples. As this is an ongoing project an overall adherence to the 1:20 frequency is monitored by the Project Manager.

B. The MS and MSD percent recoveries were within the limits defined in the QAPP of VOA at 70 – 130% with five compounds allowed to be within 60 – 140%.

Yes _____ No __X__ NA _____

The full target list has been spiked. Qualifiers are added for all outliers as described here. Please refer to the project EDD for a detailed list of qualifiers added.

The table below shows the outliers observed in MS/MSD samples for this report. Results have been qualified per the QAPP as "JMS#", where # is the % recovery. Data could be biased high or low in approximate proportion to the spike recovery. Only detected data are qualified for high recovery. Data are not qualified if four times (4x noted in table) =the spike amount is less than the amount in the parent sample. Only the parent sample is qualified.

SDG	Parent Sample	Analyte	MS	MSD	Bias	Qualifier
L110xxxxx	XXX	Carbon disulfide	124	132	high	None, ND

C. The MSD relative percent differences (RPD) were within the defined contract limits for VOA of 30% water or 40% soil, with five compounds allowed to be > 40% RPD.

Yes __X__ No _____ NA _____

Qualifiers are added only when the MS or MSD recovery is also out of limits. Data are qualified "JD#", where # is the RPD. As the RPD increases, the matrix precision decreases.

D. The MS/MSD were client samples.

Yes __X__ No _____ NA _____

Attachment 10-1

VIII. LABORATORY CONTROL SAMPLE

A. Laboratory Control Sample (LCS) was analyzed for every analysis performed and for every 20 samples.

Yes __X__ No _____

B. The LCS percent recoveries were within the limits defined in the QAPP for VOA of 80 – 120% for water or 75 – 125% for soil. Five compounds are allowed to be 60 – 140%. If an LCS and LCSD are analyzed, both samples must have the same compounds out for data to be qualified.

Yes _____ No __X__

The full target list has been spiked. When a high LCS recovery is associated with a non-detect in samples, no qualifier is added since the indicated bias is high. When the target is detected, data are qualified "JL#", where # is the recovery. Data could be biased high proportional to the LCS percent recovery. All results associated with low recoveries are qualified, and data could be biased low.

The table below shows the outliers and the limits applied per the QAPP. The limits are specified based on the matrix. Qualifiers are added for all outliers as described here, but the Project Manager may consider reversing some of these when the limits fall within the marginal exceedance limits (60 – 140%). Please refer to the project EDD for a detailed list of qualifiers added.

SDG	Batch	Analyte	% Recovery (LCS/LCSD)	Bias	Qualifier
L110xxxxx/ L110xxxxy	WGxxx	Dichlorodifluoromethane	129	high	None, ND

IX. BLANKS

A. Method Blanks were analyzed at the required frequency and for each matrix and analysis.

Yes __X__ No _____

B. No blank contamination was found in the Method Blank.

Yes __X__ No _____

C. If Field Blanks were identified, no blank contamination was found.

Yes _____ No __X__ NA ____

When analytes are present in both the Field Blank and the associated samples, the results in the samples are qualified in the same manner as for Method Blanks. For clarity, the qualifiers used in this case are "UTB#" for Trip Blanks or "UFB#" for Rinse Blanks, where # is the associated blank value. Results added are shown in the table below. No qualifiers have been applied.

SDG	Sample	Analyte	Result	Qualifier
L110xxxxx	XXX	Methylene Chloride	0.538	None, ND

X. FIELD QC

If Field Duplicates were identified, they met guidance for VOA of RPD of < 30% for water or < 50% for soil. For values reported at < 5 × the reporting limit (RL), a difference of 2 × RL for water or 3.5 × RL for soil samples is used as guidance. This is referred to as the CRDL Rule. Data are not qualified for Field Duplicates as these are evaluated for the total project by the Project Manager.

Yes _____ No __X__ NA _____

There are xxx identified field duplicates as described in the following table.

SDG	Parent Sample	Field Duplicate	Observations
L110xxxxx	XXXMW-21-LS-13	DUP-1	OK

XI. SYSTEM PERFORMANCE

A. The RICs, chromatograms, tunes and general system performance were acceptable for all instruments and analytical systems.

Yes _____ No _____ NA __X__

Not part of this review level.

B. The suggested EQLs for the sample matrices in this set were met.

Yes __X__ No _____ NA _____

XII. TCL COMPOUNDS

A. The identification is accurate and all retention times, library spectra and reconstructed ion chromatograms (RIC) were evaluated for all detected compounds.

Yes _____ No _____ NA __X__

Not part of this review level.

B. Quantitation was checked to determine the accuracy of calculations for representative compounds in each internal standards quantitation set.

Yes _____ No _____ NA __X__

Not part of this review level.

XIII. TENTATIVELY IDENTIFIED COMPOUNDS

TICs were properly identified and met the library identification criteria.

Yes _____ No _____ NA __X__

Not part of this review level.

XIV. OVERALL ASSESSMENT OF THE CASE

The laboratory has complied with the requested method. Data are fully usable after consideration of qualifiers. The following is noted:

<u>Sample Preservation</u>

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

SDG L110xxxxx: The laboratory notes that XXX samples had headspace > 6 mm.

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

Continuing Calibrations

See the table within the body of this report. When there are no detections, no qualification is required unless the % D is biased low and so large as to indicate a significant probability of false negatives. Qualification is required for a % D biased high for detected compounds only. This requires that the RF is acceptable to verify the non-detect status, which is the case here. No qualifiers have been applied.

Matrix Spikes

There are eight MS/MSD samples associated with the 77 non-QC client samples.

See the table within the body of this report. Results have been qualified per the QAPP as "JMS#", where # is the % recovery. Data could be biased high or low in approximate proportion to the spike recovery. Only detected data are qualified for high recovery. Data are not qualified if four times the spike amount is less than the amount in the parent sample. Only the parent sample is qualified. Data have been qualified with a low bias only for cis-1,2-dichloroethene in samples XXXX.

Laboratory Control Samples

See the table within the body of this report. When a high LCS recovery is associated with a non-detect in samples, no qualifier is added since the indicated bias is high. When the target is detected, data are qualified "JL#", where # is the recovery. Data could be biased high proportional to the LCS percent recovery. Data for vinyl chloride and carbon tetrachloride have been qualified for slightly high recoveries.

Field Blanks

See the table within the body of this report. When analytes are present in both the Field Blank and the associated samples, the results in the samples are qualified in the same manner as for Method Blanks. For clarity, the qualifiers used in this case are "UTB#" for Trip Blanks or "UFB#" for Rinse Blanks, where # is the associated blank value. No data have required qualification.

Field QC

There are seven identified Field Duplicates. See the table within the body of this report. No data have been qualified.

Attachment 10-2

EXAMPLE DSA ORGANIC DATA QUALITY REVIEW REPORT – AIR SAMPLES

ORGANIC AIR QUALITY REPORT

METHOD TO-15

SDG: _____

PROJECT: Memphis Defense Depot DDMT Fluvial soil vapor extraction for HDR Inc. (formerly e2m)_

LABORATORY: Microbac, subcontracted to Columbia Analytical Services Laboratories, CA_____

SAMPLE MATRIX: Air_____

SAMPLING DATE (Month/Year): March, 2011_____

NUMBER OF SAMPLES: 9 samples including one field duplicate_____

ANALYSES REQUESTED: Summa Canister VOA TO-15_____

SAMPLE NO.: See project EDD for sample IDs_____

DATA REVIEWER: Diane Short_____

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

Telephone Logs included Yes____ No __X__

Contractual Violations Yes____ No __X_

The EPA CLP National Functional Guidelines for Organic Data Review, 2001 (SOP), EPA Method TO-15 current updates have been referenced by the reviewer to perform this data validation review. The EPA qualifiers have been expanded to include a descriptor code and value to define QC violations and their values, per the approval of the HDR/e2m Project Manager. Per the Scope of Work, the review of these samples includes validation of all QC forms and submitted calibrations referencing the QC limits in the above documents.

Attachment 10-2

DELIVERABLES

All deliverables were present as specified in the Statement of Work (SOW) or in the project contract.

Yes _X__ No ____

Note an extended list of volatile compounds was reported. Level III data packages were submitted and Level III validation was performed for holding times, chain of custody, calibrations and QC.

II. ANALYTICAL REPORT FORMS

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

B. Holding Times
The contract holding times were met for all analyses (Time of sample receipt to time of analysis (VOA) or extraction and from extraction to analysis). Contract holding times for TO-15 canisters is 30 days from date of collection.

Yes ___X__ No____

C. Chains of Custody

Chains of Custody were present and were complete with signatures, sign-offs and complete entry of data. Canisters were properly sampled and received.

Yes __X___ No ____

The project manager is informed of the following and the project record is being updated.

There are gaps from relinquishment to sample receipt. The courier is identified as FedEx and there is no airbill number on the chain or log-in. The client notes that the coolers are often sealed before the airbill number is known.

D. Canister Pressure
Canister pressures were measured and recorded for initial vacuum check, initial field vacuum, final field reading, lab initial pressure and final pressure.

Yes ___X___ No _____ NA _____

Attachment 10-2

Pressures were reported for the field initial and final pressures and the laboratory final pressure. The pressure changes are not as large as for some other samplings, but these are highly contaminated samples most of which have required some dilutions even at the reported volume.

All readings met the limits or exceptions were noted and pressure corrected

Yes ___X__ No ____ NA ____

III. INSTRUMENT CALIBRATION

A. Initial Calibration – GC/MS
1. The Relative Response Factors (RRF) and average RRF for all compounds for all analyses met the required criteria.

Yes __X__ No____ NA____

Minimum response factors are not defined by the method but meet routine Method 8260 limits. This method does not involve purging water samples. Consequently, all targets, including the typically poor-purging compounds, normally have response factors that are acceptable per validation criteria for volatiles.

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

Yes ___X__ No ____

B. Continuing Calibration – GC/MS
1. The RRF standard was analyzed for each analysis at the required frequency and the QC criteria were met

Yes __X__ No____ NA____

Minimum response factors are not defined by the method, but met validation guidance. There were 3 calibrations as there were 2 days of analysis for dilutions.

2. The percent difference (%D) limits of 30% were met.

Yes __X__ No ____

Outliers were not client compounds.

IV. GC/MS INSTRUMENT PERFORMANCE CHECK

Attachment 10-2

A. The BFB performance check was injected once at the beginning of each 12-hour period and relative abundance criteria for the ions were met.
Yes ___X__ No____ NA ____

Tunes were provided and were acceptable.

V. INTERNAL STANDARDS

A. Area Limits
The Internal Standards met the 100% upper and -50% lower limits criteria and the Retention times were within the required windows.

Yes __X___ No____ NA ____

B. Retention Times
The relative retention times of the internal standards and sample compounds met the ± 0.06 RRT units limit.

Yes __ X ___ No____ NA ____

VI. SURROGATE

Surrogate spikes were analyzed with every sample.

Yes __X___ No ____

Note that three surrogates are used. Method 8260 requires 3 surrogates, but one is acceptable for TO-15.

And met the recovery limits defined in the current contract

Yes __ X _ No ____

VII. MATRIX SPIKE/MATRIX SPIKE DUPLICATE

A. Matrix spike (MS) and matrix spike duplicates (MSD) were analyzed for every analysis performed and for every 20 samples or for every matrix whichever is more frequent.

Yes____ No ____NA ___X__

Spikes are not amenable to canister analysis and are not required. Laboratory duplicates are required and are provided by the laboratory. See below.

B. The laboratory duplicate relative percent differences (RPD) were within the defined contract limits. Method requirements are 25% maximum RPD.

Yes ____ No ____ NA__X_

For validation purposes, only results > 5x PQL are qualified for RPD outliers. For results < 5x PQL, results are qualified if the absolute difference is greater than 2x PQL. The qualifier added is JD#, where # is the RPD or the absolute difference observed, as appropriate.

The laboratory duplicates was sample FSVE-SVEC-2Q11-NS. RPDs were reported above the limit, but these were low level results that met the 2 x PQL criteria.

VIII. DUPLICATE CONTROL SAMPLES

A. Duplicate Control and Duplicate Control Sample Duplicates similar to Laboratory Control Samples (LCS) were performed for every set.

Yes _X_ No ___

B. And percent recoveries were acceptable at 70 – 130%.

Yes _X___ No ____

For air data, the laboratory limits are used as there are no air limits defined in the QAPP. There was an LCS reported for all 3 days of analysis.

C. And Relative Percent Differences were within lab limits.

Yes ___ No____ NA__X__

LCSDs have not been performed, and are not required by the method.

IX. SHIFT CHECKS

Shift checks were performed and were within time limits.

Yes __X__ No____

X. BLANKS

Attachment 10-2

A. Method Blanks were analyzed at the required frequency and for each matrix and analysis.

Yes __X___ No____

This is a nitrogen blank run with each set.

B. The method blank was free of contamination.

Yes _____ No ___X__

Methylene chloride was reported in all 3 blanks

4/26/11 at 0.14 ug/m3; 4/28/11 at 0.16 ug/m3; 4/29 at 0.15 ug/m3.

The samples have been diluted for most reported results. The blank is multiplied by a factor determined from the reported MRL for each sample/compound. See the EDD and table at the end of the report for the final blank value as data are qualified BMB#, where # is the blank corrected value. Only data that are less than 10 x the blank (corrected for dilution) are qualified.

C. If Field Blanks were identified, they were free of contamination.

Yes _____ No ____ NA ___X___

There were no field blanks identified.

D. Contamination level was less than 0.03 mg/cubic meter before samples were analyzed per the method.
Yes __X__ No____ NA _____

Reporting units include both ppbv and ug/m3.

XI. FIELD QC

A. If Field duplicates or Performance Check Compounds were identified, they met the RPD or % recovery criteria for the project.

Yes ___ No __X__ NA___

One field duplicate pair is reported: FSVE-SVEG-2Q11: -NS and DUP. These are regularly sampled locations and the precision is built in as the sites are sampled routinely. Qualifiers of JFD#, where # is the RPD (or the difference for low level values) are added for field duplicate differences. When results are > 5x the reporting limit, a 35% RPD is used to identify potential deviations. When results are < 5x the

reporting limit, an absolute difference between the results that is < 2x PQL is considered to be acceptable reproducibility. (The laboratory uses MRL not PQL for these reports). Note that the times of collection indicate that these samples are not collected at the same time, but are sequential samples. An RPD of 50% is recommended for a precision limit for these samples. The QAPP does not define air QC.

Parent		DUP	RPD	Qualifier
Cis-1,2-dichlorothene	32	59 ug/m3	59	JFD59
Chloroform	1000	1800 ug/m3	57	JFD57
Carbontetrachloride	190	360 ug/m3	62	JFD62
Trichlorethene	580 (not diluted)	960 ug/m3 (diluted)	49.3	JFD49
Tetrachloroethane	80	150 ug/m3	61	JFD61
1,1,2,2-tetrachloroethane	560	910 ug/m3	48	JFD48

XII. TCL COMPOUNDS

A. The identification is accurate and all retention times, library spectra and reconstructed ion chromatograms (RIC) were evaluated for all detected compounds:

Yes __X_ No____ NA___

It is noted that there are high dilutions for tetrachloroethane; 1,1,2,2-tetrachloroethane, trichloroethene and chloroform and some other compounds. No dilution factors are noted on the results forms but dilutions have been estimated from the MDLs. For compounds that are reported from a re-analysis at the higher dilution, the lab has added a "D" flag. All the samples are at least 1.5 times the MRL of the blanks indicating a general overall lower volume or dilution. Sample SVEF has MRLs 15 times higher than the baseline (method blank), SVEA at 4 times, EFF at 2.4 times and SVEG and DUP at 7.5 times.

B. Quantitation was checked to determine the accuracy of calculations for representative compounds in each internal standard set

Yes___ No ____ NA___X__

OVERALL ASSESSMENT

Data are considered to be usable for project purposes after consideration of qualifiers or comments. Points of significance are summarized below:

Attachment 10-2

Note an extended list of volatile compounds was reported. Level III data packages were submitted and Level III validation was performed for holding times, chain of custody, calibrations and QC.

Calibration

Minimum response factors are not defined by the method, but met validation guidance. There were 3 calibrations as there were 2 days of analysis for dilutions.

Laboratory Duplicate

The laboratory duplicates was sample FSVE-SVEC-2Q11-NS. RPDs were reported above the limit, but these were low level results that met the 2 x PQL criteria.

Method Blanks

Methylene chloride was reported in all 3 blanks

4/26/11 at 0.041 ppbv; 4/28/11 at 0.046 ppbv; 4/29 at 0.042 ppbv.

The samples have been diluted for most reported results. The blank is multiplied by a factor determined from the reported MRL for each sample/compound. See the EDD and table at the end of the report for the final blank value as data are qualified UMB#, where # is the blank corrected value. All qualifiers are from the 4/26/11 analysis. Only data that are less than 10 x the blank (corrected for dilution) are qualified.

Field Duplicates

One field duplicate pair is reported: FSVE-SVEG-2Q11: -NS and DUP. These are regularly sampled locations and the precision is built in as the sites are sampled routinely. Qualifiers of JFD#, where # is the RPD (or the difference for low level values) are added for field duplicate differences. When results are > 5x the reporting limit, a 35% RPD is used to identify potential deviations. When results are < 5x the reporting limit, an absolute difference between the results that is < 2x PQL is considered to be acceptable reproducibility. (The laboratory uses MRL not PQL for these reports). Note that the times of collection indicate that these samples are not collected at the same time, but are sequential samples. An RPD of 50% is recommended for a precision limit for these samples. The QAPP does not define air QC.

Attachment 10-2

Parent		DUP	RPD	Qualifier
Cis-1,2-dichlorothene	32	59 ug/m3	59	JFD59
Chloroform	1000	1800 ug/m3	57	JFD57
Carbon tetrachloride	190	360 ug/m3	62	JFD62
Trichlorethene	580 (not diluted)	960 ug/m3 (diluted)	49.3	JFD49
Tetrachloroethane	80	150 ug/m3	61	JFD61
1,1,2,2-tetrachloroethane	560	910 ug/m3	48	JFD48

Reporting Limits

It is noted that there are high dilutions for tetrachloroethane; 1,1,2,2-tetrachloroethane, trichloroethene and chloroform and some other compounds. No dilution factors are noted on the results forms but dilutions have been estimated from the MDLs. For compounds that are reported from a re-analysis at the higher dilution, the lab has added a "D" flag. All the samples are at least 1.5 times the MRL of the blanks indicating a general overall lower volume or dilution. Sample SVEF has MRLs 15 times higher than the baseline (method blank), SVEA at 4 times, EFF at 2.4 times and SVEG and DUP at 7.5 times.

TABLE OF QUALIFIED DATA

Lab ID	Client ID	Compound	Result ppbv	Qualifier
P1101503-001	FSVE-SVEA-2Q11-NS	Methylene Chloride	0.75	BMB.17
P1101503-002	FSVE-SVEB-2Q11-NS	Methylene Chloride	0.1	BMB.063
P1101503-003	FSVE-SVEC-2Q11-NS	Methylene Chloride	0.6	BMB.062
P1101503-004	FSVE-SVED-2Q11-NS	Methylene Chloride	0.095	BMB.062
P1101503-005	FSVE-SVEE-2Q11-NS	Methylene Chloride	0.084	BMB.063
P1101503-006	FSVE-SVEF-2Q11-NS	Methylene Chloride	3.4	BMB.61
P1101503-007	FSVE-SVEG-2Q11-NS	Methylene Chloride	2.4	BMB.31
P1101503-003DUP	FSVE-SVEC-2Q11-NS	Methylene Chloride	0.576	BMB.06
P1101503-007	FSVE-SVEG-2Q11-NS	cis-1,2-Dichloroethene	8	JFD59
P1101503-007	FSVE-SVEG-2Q11-NS	Chloroform	210	JFD57
P1101503-007	FSVE-SVEG-2Q11-NS	Carbon Tetrachloride	30	JFD62
P1101503-007	FSVE-SVEG-2Q11-NS	Trichloroethene	110	JFD49
P1101503-007	FSVE-SVEG-2Q11-NS	Tetrachloroethene	12	JFD61
P1101503-007	FSVE-SVEG-2Q11-NS	1,1,2,2-Tetrachloroethane	81	JFD48
P1101503-009	FSVE-SVEG-2Q11-DUP	cis-1,2-Dichloroethene	15	JFD59
P1101503-009	FSVE-SVEG-2Q11-DUP	Chloroform	370	JFD57
P1101503-009	FSVE-SVEG-2Q11-DUP	Carbon Tetrachloride	58	JFD62
P1101503-009	FSVE-SVEG-2Q11-DUP	Trichloroethene	180	JFD49
P1101503-009	FSVE-SVEG-2Q11-DUP	Tetrachloroethene	22	JFD61
P1101503-009	FSVE-SVEG-2Q11-DUP	1,1,2,2-Tetrachloroethane	130	JFD48

Attachment 10-3

DSA PROPRIETATRY LIST OF DATA VALIDATION QUALIFIERS

General to all:

JD# duplicate precision, # = value of the Relative Percent Difference (RPD)

JH# holding time exceeded, # = number of days (hours for some wet chem. analytes)

JL# laboratory control sample recovery, # = value of the percent recovery of the LCS

JMS# matrix spike recovery, # = value of the percent recovery of the spike

JT# temperature exceedence, # = temperature in degree C. exceeding holding time

R_# rejected data for associated reason noted in this list or below (R replaces 'J')

UB# blank contamination for the following contaminant sources, the qualifier can be expanded:

UEB equipment blank

UFB field blank

UPB laboratory preparation blank

Organic Data:

JC# calibration accuracy, # = a) a whole number for initial and continuing calibration % RSD or %D, b) a decimal number for response factors, or c) 0.9xx for linear curve

JCCAL or JICAL– denotes continuing or initial calibration if that level of specificity is required on the project

JI# internal standard recovery, # = value of the percent recovery of the internal standard for the specific sample

JN tentatively identified compound

JP# second column confirmation when 2 column difference > 25%, # = RPD of 2 results (for low level values, < 5 x RL, just JP, no number)

JS# surrogate recovery, # = percent recovery of surrogate spike, can be further specified as JSUR#

JQ identification issue, usually poor spectra or interferences

UB# blank contamination, # = highest concentration of method blank affecting data

UTB# trip blank contamination, # = value of TB compound (x dilution)

Attachment 10-4

DIANE SHORT & ASSOCIATES, INC.

PROPRIETARY DATA VALIDATION USABILITY SUMMARY

GENERAL TO ALL ANALYSES

Data are qualified referencing the USEPA Contract Laboratory Program (CLP) data validation guidance with the usability modifications defined by the Data Validator. Data validation qualification is noted by a "J" or "R" qualifier next to the reported data value. The "J" indicates that one or more of the method quality control (QC) limits have been violated and the data may be estimated. The "R" qualifier indicates that the data are considered to be rejected due to significant deviations from the acceptance limits. In order for the qualifiers to be useful in determining the effect of the violation on the data, codes for the violation(s) and a numeric value of the violation are appended to the qualifier.

The USEPA CLP laboratories 'flag' data on the Form I in the "Q" field. These 'flags' are not to be confused with the data validation qualifiers. 'Flags' are notations of laboratory procedures and/or QC alerts and are not necessarily indicative of data qualification. They are not to be used as qualifiers of data. The only code used by the laboratory that must be transferred over with lab data is the "U", undetected, code indicating that the value reported is the project reporting limit and that the analyte was not detected in the sample above the method detection limit.

Holding Times or sample integrity

JH#**,** where # is the number of days exceeding the holding time. Some wet chemistry methods have holding times in hours which will be noted in the report. Data could possibly be biased low as the number of days (hours) exceeds the allowed holding time due to loss of the compound.

JT#, where # is the degrees in temperature over the method temperature limit. Data could be biased low due to analyte degradation or other metabolic conversion.

JP, denotes a preservation issue, usually improper pH for inorganics. If there is a preservation issue for organics, a new qualifier may need to be defined in order to avoid confusing it with the 2 column JP qualifier.

Matrix Spike Accuracy

JMS#, where # is the value of the percent recovery of the matrix spike. The QC limit is usually defined by the laboratory using historic control charts for the particular matrix of the sample. For inorganic data a set limit of 75-125% is often used. Given that a matrix spike is also a duplicate sample, the limit for soil samples is extended for some projects. Data whose percent recoveries are less than the established limit could possibly be biased low with respect to the extent of the recovery. Data whose percent recoveries are greater than the limit could possibly be biased high with respect to the extent of the recovery. Undetected data are not qualified for high spike recoveries. For organics, only the parent sample is qualified. For inorganics, the entire set associated with the QC sample is qualified. The %R can indicate:

- the accuracy of the laboratory analytical procedures and instrumentation,
- the geochemical interaction of the specific analyte with the sample matrix, or
- the homogeneity or reproducibility of the sample aliquot.

Laboratory Control Sample (LCS) Accuracy

JL#, where # is the value of the percent recovery of the LCS from the true value or established laboratory limits. The LCS is a measure of laboratory method accuracy. All data associated with the LCS are qualified. If the solid LCS is outside of the defined range, the reviewer calculates a percent recovery from the mean of the range and reports that recovery. Undetected data are not qualified for high LCS recoveries. Organic data may only be qualified for the LCS when the MS/MSD is also out of control for the same compounds. The percent recovery of each analyte from the LCS standard can indicate:

- the percent efficiency of the sample digestion procedure in the laboratory, or accuracy of the analytical method and instrumentation.

ORGANIC ANALYSES

BLANK CONTAMINATION

UB#, where # is the value of the highest blank affecting the data. These data are considered to be fully usable as undetected values. The reported value is considered to be due to contamination from preparation, general laboratory contamination or solvents, or from field contamination. The extent of the contamination is reflected in the value of the blank. Any positive value reported above the MDL is considered to be contamination. When the blank value has not been reported and the determination has

been made from a direct comparison of the raw data blank to the raw data sample, no number follows the qualifier. For the common laboratory contaminants (e.g., methylene chloride, acetone, phthalates), only values less than 10 times the blank value are qualified. For other contaminants, values less than 5 times the blank value are qualified. If there are field blanks, the qualifier may be modified to UMB for method blank contamination and UFB for field blanks or UTB for trip blanks.

MATRIX SPIKE DUPLICATE PRECISION

JD#, where # is the value of the relative percent difference (RPD) between the %Rs of the spikes. The RPD is an absolute number and a high or low bias cannot be determined. The larger the number is over the limit, the greater the difference between the two reported sample values. This may indicate the nonhomogeneity of the sample matrix or inadequate sample preparation. Data should be used knowing that there is a range of values around the reported value. Only the parent sample is qualified, and usually only if there is also a spike out of control.

SURROGATE SPIKE ACCURACY

JS#, where # is the surrogate spike recovery. See matrix spike as application is the same.

Calibration Accuracy

JC#, where # is a whole number for initial and continuing calibration curve data or a decimal number (0.0xx) for response factors or correlation coefficient (.9xx). JICAL may also be used for initial calibration and JCCAL for continuing calibration if the distinction is determined to add to the usability of the data.

For initial calibration, the number indicates the deviation from the relative response factor per the relative standard deviation (RSD). The RSD limit is usually 20-30 percent. For continuing calibration, # is the percent difference (%D) of the continuing calibration standard from the initial calibration average response factor. The limit is usually from 15-25 percent. As the value increases, the range of the quantitation could possibly increase, therefore increasing the estimation of the reported value. Undetected data are not qualified for these calibration violations if the response factors indicate that the compound can be detected if present.

When # is a decimal value, usually less than 0.05, it is the value of the average initial response factors or daily continuing calibration response factor. These data could be false non-detects or biased low due to the lack of sensitivity of the method for the qualified compound.

When # is a decimal value, usually less than 0.995, the value is the correlation coefficient of the linear curve used for initial calibration. As the value decreases, the range of the quantitation could possibly increase, therefore increasing the estimation of the reported value. Undetected data are not qualified for these calibration violations if the response factors indicate that the compound can be detected if present.

Internal Standards

JI#, where # is the value of the percent recovery of the internal standard (IS) area counts for the specific sample. The %R is calculated from the 12-hour average. Values greater than 100 percent indicate quantitation could be biased high. Values less than 50 percent indicate quantitation could be biased low. The raw data should be evaluated, however, to determine if there is an overall suppression of the results. Evaluation of the surrogate and matrix spike recoveries can sometimes give an indication of the impact of the internal standard on the data calculation. Data associated with the given IS are qualified. If there are several IS compounds out of limits and there is a trend to the recoveries, all data will be qualified per the average recovery.

LINEAR RANGE

JE indicates that the value exceeded the linear range of the instrument. A numeric bias cannot be determined, but the bias would be low. These compounds are routinely re-analyzed at a dilution and that value should be used for project decisions. For all other compounds, the lowest dilution should be reported.

Tentatively Identified Compounds (TICs)

JN. The laboratory flags TICs that are not on the method list of standard compounds, but which are reported from electronically stored library spectra. Identification may be accurate, and quantitation is estimated due to lack of daily standard. The JN may also be applied when second column confirmation has not been performed for identified compounds.

Attachment 10-4

Second Column Confirmation

JP indicates that the reported number has a percent difference greater than 25 percent from the second column (quantitation column or confirmation column). When the difference between the two values is greater than 25 percent, it is possible that the higher of the two values was due to interferences. The # after the 'P' is the value of the % difference between the values. Most laboratories report the lower of the two column results in their analytical report. However, data usability reviews for those compounds with greater than 25% RPD will be completed and the more accurate of the two column results will be reported. If no distinction can be made between the two results, the higher of the two will be included in the final report.

DDT/Endrin Breakdown

JX#, where # is the value of the DDT/endrin breakdown. When this number is >20%, it is possible that DDT is being broken down due to column or instrument problems and the data could be biased low due to loss of the compound.

IDENTIFICATION ISSUES

JQ is usually used to denote that an identification is questioned due to poor spectra (missing masses, or mass ratios that do not match reference) or chromatographic interferences.

STANDARD OPERATING PROCEDURE 11 – FIELD SAMPLING TECHNICAL SYSTEMS AUDIT

Lead Organization: Department of the Army (DA)

Preparing Organization: HDR

SOP Approved by: Field Team Leader: Justin Bills

Project QA Officer: Lynn Lutz

Project Manager: Tom Holmes

1.0 PURPOSE AND SUMMARY

This Standard Operating Procedure (SOP) provides guidance and a checklist for the performance of a field sampling technical systems audit (field audit). Activities to be audited include field sample collection, sample handling, documentation, and field analysis of samples collected at Defense Depot Memphis, Tennessee (DDMT). Review of this SOP and other applicable field and sampling SOPs is mandatory prior to the start of each field audit.

2.0 HEALTH AND SAFETY

Each individual is required to have read and understood the Health and Safety Plan (HASP) for the specific project activity. Upon arrival at the site, each person shall sign the acknowledgement sheet confirming their review of the HASP. Personal protective equipment and other provisions for site safety requirements are discussed in the HASP. Any questions should be addressed to the Field Team Leader (FTL).

3.0 PERSONNEL QUALIFICATIONS AND RESPONSIBILITIES

The field audit will be conducted by the project quality assurance (QA) Officer or a designated representative familiar with field sampling techniques and requirements, field analyses, and sample handling and documentation requirements.

4.0 EQUIPMENT AND SUPPLIES

The auditor will use the field audit checklist (Attachment 11-1 to this SOP) and a pen with blue or black waterproof ink to record field activities and sample handling at the field office. The audit report will be prepared in Microsoft Word.

5.0 PROCEDURE

The auditor will first read and become familiar with the applicable SOPs, specifically those dealing with sample collection, documentation, handling, and analysis of field parameters.

The auditor will then accompany the sampling team to the field location and observe the activities, comparing activities performed against the SOP requirements, and note discrepancies.

The auditor will use the checklist (Attachment 11-1) to ensure that all necessary activities are performed by the field sampling team and observed by the auditor. If the auditor notes discrepancies, he or she will make notations on the checklist and discuss the issues with the FTL at the end of the audit.

If the auditor notes serious discrepancies that would adversely impact the data quality, these issues will be immediately brought to the attention of the FTL and the project manager (PM). Discussion may result in changes to be implemented immediately in the field, and these changes along with the date and time of implementation will be noted on the audit checklist. The field sampling team will also record the changes along with the date and time of change in the field notes. Such changes would likely result in changes to the applicable SOP(s).

5.1 Using the Field Audit Checklist

The sections below describe the general areas of evaluation as listed on the checklist (Attachment 11-1).

5.1.1 Part 1: Sampling and Analysis Plan

Part 1 asks questions to ensure the existence and adequacy of a Sampling Analysis Plan (SAP) and relevant SOPs. The auditor will review these documents before going into the field.

5.1.2 Part 2: Organization, Management& Personnel

Part 2 asks questions to determine whether sampling staff have been adequately and appropriately trained for the field activities. The auditor will review these records before going into the field.

5.1.3 Part 3: Equipment

Part 3 asks questions to determine whether the sampling equipment, including sample containers, are the appropriate equipment, are ready to use, and have a routine maintenance plan to ensure performance. The auditor will review these records and will also interview the FTL before going into the field.

5.1.4 Part 4: Sampling Event Information

Part 4 asks questions to ensure that the sampling event and sample identifications have been adequately planned and that samples are clearly identified and handled properly. The auditor will review these records before and after going into the field for sample collection.

5.1.5 Part 5: Sample Management

Part 5 asks questions to ensure that samples are clearly identified and handled properly. The auditor will observe these activities in the field during sample collection, storage, and shipment.

5.1.6 Part 6: Field Analyses

Part 6 asks questions to ensure field analyses are performed according to the SOPs and recorded accurately and adequately. The auditor will make these observations in the field.

5.2 Field Sampling Technical Systems Audit Report

The auditor will prepare a Field Sampling Technical Systems Audit Report for delivery to the PM and the FTL within one month of the completion of the on-site audit. As noted in Section 5.0, any changes to procedures that were made in the field will be documented in the report, and these changes must also be added to the relevant SOP, if applicable. The SOP changes will be made prior to the next field sampling event, and the FTL will be responsible for ensuring that all field sampling personnel have read the revised SOP and are aware of and familiar with the changes.

6.0 DATA AND RECORDS MANAGEMENT

The field auditor will complete all sections of the audit checklist, scan the checklist into a PDF file, and save the file to the appropriate location on the HDR network Z: drive.

The field auditor will prepare a summary report of audit results and save the file to the appropriate location on the HDR network Z: drive.

7.0 QUALITY CONTROL AND QUALITY ASSURANCE

All work will be performed in accordance with the Quality Assurance Project Plan (QAPP), the HASP, the specific work plan, the specific sampling plan details (SPD) and applicable SOPs. No erasures or

mark outs will be made on the checklist or other field notes. A single line will be used to strike out errors and will be annotated with the initials and date of the editor.

8.0 REFERENCES

Field audit checklist:
http://www.epa.gov/greatlakes/qmp/auditing/Example%20_Field_Sampling_Audit_Checklist_Final_Dec2010.pdf

ATTACHMENT 11-1

Example Checklist for Field Sampling Audit

Sampling Organization	Date of Evaluation	Name and Affiliation of Evaluator

Item to be Evaluated	*Y*	*N*	*N/A*	*Comments*
Part 1: Sampling and Analysis Plan (SAP)				
1.1 General Information				
Is there a sampling plan?				
Are there procedures for the transportation, handling, protection, storage, retention and/or disposal of samples, including all provisions necessary to protect the integrity of the sample?				
Is there a documented system for uniquely identifying all samples and subsamples, to ensure that there can be no confusion regarding the identity of such samples at any time?				
Does the sampling process address the factors to be controlled to ensure the validity of the environmental test and calibration results?				
Is there a process for documenting corrective actions taken in the field?				
1.2 Standard Operating Procedures (SOPs)				
Are there SOPs for field activities available at the location where sampling is taking place and are they accessible to all sample collectors?				
Are the SOPs official documents (e.g., signed and dated)?				
Have the SOPs been approved for the project?				
Part 2: Organization, Management & Personnel *(not checked on site)*				
Are the sampling personnel's qualifications and/or training certifications adequate for the tasks performed?				
Are names of all sampling personnel recorded?				
Do sampling personnel meet minimum qualifications specified in the contract?				
If the sampling organization is supporting a larger organization, are there any arrangements that could cause a conflict of interest?				
Does the sampling agency have policies or procedures to ensure client confidentiality and propriety rights?				
Are staff training records maintained and up to date?				

ATTACHMENT 11-1

Item to be Evaluated	*Y*	*N*	*N/A*	*Comments*
Part 3: Equipment				
3.1 General Equipment information				
Is the type of equipment sufficient for the sampling project?				
Is the quantity of equipment sufficient for the sampling project?				
Is the following information recorded for each piece of equipment that will be used for sampling project:				
Maintenance and repair procedures for equipment or instrument?				
Routine cleaning procedures?				
Filling solution replacement for probes?				
Parts replacement for instruments or probes?				
Calendar date for each procedure performed?				
Names of personnel performing maintenance and repair tasks?				
Description of malfunctions associated with any maintenance and repair?				
Vendor service records?				
Inclusive rental dates, types and unique descriptions of rental equipment?				
Is the equipment storage procedure acceptable?				
Is there an existing QC check on sampling equipment?				
3.2 Field Instrument Calibration				
Is information about all calibration standards and reagents used for field testing linked to the calibration information associated with the field testing measurements for the project?				
Are field instruments properly calibrated and calibrations recorded in a bound field log book?				
For each instrument unit used for the sampling project, is the following information recorded for all calibrations:				
Unique identification (designation code) for the instrument?				
Date and time of each calibration and calibration verification?				
Instrument reading or result (display value) for all calibration verifications, with appropriate measurement units?				
Names of analyst performing each calibration of verification?				
Designation of each calibration standard used linked to the associated records for the calibration standard?				
The acceptance criteria for each calibration and verification standard used?				
The assay specifications or acceptance criteria for any QC standard or sample used to independently verify the calibration of the standard?				

ATTACHMENT 11-1

Item to be Evaluated	*Y*	*N*	*N/A*	*Comments*
Are all corrective actions performed on the instrument prior to attempting re-verification or recalibration of the instrument linked to the records required for preventive maintenance?				
Does the field instrumentation documentation include the standard concentrations used for calibration?				
Did all field-testing equipment and instrumentation brought to the field appear to function properly?				
Are manufacturer's suggested maintenance activities and any repairs performed and documented for all applicable equipment and instruments?				
3.3 Containers				
Are sample containers well organized, properly prepared, protected from contamination, and ready for use?				
Are proper sample containers and sizes used for each type of sample?				
Are certificates of analysis for pre-cleaned bottles maintained on file?				
Are all containers and container caps free of cracks, chips, discolorations and other features that might affect the integrity of the collected samples?				
3.4 Sampling Equipment				
Is the appropriate equipment used for the sampling project? Check all relevant equipment used for sample collection, handling, storage and transport.				
Is equipment constructed of materials appropriate for the analytes of interest?				
Is equipment brought to the field precleaned?				
For equipment decontaminated on-site in the field, are the date and time of the cleaning procedure recorded in the field records or referenced in an internal SOP?				
Are cleaning steps in all procedures used for decontamination documented either by description or reference to an SOP?				
Are there current maintenance records for all field equipment?				
Part 4: Sampling Event Information				
For all samples, is the following information recorded and transmitted to the client?				
Site name and address?				
Date and time of sample collection?				
Name of sampler responsible for sample transmittal?				
Unique field identification code for each sample container or group of containers?				
Total number of samples collected?				
Required analyses for each sample container or group of containers?				
Sample preservation used for each container or group of containers?				

ATTACHMENT 11-1

Item to be Evaluated	*Y*	*N*	*N/A*	*Comments*
Comments about samples, sample sources or other relevant field conditions?				
Identification of common carrier used to transport the samples, when applicable?				
Are shipping invoices and related records from common carriers archived with the field records, when applicable?				
Are sampling locations adequately documented in a bound field log book using indelible ink?				
Are photos taken and is a photo log maintained?				
4.1 Field QC				
Are trip blanks and/or field blanks collected as specified in the approved sampling plan?				
Are field blanks collected after equipment is decontaminated in the field?				
Are field blanks collected if no equipment was cleaned by the sampling organization?				
Are additional samples for matrix spike/matrix spike duplicate analyses collected?				
Are all QC samples collected in the same manner as the routine field samples?				
Part 5: Sample Management				
5.1 Collection				
Are the samples taken from a representative point of the source?				
Are the samples homogenous where appropriate?				
When possible, does sampling originate from the suspected least contaminated location first and progress to the suspected most contaminated location?				
Are samples for different analyte groups collected in the appropriate order?				
Are samples collected for all required analyses?				
Are samples to be tested for dissolved metals filtered prior to preservation?				
Is every effort made to prevent cross-contamination of samples?				
Are gloves worn by all samplers handling purging equipment, sampling equipment, measurement equipment, and sample containers?				
Are new, clean unpowdered gloves used for each glove change?				
Is care taken to avoid contact with sample and sample container interiors?				
Are VOC sample containers protected from any fuel sources and fuel-powered equipment?				
Do VOC sample containers remain capped until just prior to sample collection and do they remain capped after sample collection?				
Where applicable, are samples collected for measurement of dissolved components, filtered, preserved with acid, and placed on ice within 15 minutes of collection?				

ATTACHMENT 11-1

Item to be Evaluated	*Y*	*N*	*N/A*	*Comments*
5.2 Collection Devices				
Is sample collected using an intermediate collection device?				
Are intermediate collection devices rinsed with ample amounts of site water prior to collecting the sample?				
Is rinse water from intermediate devices discarded away from and downstream of the sampling location?				
Is the use of intermediate collection devices avoided when sampling for VOC's, oil and grease, or microbiologicals, where practical?				
Are any intermediate collection devices constructed of material appropriate for the analytes to be measured?				
Are sample containers submerged neck first, inverted into the oncoming direction of flow where applicable, slowly filled, and returned to the surface for preservation, if applicable?				
5.3 Sample Labeling				
Is each sample container or group of containers tagged or labeled with a unique field identification code that distinguishes the sample from all other samples?				
Are the unique identification codes for samples recorded in a manner that links the codes to all other field records associated with the samples?				
Is waterproof indelible ink used to label containers?				
5.4 Storage				
Are samples for different parameters segregated during storage?				
Are samples stored on ice?				
Is the cooler clearly labeled?				
Are samples properly preserved (if applicable)?				
5.5 Preservation				
Do all sample preservation techniques conform to SOP or method requirements?				
Are all samples properly preserved within 15 minutes, as applicable?				
Are the preparation and dispensing of preservatives documented and traceable?				
Is preservation information and verification recorded for each sample, as applicable?				
Are samples placed on ice immediately after collection, if applicable?				
If the samples are found to contain cyanide, are they NOT acidified?				
5.6 Delivery				
Are samples protected during delivery to prevent breakage?				
Are samples shipped in a timely manner?				

ATTACHMENT 11-1

Item to be Evaluated	*Y*	*N*	*N/A*	*Comments*
5.7 Disposal				
Are wastes generated as a result of the sampling project containerized and stored for proper disposal according to applicable local, state, and federal regulations?				
Are all sampling-derived waste containers properly labeled?				
Is all sampling-derived waste properly disposed of?				
5.8 Documentation				
Is waterproof ink used for all paper documentation?				
Are the date and time of sample collection recorded for all samples?				
Are the ambient field conditions recorded for all samples?				
Is a specific description of each sampling location (source) recorded?				
Does the chain of custody/traffic report include the following: date, time, sample numbers, sampler names, shipping method, number of samples, matrix, and comments?				
Is preservation information recorded on the chain of custody/traffic report?				
Are copies of traffic reports or COC sent to the proper recipients?				
Are deviations, additions, or exclusions from the documented sampling procedure recorded in detail with the associated sampling information?				
Are these deviations included in all documents containing environmental test and/or calibration results?				
Are these deviations communicated to the appropriate personnel?				
Are all errors in documentation (if applicable) corrected and initiated without obliteration?				
5.9 Field Reagents				
Are the concentration (or other assay value), the vendor catalog number and the description of the standard or reagent recorded for all preformulated solutions, neat liquids, powders, and blank water?				
Are certificates of assay, grade and other vendor specifications for all standards and reagents retained and recorded for the standards and reagents?				
Are the lot numbers and inclusive dates of use recorded for all reagents, detergents, solvents, and other chemicals used for decontamination and preservation of samples?				
Are the expiration dates for all calibration standards and reagents recorded?				
Are expired standards and reagents verified prior to use during sample collection?				
Are all steps used for preparation of standards or reagents in-house documented either by description or reference to an SOP?				

ATTACHMENT 11-1

Item to be Evaluated	*Y*	*N*	*N/A*	*Comments*
Part 6: Field Analyses				
6.1 General Field Test Information				
Are all field measurement tests and related data recorded and linked to the project, the date, and the sample source?				
Are all field measurements recorded with the appropriate units, the value of the test result, the parameter measured, the name of the analyst performing the test, the time of the measurement and the unique identification for the test instrument used?				
6.2 pH				
Are all samples requiring pH adjustment tested for proper pH preservation?				
Is at least one sample per analyte group requiring pH adjustment tested for proper preservation during repeat sampling?				
Is pH paper or a pH electrode inserted into sample containers?				
Do the pH meter and electrode system meet SOP specifications for accuracy, reproducibility and design?				
Are all measurements corrected for temperature (manual or automatic)?				
Is a pH 7 buffer used as the first calibration standard?				
For pH, do all calibration verifications meet the acceptance criteria?				
If the calibration and/or calibration verifications did not meet the acceptance criteria, is the calibration or verification identified as a failure and was this documented in the calibration log?				
Are all sample measurements associated with acceptable calibration verifications?				
Is the pH meter system checked on a weekly basis to ensure >90% theoretical electrode slope?				
Are the field instrument probes rinsed with deionized or distilled water between standard solutions and between sample measurements?				
Are instrument pH readings allowed to stabilize before pH values are recorded?				
6.3 Filtration				
Are samples collected for analysis of dissolved components filtered within 15 minutes of collection and before addition of chemical preservatives where appropriate?				
Unless otherwise specified, are applicable samples filtered using a 0.45-µm pore size?				
6.4 Temperature				
Do the temperature measurement devices meet SOP and/or sampling event specifications for design and measurement resolution?				

ATTACHMENT 11-1

Item to be Evaluated	*Y*	*N*	*N/A*	*Comments*
Are all sample measurements associated with calibration verifications of the temperature measurement device at a minimum of two temperatures using a NIST-traceable thermometer?				
If the calibration and/or calibration verifications did not meet the acceptance criteria, is the calibration or verification identified as a failure and was this documented in the calibration log?				
Are all temperature measurements chronologically associated with acceptable calibration verifications?				
Are the temperature device readings allowed to stabilize before measurement values were recorded?				
6.5 Conductivity				
Do the specific conductance meter and electrode system meet the SOP and/or sampling event specifications for accuracy and reproducibility?				
Do all calibration verifications meet the acceptance criterion?				
If the calibration and/or calibration verifications did not meet the acceptance criteria, is the calibration or verification identified as a failure and was this documented in the calibration log?				
Are all conductivity measurements chronologically associated with acceptable calibration verifications?				
Are all conductivity measurements corrected for temperature (manual or automatic)?				
Is the instrument allowed to stabilize before measurement values are recorded?				
6.6 Turbidity				
Does the turbidimeter meet the SOP and/or sampling event specifications for accuracy and reproducibility?				
Are all sample measurements associated with acceptable calibration verifications?				
If the calibration and/or calibration verifications did not meet the acceptance criteria, is the calibration or verification identified as a failure and was this documented in the calibration log?				
Are the sample cells (optical cuvettes) inspected for scratches and discarded or coated with a silicone oil mask, as necessary?				
Are the sample cells (optical cuvettes) optically matched for calibrations and sample measurements?				
Are the sample cells (optical cuvettes) cleaned with detergent and deionized or distilled water between standard solutions and between sample measurements, as applicable?				
Are the sample cells (optical cuvettes) rinsed with sample prior to filling with sample for measurement?				

ATTACHMENT 11-1

Item to be Evaluated	*Y*	*N*	*N/A*	*Comments*
Is the exterior of the sample cell (optical cuvette) kept free of fingerprints and dried with a lint-free wipe prior to insertion in the turbidimeter?				
6.7 Dissolved Oxygen				
Do the dissolved oxygen meter and electrode system meet the SOP and/or sampling event specifications for accuracy and reproducibility?				
Are all sample measurements associated with acceptable calibration verifications?				
If the calibration and/or calibration verifications did not meet the acceptance criteria, is the calibration or verification identified as a failure and was this documented in the calibration log?				
Are all measurements corrected for temperature (manual or automatic)?				
Are all measurements corrected for salinity, where applicable (manual or automatic)?				
Is the salinity (conductivity) sensor calibration verified?				
Is the dissolved oxygen electrode stored in a water-saturated air environment when not in use?				
Are the dissolved oxygen readings allowed to stabilize before measurement values were recorded?				

APPENDIX C

LABORATORY STANDARD OPERATING PROCEDURES

SOP L1 – Analysis of Volatile Organic Analytes by Methods 8260A and 8260B, 11 November 2013 (MSV01_Rev.19) (Microbac)

SOP L2 – Organic Carbon, Total (Oxidation), EPA 415.1/SW846 9060A/SM5310C-2000 (2011 Editorial Revision), 20 March 2014 (K4151_Rev.16) (Microbac)

SOP L3 – Organic Analysis of Metabolic Acids, Method 830MBA, 23 July 2014 (HPLC03_Rev.7) (Microbac)

SOP L4 – Analysis of Dissolved Gases in Groundwater, EPA RSKSOP-175, 26 June 2014 (RSK01_Rev.17) (Microbac)

SOP L5 - Perkin Elmer Optima 4300 Inductively Coupled Plasma Atomic Emission Spectroscopy SW846 6010/EPA 200.7, 15 April 2014 (ME600E_Rev.15) (Microbac)

SOP L6 - Determination of Volatile Organic Compounds in Air Samples Collected in Specially Prepared Canisters and Gas Collection Bags by Gas Chromatography/Mass Spectrometry (GC/MS), 15 February 2014 (VOA-TO15_Rev.21) (ALS)

SOP L7 - Microwave Digestion – Aqueous, SW846 3015A, 15 December 2013 (ME407_Rev.15) (Microbac)

SOP L8 - Sample Receiving and Login, 12 November 2013 (LOGIN01_Rev.16) (Microbac)

SOP L9 – SW846 Methods 5030 and 5035, Purge and Trap for Volatile Organics, 15 June 2011 (PAT01_Rev.13) (Microbac)

SOP L10 - Sample Receiving, Acceptance and Log-In, 22 February 2014 (SMO-SMPL-REC_Rev.14) (ALS)

STANDARD OPERATING PROCEDURES
ANALYSIS OF VOLATILE ORGANIC ANALYTES
BY METHOD 8260 and SM6200B

Issue/Implementation Date: 11 November 2013

Last Review Date: 11 November 2013

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Approved By:

Michael D. Albertson, Volatiles Supervisor — Date: 11/12/13

Wade T. DeLong, Quality Assurance Officer — Date: 11-21-13

Leslie S. Bucina, Laboratory Manager — Date: 11-21-13

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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 8000B (December 1996), 8000C (March 2003), 8260A (September 1994), 8260B (December 1996), Standard Method 6200B (21st Edition), 5030A (July 1992), 5030B (December 1996), 5030C (May 2003), and 5035A (July 2002); AFCEE QAPP's 1998, 2001, and 2005. SOP MSVO1 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 Soils collected in widemouth bottles for compliance with the Ohio EPA Contract must be reported with references to SW-846 method 5030A and 8260A. This SOP fulfills the requirements of Methods 8260A and 8260B. Method 8260A must not be used for South Carolina samples.

1.3 Table 1 lists the target compound list for this method.

1.4 Appendix I contains procedures for selected ion monitoring (SIM) analysis for 1,4-dioxane. Appendix II contains suggested primary and secondary characteristic ions. Appendix III contains the procedures for "Ultra low-level water analysis". Section 11.13 contains information regarding the analysis of wipe samples.

1.5 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

BFB	Bromofluorobenzene
CCV	Continuing calibration verification
DI water	Deionized water
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometer
HCl	Hydrochloric acid
ICAL	Initial calibration
ICV	Initial calibration verification
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
LIMS	Laboratory Information System
LOD	Limit of Detection
LOQ	Limit of Quantitation

MB	Method blank
MDL	Method detection limit
MS	Mass Spectrometer
MS	Matrix spike
MSD	Matrix spike duplicate
MSDS	Material Safety Data Sheets
NCR	Nonconformance report
PFTBA	Perfluorotributylamine
PPE	Personal Protective Equipment
QC	Quality control
RGT	Reagent
RL	Reporting limit
RT	Retention time
SOP	Standard Operating Procedure
STD	Standard
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compounds

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

2.0 SAFTY PRECAUTIONS

2.1 Standard laboratory safety procedures must be followed when working with unknown samples. Gloves must be worn while handling any chemicals, standards, or samples. Other required PPE includes lab coats and safety glasses with sideshields.

2.2 **WARNING:** The following VOC's have been tentatively classified as known or suspected human or mammalian carcinogens:

benzene	chloroform
carbon tetrachloride	vinyl chloride

The toxicity or carcinogenicity of the other reagents and analytes used in this method have not been precisely defined, therefore, each chemical and sample must be treated as a potential health hazard and exposure reduced to the lowest possible level. Procedures involving primary standards and sample preparation shall be performed in a fume hood.

2.3 MSDS for each analyte and reagent used within the laboratory are available to all employees. Consult MSDS's prior to handling chemicals.

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 5030B/C and 5035A. Soil samples not utilizing Method 5035A must be collected in 125 mL pre-cleaned glass screw cap jars with teflon-lined lids. Soil samples collected via 5035A must also be collected in Encore (or equivalent) containers then transferred to 40 mL VOA vials for analysis. Refer to Microbac SOP PAT01 for additional requirements.

3.2 Water samples preserved with HCl (pH < 2) must be analyzed within 14 days of sample collection. Unpreserved water samples (pH ≥ 2) must be analyzed within 7 days of sample collection. Waste, soil, and sludge samples do not require the addition of preservative but must be stored at ≤ 6° C. Solid samples utilizing Method 5035A 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 (8260A), are stored at ≤ 6° C. Samples collected via 5035A 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. Temperatures are recorded daily.

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, 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 and were established per Microbac SOP 45. Actual project RLs may be higher.

4.5 Precision and accuracy data were derived from an initial demonstration of capability using spiked control samples. The laboratory uses results from LCS to assess precision/accuracy and to annually evaluate the associated control limits.

4.6 AFCEE and other specific QA objectives may be found in the appropriate Statement-of-Work or QAPP.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Samples for volatile organics analyses are susceptible to laboratory contaminants (e.g.: methylene chloride, acetone, n-hexane). To eliminate the potential for interferences from other areas of the laboratory, the Volatiles Laboratory has an independent air intake system and positive air pressure is maintained in the laboratory.

5.2 Samples preserved with HCl or sodium bisulfate may result in the loss of 2-chloroethylvinylether as a target or spiked analyte due to its reactivity with the preservatives.

5.3 Soil analyses may result in low internal and/or surrogate standard recovery due to the poor purging efficiencies of some matrices. Reanalysis must be performed to confirm matrix interference.

5.4 Carry-over contamination may occur when a sample containing low levels of VOC's is analyzed immediately following a sample containing high levels of VOC's. If this situation occurs during a non-monitored analysis, the sample containing the low concentration VOC's may require reanalysis.

5.5 Samples may become contaminated by diffusion of volatile organics through the septum seal into the sample during shipment and storage. A trip blank prepared

from organic-free reagent water and carried through the sampling, handling, and analysis steps serves as a check on such contamination.

5.6 Storage blanks are placed in refrigerator and freezer units used for the storage of samples for volatiles analysis. Refer to Section 13.0 for storage blank procedures.

6.0 EQUIPMENT AND SUPPLIES

6.1 GC/MS systems: Hewlett-Packard (HP) 6890 Gas Chromatographs equipped with HP 5973 Mass Spectrometers. Systems utilize HP Enviroquant software. Refer to Table 4 for suggested operating parameters.

6.2 Purge-and-trap units: Tekmar liquid sample concentrator (LSC) 2000, 3000, Velocity, and Stratum; Varian (or equivalent), Archon auto-sampler; Tekmar Atomx liquid sample concentrator / autosampler. Refer to Table 4 for suggested operating parameters.

6.3 Top loading balances: Ohaus Navigator, Mettler PE600,

6.4 Capillary columns: 60 M Restek 502.2, 0.32 mm ID, 1.8 μm film thickness

6.5 Traps: Supelco Vocarb 3000; Tekmar trap #9.

6.6 Volumetric flasks: Class A; 1 mL to 200 mL

6.7 Mininert vials with septum valves: 1 mL to 10 mL

6.8 40 mL VOA vials: I-Chem, Thermo Scientific

6.9 Syringes: Hamilton Gas tight with Luer lock tip: 25 mL, 5 mL; Gas tight with fixed needles: 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 Oven: Blue M baking oven

6.13 Equivalent equipment and supplies may be used.

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
HPMS10	Windows XP Professional	C10031	1 Gbps	Enviroquant Chemstation C.00.00
HPMS11	Windows XP Professional	HPMS11	10/100 Mbps	Enviroquant Chemstation D.00.00

7.0 STANDARDS AND REAGENTS

All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.1 Primary calibration standards:

STANDARD	VENDOR	PART NUMBER	CONCENTRATION
502.2 Mega Mix with MTBE	Ultra	DWM-596	2000 ug/mL
Custom Standard (Gases)	Ultra	CUS-12953	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
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
8260A / 8260B Internal Standards	Ultra	STM-520	2500 ug/mL (fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4)
8260A / 8260B 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
Custom Ma. Oxygenates Standard	Restek	567802	2000/4000 ug/mL
Volatile Organic Compounds Mix 6	Supelco	48799-U	2000 ug/mL
1,1,2-Trichloro-1,1,2-trifluoroethane	Accustandard	M-REF-14	200 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%

NOTE: Source of LCS/MS/ICV standards different then primary calibration standards.

7.4 Primary 4-bromofluorobenzene (BFB) standard: Ultra STS-112, 2500 ug/mL

7.5 Intermediate calibration standards: Primary calibration standards diluted to prepare intermediate standards as follows:

Intermediate Standard	Primary Standard	Concentration (ug/mL)	Volume (uL)	Final Volume (mL methanol)	Final Concentration (ug/mL)
VOA Mix 1	502.2 Mega Mix with MTBE	2000	500	5	200
	Custom Standard (Gases)	2000	500		
VOA Mix 2	Custom Concentrated Ketones #2	2000	500	5	200
	Vinyl Acetate	2000	500		
	2-Chloroethyl vinyl ether (2-CVE)	5000	200		
VOA Mix 3	1,3-Butadiene	2000	500	5	200
	Custom VOC Mix 3	2000	500		
	1-Bromopropane	10000	100		
VOA Mix 4	Acrolein-Acrylonitrile Mix	2000	500	10	100-400
	Mass. Oxygenates Standard	2000-4000	1000		

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	250	1000	10	25

7.7 Intermediate LCS / MS / ICV standards prepared as follows:

PRIMARY STANDARD	CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL METHANOL)	FINAL CONCENTRATION (ug/mL)
1-Bromopropane #1	99%	75	10	10038
VOC Mix **	200	1000	10	20
VOC Adds Mix	200	1000	10	20
Custom Gases **	250	800	10	20
Vinyl acetate ***	2000	100	10	20
Acrolein ****	5000	200	10	100
1,3-Butadiene ***	200	1000	10	20
MA Oxygenates ****	2000-4000	500	10	100-200
1-Bromopropane Intermediate *** #2	10038	20	10	20

** Denotes combined into one solution
*** Denotes combined into one solution
**** Denotes combined into one solution

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	2500	100	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 STANDARD, CONCENTRATION	WORKING STANDARDS CONCENTRATIONS (ug/Kg)								
	0.5	1 [5]	2 [25]	5 [50]	20 [80]	50* [100]	100 [200]	200	300 [300]
VOA Mix 1 (200 ug/mL)	N/A	N/A	N/A	N/A	5 uL	12.5uL	25 uL	50 uL	75 uL
VOA Mix 2 (200 ug/mL)	N/A	N/A	N/A	N/A	5 uL	12.5uL	25 uL	50 uL	75 uL
VOA Mix 3 (200 ug/mL)	N/A	N/A	N/A	N/A	5 uL	12.5uL	25 uL	50 uL	75 uL
VOA Mix 4 (200-400 ug/mL)	N/A	2.5 uL	6.25 uL	12.5uL	20 uL	25 uL	50 uL	N/A	75 uL
20 ppm mix 1+2+3 Intermediate Std	2.5 uL	5 uL	5 uL	12.5uL	N/A	N/A	N/A	N/A	N/A
Surrogate Standard (20 ug/mL)	N/A	5 uL	5 uL	12.5uL	N/A	N/A	N/A	N/A	N/A
Surrogate Standard (200 ug/mL)	N/A	N/A	N/A	N/A	5 uL	12.5uL	25 uL	50 uL	75 uL
Final Volume, DI Water (mL)	100	100	50	50	50	50	50	50	50

20 ppm intermediate = Mix 1 + Mix 2 + Mix 3

$$(20ppm\,intermediate = 50uL\,Mix1 + 50uL\,Mix2 + 50uL\,Mix3 + 350uL\,MeOH)$$

* Denotes CCV
[ ] Denotes Oxygenates

Water Initial Calibration Standards, ug/L
(suggested preparation)

STOCK STANDARD, CONCENTRATION	WORKING STANDARDS CONCENTRATIONS (ug/L)									
	0.3	0.4	1 [5]	2 [25]	5 [50]	20 [80]	50* [100]	100 [200]	200	300 [300]
VOA Mix 1 (200 ug/mL)	N/A	N/A	N/A	N/A	N/A	5 uL	12.5 uL	25 uL	50 uL	75 uL
VOA Mix 2 (200 ug/mL)	N/A	N/A	N/A	N/A	N/A	5 uL	12.5uL	25 uL	50 uL	75 uL
VOA Mix 3 (200 ug/mL)	N/A	N/A	N/A	N/A	N/A	5 uL	12.5uL	25 uL	50 uL	75 uL
VOA Mix 4 (200-400 ug/mL)	N/A	N/A	2.5 uL	6.25 uL	12.5uL	20 uL	25 uL	50 uL	N/A	75 uL
20 ppm mix 1+2+3 Intermediate Std	3 uL	2 uL	5 uL	5 uL	12.5uL	N/A	N/A	N/A	N/A	N/A
Surrogate Standard (10 ug/mL)	N/A	N/A	5 uL	5 uL	12.5uL	N/A	N/A	N/A	N/A	N/A
Surrogate Standard (100 ug/mL)	N/A	N/A	N/A	N/A	N/A	5 uL	12.5uL	25 uL	50 uL	75 uL
Final Volume, DI Water (mL)	200	100	100	50	50	50	50	50	50	50

20 ppm intermediate = Mix 1 + Mix 2 + Mix 3

$$(20ppm\,intermediate = 50uL\,Mix1 + 50uL\,Mix2 + 50uL\,Mix3 + 350uL\,MeOH)$$

* Denotes CCV
[ ] Denotes Oxygenates

7.9.2 Procedure for preparing working standard in volumetric flask: The appropriate volume of intermediate standard is injected into the expanded area of a volumetric flask containing DI water. The flask is adjusted to volume then inverted three times. An aliquot is transferred to a 5 mL Luer lock syringe or 40 mL VOA vial and placed on the autosampler.

7.9.3 Procedure for preparing standard in 5 mL Luer lock syringe: The volume of stock standard is injected into a 5 mL Luer lock syringe containing DI water.

7.10 Working standards used for LCS/MS/ICV are prepared by diluting intermediate standards in DI water as follows:

Water Analyses

STOCK STANDARD, CONCENTRATION	LCS/MS VOLUME (uL)	ICV VOLUME (uL)	FINAL VOLUME DI Water (mL)	LCS/MS FINAL CONCENTRATION (ug/L)	ICV FINAL CONCENTRATION (ug/L)
VOC mixture (20 ug/mL)	50	125	50	20	50
VOC additional Mix (20 ug/mL)	50	125	50	20	50
Vinyl acetate (20 ug/mL)	50	125	50	20	50
Acrolein (100 ug/mL)	50	50	50	100	100
1,3-Butadiene (20 ug/mL)	50	125	50	20	50
MA Oxy Alt. Source (50-100 ug/mL)	100	100	50	100-200	100-200

Soil Analyses

STOCK STANDARD, CONCENTRATION	LCS/MS VOLUME (uL)	ICV VOLUME (uL)	FINAL VOLUME DI Water (mL)	LCS/MS FINAL CONCENTRATION (ug/Kg)	ICV FINAL CONCENTRATION (ug/Kg)
VOC mixture (20 ug/mL)	5	12.5	5	20	50
VOC additionals Mix (20 ug/mL)	5	12.5	5	20	50
Vinyl acetate (20 ug/mL)	5	12.5	5	20	50
Acrolein (100 ug/mL)	5	5	5	100	100
1,3-Butadiene (20 ug/mL)	5	12.5	5	20	50
MA Oxy Alt. Source (50-100 ug/mL)	10	10	5	100-200	100-200

7.11 50 ng 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)

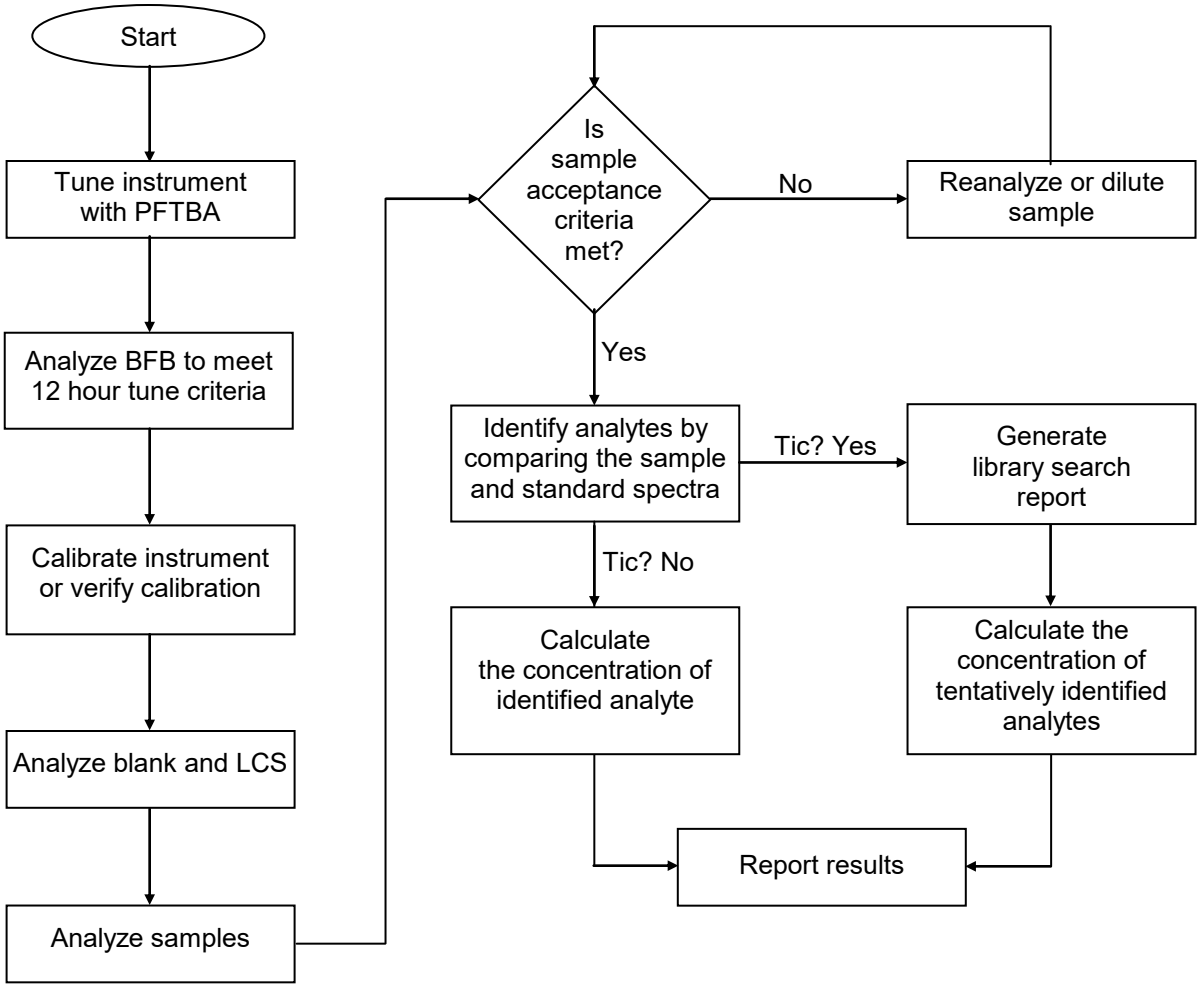
7.15 Concentration of calibration standards may vary depending on, but not limited to, availability, purity, and project requirements, therefore, recipes for standards preparation will be adjusted accordingly. Archon autosamplers add 1 uL of 250 ug/mL internal standards mixture.

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.

7.18 If required, sodium bisulfate is added to low-level soil standards and QC samples.

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



9.0 SAMPLE PREPARATION

9.1 Purge-and-trap procedures are found in Microbac SOP PAT01 for 5030C and 5035A.

10.0 CALIBRATION PROCEDURES

10.1 The GC/MS system is hardware-tuned via auto-tune or manual tune.

10.2 50 ng 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 for each analyte.

10.4 Initial calibration standards are analyzed using the introduction method of choice (5030C or 5035A). Standards used for soil calibration are loaded into 40 mL VOA vials containing 5.00 g (±0.1g) of oven baked reagent sand and utilize a heated purge (40° C).

10.5 Following analysis of the initial calibration, relative response factors (RRF) and average RRF for each surrogate and target analyte are calculated.

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

COMPOUND	MINIMUM $\overline{RRF}$
chloromethane	0.10
1,1-dichloroethane	0.10
bromoform	0.10
chlorobenzene	0.30
1,1,2,2-tetrachloroethane	0.30

10.7 The percent relative standard deviation (%RSD) is calculated for all surrogates and target analytes. The %RSD for all target analytes must be less than 15%,

however 6 analytes designated calibration check compounds (CCC) must have %RSD less than or equal to 30%. The CCC's are:

COMPOUND	ICAL MAX %RSD	CCV MAX %D
1,1-dichloroethene	± 30	± 20
chloroform	± 30	± 20
1,2-dichloropropane	± 30	± 20
toluene	± 30	± 20
ethyl benzene	± 30	± 20
vinyl chloride	± 30	± 20

10.8 Method 8260 Calibration Options

Linear – Using Average RF with RSD ≤ 15%

If the % RSD for all target analytes is less than or equal to 15%, then the response factor is assumed constant over the calibration range. Average response factor, therefore, may be used for quantitation. If the CCC's are not target analytes for a specific project, all required analytes must be ≤ 30% RSD.

If more than 5 calibration levels were analyzed, high and/or low points for poor responding and/or saturated compounds can be removed. The low calibration levels must be at or below the required reporting limit. The curve still must contain a minimum of 5 levels.

The average RF option is the preferred method of GC/MS calibration, since linearity may be assumed throughout the full calibration range. However, linear and quadratic models may be used under the conditions discussed in the following sections. If the % RSD for any target analyte is greater than 15%, one of the following procedures may be employed.

Linear Regression with Coefficient of Determination (COD) r^2 ≥ 0.99

Linear regression is an alternative to average RF, but has the potential for significant bias at the lower concentration levels. If a particular analyte exceeds 15% RSD, then linear regression may be utilized for that analyte. The fit for the equation (r^2) must be ≥ 0.99.

Quadratic Calibration with COD (r^2) ≥ 0.99

Several compounds on the 8260/8270 extended lists and the EPA Appendix IX list do not display consistently linear behavior. Quadratic calibration, employing

at least six calibration points, may be used to improve accuracy for these analytes, particularly at the lower calibration levels, and is a better alternative than linear regression when linear fails the re-fitting test. Quadratic calibration must never be used to compensate for a poorly maintained GC/MS system, and should not be used for analytes with a previous history of linear performance. Quadratic regression can be employed provided the COD (r^2) is ≥ 0.990. Those analytes utilizing first and/or second order calibration are noted on the initial calibration report.

NOTE: Origin not forced when using linear and quadratic regressions. Quadratic regression cannot be used to extend the calibration range.

For samples received from California, quadratic models for analytes that normally display linear responses in the calibration ranges will not be employed. Listed below are 8260 compounds that do not consistently exhibit linear behavior:

8260 Compounds	**Additional 8260 Compounds**	
vinyl acetate	t-butyl alcohol	paraldehyde
vinyl chloride	1,4-dioxane	1-bromopropane
2-chloroethylvinyl ether	propionitrile	isobutyl alcohol
naphthalene	tetrahydrofuran	1-butanol
acetone	acrolein	1-chlorohexane
	iodomethane	

10.9 Following the initial calibration an ICV is performed. Acceptance criteria is ± 25% drift.

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 8.6.

10.11.2 CCC's in Section 8.7 ≤ 20 % difference when using average response factor or ≤ 20 % drift when using regression fit. Non-CCC's must be ≤ 20% difference/drift with sporadic marginal failures permitted to ≤ 40 % difference/drift.

The following poor performing analytes may exceed ± 40% difference/drift:

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	

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 require reanalysis using a multi-point calibration curve.

10.13 The separation of 2-chlorotoluene and 4-chlorotoluene will be evaluated for standards and QC samples using the “evaluation resolution” option in Chemstation. The resolution between 2-chlorotoluene and 4-chlorotoluene must be greater than 25% as evaluated by Chemstation.

10.14 Refer to Section 13.0 for quality control requirements and corrective action.

10.15 Calibration training materials are available on the intranet home page in the “General” links section, “Calibration Training”. Review of “Calibration Models” and ”The Effect that Saturation of the Detector has Upon Calibration” are recommended training for all new analysts. There are additional calibration training materials available through the same link on the homepage.

11.0 ANALYTICAL PROCEDURES

11.1 Prior to sample analysis, instruments must pass tuning and calibration criteria per Section 8.0.

11.2 Method blank: Analyzed prior to environmental samples. Method blanks are matrix specific.

11.2.1 Preparation of method blank

Water blank preparation: Fill a 40 mL VOA vial with UV-treated DI water (headspace not present). Place vial in Autosampler. Archon autosampler adds 1 uL of 250 ug/mL internal and surrogate standard mixtures.

Soil blank preparation: 5.00 g (±0.1g) of oven baked reagent sand is weighed into a tared 40 mL VOA vial containing a stir bar. 5 mL of UV-treated DI water is added to the vial. The vial is placed on the Autosampler. The Archon autosampler adds 5 mL of UV-treated DI water containing 1 uL of 250 ug/mL internal and surrogate standards mixtures. A 2 minute preheat (40° C) and heated purge (40° C) is utilized.

Middle-level extraction blank: 5.00 g (±0.1g) of oven baked reagent sand is weighed into a tared 40 mL VOA vial. 10 mL of methanol is added to the vial. The vial is shaken then allowed to settle. A 50x dilution is performed on the extract. The Archon 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 8260B target analytes is analyzed. An LCS/LCS duplicate analyses is performed when the client does not provide sufficient volume for MS/MSD analyses.

Water and low-level soil LCS preparation: Refer to Section 7.0. **NOTE:** For low-level soil LCS, 2 minute preheat and heated purge (40° C) is utilized.

Middle-level extraction LCS: 5.00 g (±0.1g) of oven baked reagent sand is weighed into a tared 40 mL VOA vial. 8.5 mL of methanol and 0.5 mL of the LCS mixtures are added to the vial (**NOTE:** 8.5 mL methanol volume is dependent upon the number of LCS mixtures added; extract final volume is 10 mL). The vial is shaken then allowed to settle. A 50x dilution is performed on the extract. The dilution is loaded on the Autosampler and analyzed.

11.4 MS/MSD are analyzed when the client provides appropriate sample volume.

11.4.1 Water MS/MSD preparation: Refer to Section 7.0 with the exception of sample used in place of DI water.

11.4.2 Low-level and mid-level soil MS/MSD preparation: Refer to Section 7.0 with the exception of 5.00 g (± 0.1 g) sample used in place of reagent sand.

11.5 Sample/sample duplicate analyses may be analyzed providing there is appropriate volume. Sample/sample duplicate analyses are generally associated with concentrated soil/waste samples and oils and are used to determine precision.

11.6 Samples are prepared per Microbac SOP PAT01.

11.7 Samples are analyzed within the 12 hour tune, which begins with the injection of BFB. At the end of tune time, a new BFB, blank, CCV, and LCS must be injected.

11.8 Once sample analysis is complete, a computer generated quantitation report containing all target analytes and their concentrations is generated. Also, detailed spectrum are generated for all target analytes detected above a nominal amount.

11.9 Qualitative analysis

An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). These standard reference spectra are obtained through analysis of the calibration standards. Two criteria must be satisfied to verify identification: (1) elution of the sample component at the same GC relative retention time (RRT) as the standard component; and (2) correspondence of the sample component and the standard component mass spectrum.

11.9.1 The sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run within the same 12 hours as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT must be assigned by using extracted ion current profiles for ions unique to the component of interest.

11.9.2 All ions present in the standard mass spectrum at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum. The relative intensities of the characteristic ions must agree within 30% of the relative intensities of these ions in the reference spectrum. Analyst judgment is permitted even if these criteria are not met. The positive identification of a hit should not be made based solely on the criteria mentioned above.

11.10 If the response for any target analyte exceeds the initial calibration range, the sample must be diluted. Dilutions are prepared so that the majority of

compounds above the calibration range fall near the midpoint of the calibration. Water and mid-level soil dilutions are prepared by using syringes or pipets to transfer aliquots of sample into a volumetric flask containing DI water. Examples of water dilutions are presented below.

DILUTION	SAMPLE VOLUME (mL)	DI WATER VOLUME (mL)	FINAL DILUTION VOLUME (mL)
10x	5	45	50
50x	1	49	50
100x	0.5	49.5	50
1000x	0.05	49.95	50

Low-level soil dilutions are prepared by weighing an aliquot less than 5.00 g in a 40 mL VOA vial. Examples of low-level soil dilutions:

DILUTION	SAMPLE AMOUNT (g)	DI WATER VOLUME (mL)
2x	2.5	5
2.5x	2.0	5
5x	1.0	5

11.10.1 Low level soils collected via 5035A must utilize the mid-level aliquot for dilutions.

Mid-level soil dilutions are prepared by diluting an aliquot of the methanol extract in a volumetric flask. Examples of mid-level soil dilutions:

DILUTION	METHANOL EXTRACT VOLUME (mL)	DI WATER VOLUME (mL)	FINAL VOLUME (mL)
50x	1	49	50
100x	0.5	49.5	50
500x	0.1	49.9	50
1000x	0.05	49.95	50

11.11 The raw data is processed using the chem station software and the data is uploaded into the LIMS. The laboratory then performs a primary and secondary review of the raw data and quality control forms.

11.12 Tentatively identified compounds (TIC): For samples containing components not associated with the calibration standards, a library search may be performed for the purpose of tentative identification. Guidelines for making tentative identification are:

Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) must be present in the sample spectrum.

The relative intensities of the major ions must agree within ± 40% for TIC's. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% -70%.)

Molecular ions present in the reference spectrum must be present in the sample spectrum.

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

Ions present in the reference spectrum but not in the sample spectrum must be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.13 Wipe Sample analysis: Wipes must be stored in 40 mL VOA vials containing 10 mL of methanol after collection. Prior to analysis, a glass rod (or similar) is inserted through the septum to completely immerse the wipe in methanol. Vortex the vial for 20 seconds. Remove ample volume of methanol by inserting the needle of a syringe (or similar) through the septum for a 50x dilution. Prepare as per the mid-level extraction procedures.

12.0 DETAILS OF CALCULATIONS

12.1 Relative response factor (RRF):

$$RRF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

where:

A_x = Area of the characteristic ion for the surrogate or compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard.
C_x = Concentration of the surrogate or compound being measured.

12.2 Average RRF $\left(\overline{RRF}\right)$:

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

12.3 Standard deviation (s):

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

12.4 Percent relative standard deviation (%RSD):

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

where:

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

s = standard deviation(s): $s = \sqrt{\frac{\sum\left(x - \bar{x}\right)^2}{n-1}}$

12.5 Percent recovery (%R)

LCS, surrogate:

$$\%R = \left(\frac{C_x}{C_t}\right)100$$

where:

C_x = the concentration of the analyte in the LCS
C_t = the theoretical spike concentration.
%R = percent recovery

MS/MSD:

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

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):

$$\%D = \left[\frac{(C_t - C_x)}{C_t}\right]100$$

where:

C_t = True concentration of the analyte or surrogate in the standard
C_x = Measured concentration of analyte or surrogate in the standard

12.8 Coefficient of correlation (r):

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

where:

X = individual values of the independent variable, i.e. concentration
Y = individual values of the dependent variable, i.e. response
n = number of pairs of data

12.8 Coefficient of determination (COD):

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

12.9 Sample concentration using $\overline{RRF}$:

Water (ug/L):

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

where :

A_x = area of characteristic ion for compound being measured
I_s = amount of internal standard injected (250ng)
A_{is} = area of characteristic ion for the internal standard
$\overline{RRF}$ = mean relative response factor for compound being measured
V_o = volume of water purged (10mL)
DF = dilution factor

Low-level soil/sediment:

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

where :

A_x, I_s, A_{is}, $\overline{RRF}$, = same as for water
W_s = weight of sample purged in grams
D = % dry weight of sample divided by 100, or 1 for a wet-weight basis

Medium-level soil/sediment:

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

where :

A_x, I_s, A_{is}, $\overline{RRF}$, V_o, DF= same as for water
W_s = weight of sample extracted in grams
V_t = volume of total extract (mL) = $V_m + \left[(W_s)\left(\frac{100-D}{100}\right)\right]$
V_m = adjusted volume of solvent
V_i = volume of extract added (mL) for purging
D = % dry weight of sample (not applicable for a wet-weight basis)

12.10 Linear calibration calculations:

The response ratio is plotted vs. the concentration ratio giving a linear equation:

$$y = mx + b$$

where:

y = Response ratio = Response(x)/Response(istd) = R_x/R_{istd}
x = Concentration ratio = Conc(x)/Conc(istd) = C_x/C_{istd}
And m and b are the slope and intercept from the regression equation

For a given response ratio we can solve for C_x/C_{istd} :

$$C_x/C_{istd} = [R_x/R_{istd} - b]/m$$

Use equations 12.13 or 12.14 to calculate the unknown concentration, C_x.

12.11 Quadratic calibration calculations:

The response ratio is plotted vs. the concentration ratio giving a quadratic equation:

$$y = ax^2 + bx + c$$

OR

$$ax^2 + bx + (c - y) = 0$$

Solving for x using the quadratic equation:

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

where:

y = Response ratio = Response(x)/Response(istd) = R_x/R_{istd}
x = Concentration ratio = Conc(x)/Conc(istd) = C_x/C_{istd}
a ,b, c are constants from the regression equation
Use equations 12.13 or 12.14 to calculate the unknown concentration, C_x

12.12 Solving for the concentration in water sample:

For a given concentration ratio, compute the unknown, C_x

$$C_x = (C_{is})(C_x/C_{istd})(V_f/V_i)(DF)(1000)$$

where:

C_{istd} = concentration of the internal standard (ug/mL)
V_f = final sample (extract) volume (mL)
V_i = initial sample volume (mL)
DF = dilution factor
C_x = concentration of the sample in ug/L

12.13 Solving for the concentration in soil sample:

$$C_x = (C_{is})(C_x/C_{istd})(V_f/W_i)(DF)(1000)$$

where:

C_{istd} = concentration of the internal standard (ug/mL)
V_f = final sample (extract) volume (mL)

W_i = initial sample volume (mL)
DF = dilution factor
C_x = concentration of the sample (ug/Kg) (as received)

12.14 Tentatively identified compounds (TIC) estimated concentration determination:

TIC water (ug/L):

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

where :

A_x = total area of the peak from the total ion chromatogram
I_s = amount of internal standard injected (250ng)
A_{is} = total area of the internal standard from the total ion chromatogram
$\overline{RRF}$ = 1
V_o = volume of water purged (10mL)
DF = dilution factor

TIC low-level soil/sediment:

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

where :

A_x, I_s, A_{is}, $\overline{RRF}$, = same as for water
W_s = weight of sample purged in grams
D = % dry weight of sample divided by 100, or 1 for a wet-weight basis

TIC medium-level soil/sediment:

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

where :

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

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

V_i = volume of extract added (mL) for purging
D = % dry weight of sample divided by 100, or 1 for a wet-weight basis
DF = dilution factor
V_m = volume of methanol added (mL)

12.15 Wipe:

$$ug/wipe = (C)(D_F)(E_V)$$

where:

C = extract concentration, ug/L
D_F = dilution factor
E_V = extract volume, L/wipe

NOTE: E_V assumed to be 0.01 L

13.0 QUALITY CONTROL REQUIREMENTS

13.1 The quality control procedures discussed in this section are intended to monitor and control the entire analytical process. Batch quality samples are specified for ICAL, MB, LCS, MS, MSD, laboratory duplicates (LD), and surrogate compounds. Additional procedures were defined in Section 8.0 for initial calibration, ICV using a second source, and CCV, and are included in the overall review process. The procedures, required frequency, acceptance criteria, and the required corrective action measures are outlined in Table 7.

13.2 Workgroups are analytical batches that contain instrument performance checks (BFB), calibration standards (ICAL, ICV, CCV), QC samples, and client samples.

13.3 Workgroups are comprised of:

- Instrument performance check: BFB tune evaluation to verify detector is working properly
- ICAL/CCV standards: used to calibrate instrument or verify accuracy of the calibration curve
- ICV: standard from an alternate source used to verify accuracy of the calibration curve

- Method blank: verify system is free of contaminants and interferences
- LCS/LCSD: verify precision and accuracy of the system
- MS/MSD: measure matrix effect of environmental sample on target analytes; measure precision
- Sample/sample duplicate: dual analysis of environmental system to measure precision
- Environmental sample: samples submitted for analysis

13.4 Method blank analyzed per method requirements. Target analytes must be less than ½ the RL. All blanks are evaluated down to the current MDL for the presence of target analytes. Any amount of target analytes found in the blank at a level greater than the current MDL are reported in the LIMS and these values will appear on the QC summary sheet for the batch.

13.5 The LCS must be evaluated using acceptance criteria listed in Tables 2 and 3, as well as any project specific criteria. Upon completion of a batch of samples, LCS summary reports are generated by the analyst, which compare the actual recoveries to the applicable acceptance ranges for the samples in the batch. The standard laboratory limits specified in Tables 2 and 3 are used in the absence of a project QAPP or program specified control limits. If more than 10% of the LCS analytes are out of the laboratory limits, the analyst must stop the analysis, prepare an NCR, and contact the department supervisor for the appropriate corrective action. If any of the identified project specific chemicals of concern (COC) are outside the control limits, the analyst must stop the analysis and prepare an NCR to be reviewed by the department supervisor.

13.6 The MS/MSD is analyzed per method requirements. MS/MSD results are included in the QC summary report and are used to monitor matrix accuracy and precision. For MS/MSD, Sample/sample duplicate, failure to meet surrogate and internal standard areas acceptance criteria does not necessarily warrant corrective action. Sample MS/MSD or sample/duplicate results can be used to confirm sample matrix interference. In obvious cases of error, reanalysis would be performed.

13.7 When ICAL acceptance criteria are not met, corrective action may include (but is not limited) to the following:

- Evaluate individual data points and reanalyze
- Evaluate calibration standards and reanalyze
- Prepare fresh calibration standards and reanalyze
- Perform instrument maintenance to include but not limited to:
  - Reanalyze calibration curve
  - Tune instrument

- Replace trap
- Bake analytical system
- Replace column
- Replace transfer line(s)
- Service auto-sampler, sample concentrator, gas chromatograph, and/or mass spectrometer

- Qualify results and address in case narrative

13.8 When CCV acceptance criteria are not met, corrective action may include (but not, limited to) the following:

- Reanalyze CCV
- Prepare fresh standards
- Bake analytical system
- Perform instrument maintenance

13.9 Surrogate is added to all standards, QC samples, and environmental samples. Table 6 lists surrogate acceptance limits.

13.10 Storage blanks are placed in sample refrigerators and freezers to monitor potential cross contamination. Storage blanks consists of 40 mL of analyte free DI water or 5 mL DI water and 5.00 g of sand stored for 14 days in each VOA refrigerator/freezer. Analyses are performed via Method 8260 with results quantitated to the MDL. Storage blanks are prepared weekly and logged into the LIMS laboratory account. Weekly, (after storage blanks have been stored for two full weeks) storage blanks are analyzed via 8260 (storage blanks must be analyzed within the 12 hour tune time).

Target analytes must be less than ½ the reporting limit with the exception of common lab contaminants. Common lab contaminants must be less than the reporting limit. During primary review of the data, the analyst will review storage blank results to ensure acceptance criteria are met. If acceptance criteria are not met the analyst must initiate corrective action. Corrective action begins with determining the non-compliant analyte(s) and recording any known reason for the failure then reanalysis of the duplicate vial of the storage blank. If reanalysis of the storage blank yields results within acceptance criteria then no further corrective action is required. If the reanalysis results confirm the initial analysis results or the reason for the initial failure is not evident then a Form NC02 is initiated. After primary review, results are uploaded to the LIMS. The laboratory will conduct an internal investigation and assess impact on associated samples if these criteria are not met with the next group of storage blanks analyzed. The laboratory will attempt to identify the source of contamination, and evaluate the impact on data reported for the contaminant during the period of storage. Clients

may be contacted based on the investigation, if the QAO judges that to be necessary.

13.11 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.11.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Table 7 does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Table 7.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch Figure 1. A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.11.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RAC, corrections, corrective action(s) and evidence of effectiveness.

13.11.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

13.12 Table 7 contains method 8260B quality control criteria.

13.13 LCS control limits are reviewed annually.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data review:

14.1.1 All data undergoes a 100% primary review to ensure method and project specific compliance, reduce the data into reportable results, and generate appropriate QC forms. All items in Figure 1 (data review checklist) are reviewed and results are uploaded to the LIMS.

14.1.1.1 Data may be reviewed by an analyst other than the primary analyst provided the reviewing analyst's initials are recorded on the "Data Checklist".

14.1.2 Following the primary review the data undergoes a 100% peer review. All items in 14.1.1 are repeated by the peer. The peer review is performed by the supervisor or designee.

14.2 Data reporting:

14.2.1 Following peer review all uploaded results are reviewed, verified, and qualified.

14.2.2 Default reporting units are "ug/L" for water and "ug/Kg" for soil/sediments/oils.

14.2.3 All uploaded results are uploaded to a maximum number of significant figures dictated by the LIMS. The number of significant figures in the final report vary per project requirements.

14.2.4 Dilution and sample matrix confirmation analyses are uploaded into the LIMS and per the client's request may be reported as separate analyses or combined (concatenated) into one set of results.

14.3 Quantitative results between the MDL and RL are qualified as "estimated" if requested by the client.

14.4 Refer to Microbac SOP 41 for acceptable procedure on manual integration if necessary.

14.5 Electronic run logs and preparation logs are reviewed electronically.

15.0 PREVENTIVE MAINTENANCE

15.1 Gas pressures are monitored daily. Other maintenance performed as needed. Laboratory maintenance log books maintained per instrument.

15.2 Instrument configuration and maintenance is recorded in the instrument maintenance log book.

15.3 Trouble-shooting involves, but is not limited to, direct injections, chromatography review, evaluating contamination, standards recoveries, injection port maintenance, and leak check.

15.4 Vendor instrument repair reports will be included in maintenance log.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician

checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2 The waste streams are as follows:

- Volatile Laboratory – non-halogenated solvents, solid waste (methanol)

16.3 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33, Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. Our procedures comply with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal hazardous waste that this is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

17.0 REFERENCES

17.1 *Test Methods for Evaluating Solid Waste,* SW-846, US-EPA, Office of Solid Waste, including updates I, II, III, IV:

8000B	December 1996
8000C	March 2003
8260A	September 1994
8260B	December 1996
5030A	July 1992
5030B	December 1996
5030C	May 2003
5035A	July 2002

17.2 AFCEE 1998 QAPP, Version 3.0, March 1998

17.3 AFCEE 2001 QAPP, Version 3.1, August 2001

17.4 AFCEE 2005 QAPP, Version 4.0, February 2005

17.5 U.S. EPA, 40 CFR, Part 136, October 26, 1984

17.6 “Standards Methods 21st Edition” Method 6200B

17.7 Microbac SOP PAT01 “Methods 5030 and 5035 Purge and Trap for Volatile ` Organics”

17.8 Microbac SOP 45 “Method Validation Procedures”

17.9 Microbac SOP 41 “Manual Integration of Chromatographic Peaks”

17.10 Microbac SOP 33 “Laboratory Waste Management”

17.11 Microbac SOP LQAP “Laboratory Quality Assurance Plan”

Appendix I
The Analysis of 1,4-Dioxane by Selected Ion Monitoring (SIM), Revision 1

1. Selected ion monitoring (SIM) is used in the acquisition of 1,4-dioxane and associated internal and surrogate standards. The following lists primary and secondary ions:

Parameter	Primary ion	Secondary ion
1,4-dioxane	88	58
1,4-dioxane-d8 (IS)	96	70
dibromofluoromethane (SS)	111	113
1,2-dichloroethane-d4 (SS)	65	67

IS: denotes internal standard
SS: denotes surrogate standard

2. Sample preparation: Water: Allow sample to warm to ambient temperature. Place sample vial in auto-sampler. Auto-sampler transfers 10mL of sample for purging. Heated purge is performed utilizing a 4 minute pre-heat at 60C then a 9 minute purge at 60C. After analysis record sample pH from opened vial. If possible, dilutions performed using un-opened vial.

3. Sample preparation: Soil: Refer to Microbac SOP PAT01.

NOTE: Standards and QC samples also utilize a heated purge; auto-sampler adds internal and surrogate standards.

4. The suggested calibration range is 2 ug/L to 200 ug/L for water; 2 ug/Kg to 200 ug/Kg for soil. The following lists suggested QC acceptance criteria:

MATRIX	RL	MDL*	ACCURACY** (%R)	PRECISION** (RPD)	TRUE VALUE, LCS, MS/MSD	SURROGATE LIMITS** (%R)	SURROGATE TRUE VALUE
Water (ug/L)	2.0	1.0	60-140	30	10	50-150	2.5
Soil (ug/Kg)	2.0	1.0	50-150	30	10	50-150	2.5

* denotes verified MDL (verified annually)
** denotes advisory limits (control limits may vary)

5. Following is corrective action procedures and QC acceptance criteria for SIM 1,4-dioxane analysis.

Quality Control Criteria
Volatile GC/MS SIM, 1,4-Dioxane

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION (6)
Mass Spectral ION Intensities (BFB criteria)	Every 12 hours prior to ICAL, ICV, or CCV	Per Table 5	Evaluate, re-inject, re-tune, instrument maintenance
Initial Calibration (ICAL)	When Continuing Calibration is out of control of when system conditions have been altered	%RSD ≤ 30%	Evaluate and use linear or higher order calibration equation, perform corrective action (1)
Second Source Calibration Verification (ICV)	After each initial calibration	% D ≤ 30 %	Reanalyze ICV; upon second failure repeat initial calibration (1)
Continuing Calibration Verification (CCV)	Every 12 hours	%D ≤ 30%	Rerun CCV once more, upon 2nd failure perform corrective action (1)
Method Blank (MB)	One per matrix/batch; maximum of 20 samples per batch	< ½ RL	Reanalyze blank, perform corrective action.
Laboratory Control Sample/Sample Duplicate (LCS/LCSD)	One per matrix/batch; maximum of 20 samples per batch	Within designated ranges (1,2)	Investigate, evaluate, perform corrective action (5)
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per matrix/batch; maximum of 20 samples per batch	Within designated ranges (1,2)	Evaluate, none, if errors not detected
Surrogate Spike	Every sample, standard, and quality control sample	Within designated ranges (1)	Reanalyze if %R < 10% 1 outlier permitted providing %R > 10%.

(1) Evaluation criteria is 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.

Appendix II
8260A and 8260B Suggested Quantitation Ions

COMPOUND NAME	SUGGESTED PRIMARY CHARACTERISTIC ION	SUGGESTED SECONDARY CHARACTERISTIC ION	MICROBAC'S PRIMARY CHARACTERISTIC ION
fluorobenzene (internal std)	**96**	**77**	**96**
dichlorodifluoromethane	85	87	85
chloromethane	50	52	50
vinyl chloride	62	64	62
1,3-butadiene	N/A	N/A	54
bromomethane	94	96	94
chloroethane	64	66	64
trichlorofluoromethane	151	101, 153	101
diethyl ether	74	45, 59	59
isoprene	67	53	67
acrolein	56	55, 58	56
trichlorotrifluoromethane	101	151	101
acetone	58	43	43
1,1-dichloroethene	96	61, 63	96
t-butyl alcohol	N/A	N/A	59
dimethyl sulfide	62	47	62
iodomethane	142	127, 141	142
methyl acetate	N/A	N/A	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	N/A	N/A	41
trans-1,2-dichloroethene	96	61, 98	96
n-hexane	N/A	N/A	57
diisopropyl ether	N/A	N/A	45
vinyl acetate	43	86	43
1,1-dichloroethane	63	65, 83	63
ethyl-t-butyl ether	N/A	N/A	59
2-butanone	72	43	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	N/A	N/A	122
ethyl acetate	88	43, 45, 61	43
bromochloromethane	128	49, 130	128
methacrylonitrile	41	67, 39, 52, 66	67
isobutyl alcohol	43	41, 42, 74	73
tetrahydofuran	N/A	N/A	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	N/A	N/A	73
carbon tetrachloride	117	119	117
1,2-dichloroethane-d4 **(surrogate)**	65	67	65
Heptane	N/A	N/A	57

Appendix II (continued)

COMPOUND NAME	SUGGESTED PRIMARY CHARACTERISTIC ION	SUGGESTED SECONDARY CHARACTERISTIC ION	MICROBAC'S PRIMARY CHARACTERISTIC ION
1,2-dichloroethane	62	98	62
1-butanol	N/A	N/A	56
benzene	78	77, 52	78
trichloroethene	95	97, 130, 132	130
methylcyclohexane	N/A	N/A	83
1,2-dichloropropane	62	112	62
Methyl methacrylate	69	41, 100, 39	41
1,4-dioxane	88	58, 43, 47	88
bromodichloromethane	83	85, 127	83
2-nitropropane	46	N/A	43
dibromomethane	93	95, 174	93
2-chloroethylvinyl-ether	63	65, 106	63
4-methyl-2-pentanone	100	43, 58, 85	58
cis-1,3-dichloropropene	75	77, 39	75
dimethyl disulfide	79	94	79
chlorobenzene-d5 (internal std)	**117**	**82**	**117**
toluene-d8 **(surrogate)**	98	100	98
toluene	92	91	91
ethyl methacrylate	69	41, 99, 86, 114	69
Paraldehyde	N/A	N/A	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	91	106	106
m+p-xylene	106	91	106
cyclohexanone	N/A	N/A	55
o-xylene	106	91	106
styrene	104	78	104
bromoform	173	175, 254	173
isopropylbenzene	105	120	105
1,4-dichlorobenzene-d4 (internal std)	**152**	**115, 150**	**152**
1,1,2,2-tetrachloroethane	83	131, 85	83
p-bromofluorobenzene **(surrogate)**	95	174, 176	95
1,2,3-trichloropropane	75	77, 110	110
trans-1,4-dichloro-2-butene	53	88, 75	53
n-propyl-benzene	91	120	91
bromobenzene	156	77, 158	156
1,3,5-trimethylbenzene	105	120	105
2-chlorotoluene	91	126	91
4-chlorotoluene	91	126	91
alpha-methyl-styrene	118	103	118
tert-butyl-benzene	119	91, 134	119
1,2,4-trimethylbenzene	105	120	105
sec-butyl-benzene	105	134	105
p-isopropyl-toluene	119	134, 91	119

Appendix II (continued)

COMPOUND NAME	SUGGESTED PRIMARY CHARACTERISTIC ION	SUGGESTED SECONDARY CHARACTERISTIC ION	MICROBAC'S PRIMARY CHARACTERISTIC ION
1,3-dichlorobenzene	146	111, 148	146
1,4-dichlorobenzene	146	111, 148	146
n-butyl-benzene	91	92, 134	91
1,2-dichlorobenzene	146	111, 148	146
1,2-dibromo-3-chloropropane	75	115, 157	157
1,2,4-trichlorobenzene	180	182, 145	180
hexachlorobutadiene	225	223, 227	225
naphthalene	128	127	128
1,2,3-trichlorobenzene	180	182, 145	180

Appendix III
8260B Ultra Low Level Water Purge-and Trap Analysis ("8260UL")

1.0 Water samples are analyzed using a 20mL purge volume to achieve reporting limits approximately one hundred times lower than standard 8260B reporting limits.

2.0 The following lists 8260UL target analytes and performance data:

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	70-130	20	0.05	0.25	0.5	0.05-10
1,1,1-trichloroethane	71-55-6	70-130	20	0.025	0.05	0.5	0.05-10
1,1,2,2-tetrachloroethane	79-34-5	70-130	20	0.05	0.25	0.5	0.05-10
1,1,2-trichloroethane	79-00-5	70-130	20	0.025	0.05	0.5	0.05-10
1,1-dichloroethane	75-34-3	70-130	20	0.025	0.05	0.5	0.05-10
1,1-dichloroethene	75-35-4	70-130	20	0.025	0.05	0.5	0.05-10
1,1-dichloropropene	563-58-6	70-130	20	0.025	0.05	0.5	0.05-10
1,2,3-trichlorobenzene	87-61-6	70-130	20	0.05	0.1	0.5	0.05-10
1,2,3-trichloropropane	96-18-4	70-130	20	0.025	0.05	0.5	0.05-10
1,2,4-trichlorobenzene	120-82-1	70-130	20	0.025	0.05	0.5	0.05-10
1,2,4-trimethylbenzene	95-63-6	70-130	20	0.25	0.5	0.5	0.05-10
1,2-dibromo-3-chloropropane	96-12-8	70-130	20	0.25	0.5	0.5	0.05-10
1,2-dibromoethane	106-93-4	70-130	20	0.1	0.2	0.5	0.05-10
1,2-dichlorobenzene	95-50-1	70-130	20	0.025	0.05	0.5	0.05-10
1,2-dichloroethane	107-06-2	70-130	20	0.025	0.05	0.5	0.05-10
1,2-dichloropropane	78-87-5	70-130	20	0.025	0.05	0.5	0.05-10
1,3,5-trimethylbenzene	108-67-8	70-130	20	0.025	0.05	0.5	0.05-10
1,3-dichlorobenzene	541-73-1	70-130	20	0.025	0.05	0.5	0.05-10
1,3-dichloropropane	142-28-9	70-130	20	0.05	0.1	0.5	0.05-10
1,4-dichlorobenzene	106-46-7	70-130	20	0.025	0.05	0.5	0.05-10
2,2-dichloropropane	594-20-7	70-130	20	0.05	0.1	0.5	0.05-10
2-chlorotoluene	95-49-8	70-130	20	0.025	0.05	0.5	0.05-10
4-chlorotoluene	106-43-4	70-130	20	0.025	0.05	0.5	0.05-10
benzene	71-43-2	70-130	20	0.025	0.05	0.5	0.05-10
bromobenzene	108-86-1	70-130	20	0.025	0.05	0.5	0.05-10
bromochloromethane	74-97-5	70-130	20	0.05	0.1	0.5	0.05-10

8260UL target analytes (continued)

bromodichloromethane	75-27-4	70-130	20	0.025	0.05	0.5	0.05-10
bromoform	75-25-2	70-130	20	0.1	0.25	0.5	0.05-10
bromomethane	74-83-9	70-130	20	0.025	0.05	0.5	0.05-10
carbon disulfide	75-15-0	70-130	20	0.05	0.10	0.5	0.05-10
carbon tetrachloride	56-23-5	70-130	20	0.025	0.05	0.5	0.05-10
chlorobenzene	108-90-7	70-130	20	0.025	0.05	0.5	0.05-10
chloroethane	75-00-3	70-130	20	0.025	0.05	0.5	0.05-10
chloroform	67-66-3	70-130	20	0.05	0.1	0.5	0.05-10
chloromethane	74-87-3	70-130	20	0.05	0.1	0.5	0.05-10
cis-1,2-dichloroethene	156-59-2	70-130	20	0.025	0.05	0.5	0.05-10
cis-1,3-dichloropropene	10061-01-5	70-130	20	0.025	0.05	0.5	0.05-10
dibromochloromethane	124-48-1	70-130	20	0.025	0.05	0.5	0.05-10
dibromomethane	74-95-3	70-130	20	0.025	0.05	0.5	0.05-10
dichlorodifluoromethane	75-71-8	70-130	20	0.025	0.05	0.5	0.05-10
ethyl benzene	100-41-4	70-130	20	0.025	0.05	0.5	0.05-10
hexachlorobutadiene	87-68-3	70-130	20	0.025	0.05	0.5	0.05-10
isopropylbenzene	98-82-8	70-130	20	0.025	0.05	0.5	0.05-10
m+p-xylene **	108-38-3 + 106-42-3	70-130	20	0.025	0.05	1.0	0.05-10
methylene chloride	75-09-2	70-130	20	0.5	1	0.5	0.05-10
naphthalene	91-20-3	70-130	20	0.05	0.1	0.5	0.05-10
n-butylbenzene	104-51-8	70-130	20	0.025	0.05	0.5	0.05-10
o-xylene	95-47-6	70-130	20	0.025	0.05	0.5	0.05-10
p-isopropyl-toluene	99-87-6	70-130	20	0.025	0.05	0.5	0.05-10
propylbenzene	103-65-1	70-130	20	0.025	0.05	0.5	0.05-10
sec-butylbenzene	135-98-8	70-130	20	0.025	0.05	0.5	0.05-10
styrene	100-42-5	70-130	20	0.025	0.05	0.5	0.05-10
tert-butylbenzene	98-06-6	70-130	20	0.05	0.1	0.5	0.05-10
tetrachloroethene	127-18-4	70-130	20	0.025	0.05	0.5	0.05-10
Toluene	108-88-3	70-130	20	0.025	0.05	0.5	0.05-10
trans-1,2-dichloroethene	156-60-5	70-130	20	0.025	0.05	0.5	0.05-10
trans-1,3-dichloropropene	10061-02-6	70-130	20	0.025	0.05	0.5	0.05-10
trichloroethene	79-01-6	70-130	20	0.025	0.05	0.5	0.05-10
trichlorofluoromethane	75-69-4	70-130	20	0.025	0.05	0.5	0.05-10
vinyl chloride	75-01-4	70-130	20	0.1	0.25	0.5	0.05-10

3.0 8260UL standards preparation:

3.1 Intermediate calibration standards: Primary calibration standards in Section 7.0 diluted to prepare intermediate standards as follows:

PRIMARY CALIBRATION STANDARD	PRIMARY CALIBRATION STANDARD CONCENTRATION	VOLUME	FINAL VOLUME (METHANOL)	INTERMEDIATE STANDARD FINAL CONCENTRATION
502.2 CAL200 Mega Mix	200 ug/mL	50 uL	10 mL	1 ug/mL
Custom Mix 3	250 ug/mL	40 uL	10 mL	1 ug/mL
Custom Mix 2	250 ug/mL	40 uL	10 mL	1 ug/mL
VOC Mix 6*	2000 ug/mL	5 uL	10 mL	1 ug/mL*
Freon 113*	1000 ug/mL	10 uL	10 mL	1 ug/mL*

* Combined to create one intermediate standard (“8260 Gases”)

3.2 Intermediate internal and surrogate standards: 25 ug/mL; prepared by diluting 100 uL of the primary standards into 10 mL of methanol.

3.3 Working standards used for instrument calibration and calibration verification are prepared by diluting intermediate standards in DI water as follows:

Water Initial Calibration Standards, ug/L
(suggested preparation)

STOCK STANDARD CONCENTRATION	WORKING STANDARD, ug/L						
	0.05	0.10	0.25	0.50	1.0**	5.0	10
VOA Mega mix (8260 Standard) 50 ug/mL	5 uL	10 uL	12.5 uL	25 uL	50 uL	5 uL	10 uL
Custom Gases 50 ug/mL	5 uL	10 uL	12.5 uL	25 uL	50 uL	5 uL	10 uL
Custom Mix 2 50 ug/mL	5 uL	10 uL	12.5 uL	25 uL	50 uL	5 uL	10 uL
Custom Mix 3 50 ug/mL	5 uL	10 uL	12.5 uL	25 uL	50 uL	5 uL	10 uL
Surrogate Standard 50 ug/mL	5 uL	10 uL	12.5 uL	25 uL	50 uL	5 uL	10 uL
Final volume, DI water (mL)	100	100	50	50	50	50	50

** Denotes CCV

3.4 Working standards used for LCS/MS/ICV are prepared by diluting intermediate standards Section 7.0 in DI water as follows:

Water Analyses

STOCK STANDARD CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL)	FINAL CONCENTRATION (ug/L)
VOC mixture 8260 QC (20 ug/mL)	2.5	100	0.5
VOC additionals Adds QC (20 ug/mL)	2.5	100	0.5
8260 extra additionals (20 ug/mL)	2.5	100	0.5

4.0 Configure instrument per the following:

GC/MS PURGE AND TRAP PARAMETERS	
purge time	11 minutes
dry purge time	1 minute
desorb preheat	245° C
desorb	1 minute at 250° C
bake	10 minutes at 260° C
LSC 2000, 3000 temps	valve 150° C, lines: 150° C
Archon 5100 temps	valve 95° C, lines 120° C
GAS CHROMATOGRAPH PARAMETERS	
carrier gas	helium, 99.999 %
injector temperature	220° C
oven temperature program	35° C (hold 4 minutes)
oven ramp	10° C/minute to 240° C (hold 2 minutes)
split ratio	25 : 1
MASS SPECTROMETER PARAMETERS	
beginning mass	45
ending mass	230
scan rate / sampling	8 scans/sec

5.0 Calibration performed per 8.0 using 8260UL standards.

6.0 Sample preparation and analysis:

6.1 Allow samples to warm to ambient.

6.2 Insert samples into auto-sampler tray.

6.3 Program auto-sampler to utilize a 20mL sample purge volume.

6.4 Record pH of samples after analysis.

6.5 Target analyte results above the upper calibration limit of the instrument require a dilution. The appropriate sample volume is diluted in a volumetric flask to obtain results within the calibrated range of the instrument.

7.0 Analytical procedures, QC requirements, and data review and reporting per MSV01 SOP, using 20mL purge volume.

NOTE: Analysis of acetone, 2-butanone, 4-metyl-2-pentanone, 2-hexanone, 2-chloroethyl vinyl ether, and vinyl acetate performed using the standard 8260B method procedures.

Appendix IV
Method 6200B

1.0 Water samples are analyzed using a 25mL purge volume to achieve reporting limits in 2.0 below.

2.0 The following lists 6200B target analytes and performance data:

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	70-130	20	0.1	0.2	10	0.2-50
1,1,1-trichloroethane	71-55-6	70-130	20	0.1	0.2	10	0.2-50
1,1,2,2-tetrachloroethane	79-34-5	70-130	20	0.1	0.2	10	0.2-50
1,1,2-trichloro-1,2,2-trifluoroethane	76-13-1	60-140	20	0.1	0.2	10	0.2-50
1,1,2-trichloroethane	79-00-5	70-130	20	0.1	0.2	10	0.2-50
1,1-dichloroethane	75-34-3	70-130	20	0.1	0.2	10	0.2-50
1,1-dichloroethene	75-35-4	70-130	20	0.1	0.2	10	0.2-50
1,1-dichloropropene	563-58-6	70-130	20	0.1	0.2	10	0.2-50
1,2,3-trichlorobenzene	87-61-6	70-130	20	0.1	0.2	10	0.2-50
1,2,3-trichloropropane	96-18-4	70-130	20	1	2	10	0.2-50
1,2,4-trichlorobenzene	120-82-1	70-130	20	0.1	0.2	10	0.2-50
1,2,4-trimethylbenzene	95-63-6	70-130	20	0.1	0.2	10	0.2-50
1,2-dibromoethane	106-93-4	70-130	20	0.1	0.2	10	0.2-50
1,2-dichlorobenzene	95-50-1	70-130	20	0.1	0.2	10	0.2-50
1,2-dichloroethane	107-06-2	70-130	20	0.1	0.2	10	0.2-50
1,2-dichloropropane	78-87-5	70-130	20	0.1	0.2	10	0.2-50
1,3,5-trimethylbenzene	108-67-8	70-130	20	0.1	0.2	10	0.2-50
1,3-dichlorobenzene	541-73-1	70-130	20	0.1	0.2	10	0.2-50
1,3-dichloropropane	142-28-9	70-130	20	0.1	0.2	10	0.2-50
1,4-dichlorobenzene	106-46-7	70-130	20	0.1	0.2	10	0.2-50
2,2-dichloropropane	594-20-7	70-130	20	0.1	0.2	10	0.2-50
2-butanone	78-93-3	40-160	20	0.5	20	10	0.2-50
2-chlorotoluene	95-49-8	70-130	20	0.1	0.2	10	0.2-50
2-hexanone	591-78-6	40-160	20	1	2	10	0.2-50
4-chlorotoluene	106-43-4	70-130	20	0.1	0.2	10	0.2-50
4-methyl-2-pentanone	108-10-1	40-160	20	1	2	10	0.2-50
Acetone	67-64-1	40-160	20	0.5	10	10	0.2-50
benzene	71-43-2	70-130	20	0.1	0.2	10	0.2-50
bromobenzene	108-86-1	70-130	20	0.1	0.2	10	0.2-50
bromochloromethane	74-97-5	70-130	20	0.1	0.2	10	0.2-50
bromodichloromethane	75-27-4	70-130	20	0.1	0.2	10	0.2-50
bromoform	75-25-2	70-130	20	0.1	0.2	10	0.2-50
Bromomethane	74-83-9	60-140	20	0.1	1	10	0.2-50
carbon tetrachloride	56-23-5	70-130	20	0.1	0.2	10	0.2-50
chlorobenzene	108-90-7	70-130	20	0.1	0.2	10	0.2-50
Chloroethane	75-00-3	60-140	20	0.1	1	10	0.2-50
chloroform	67-66-3	70-130	20	0.1	0.2	10	0.2-50
chloromethane	74-87-3	60-140	20	0.1	0.2	10	0.2-50
cis-1,2-dichloroethene	156-59-2	70-130	20	0.1	0.2	10	0.2-50
cis-1,3-dichloropropene	10061-01-5	70-130	20	0.1	0.2	10	0.2-50
dibromochloromethane	124-48-1	70-130	20	0.1	0.2	10	0.2-50
dibromomethane	74-95-3	70-130	20	0.1	0.2	10	0.2-50
dichlorodifluoromethane	75-71-8	60-140	20	0.1	0.2	10	0.2-50
Diisopropyl ether	108-20-3	40-160	20	0.1	0.2	10	0.2-50

6200B Target Analytes and Performance Data (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)
Ethanol	64-17-5	40-160	20	25	50	500	50-2000
ethyl benzene	100-41-4	70-130	20	0.1	0.2	10	0.2-50
hexachlorobutadiene	87-68-3	70-130	20	0.1	0.2	10	0.2-50
isopropylbenzene	98-82-8	70-130	20	0.1	0.2	10	0.2-50
m+p-xylene **	108-38-3 + 106-42-3	70-130	20	0.1	0.2	20	0.4-100
methylene chloride	75-09-2	70-130	20	0.1	0.2	10	0.2-50
methyl-tert-butyl ether	1634-04-4	70-130	20	0.1	1	10	0.2-50
naphthalene	91-20-3	70-130	20	0.1	1	10	0.2-50
n-butylbenzene	104-51-8	70-130	20	0.1	0.2	10	0.2-50
o-xylene	95-47-6	70-130	20	0.1	0.2	10	0.2-50
p-isopropyl-toluene	99-87-6	70-130	20	0.1	0.2	10	0.2-50
propylbenzene	103-65-1	70-130	20	0.1	0.2	10	0.2-50
sec-butylbenzene	135-98-8	70-130	20	0.1	0.2	10	0.2-50
styrene	100-42-5	70-130	20	0.1	0.2	10	0.2-50
tert-butylbenzene	98-06-6	70-130	20	0.1	0.2	10	0.2-50
tetrachloroethene	127-18-4	70-130	20	0.1	0.2	10	0.2-50
Toluene	108-88-3	70-130	20	0.1	0.2	10	0.2-50
trans-1,2-dichloroethene	156-60-5	70-130	20	0.1	0.2	10	0.2-50
trans-1,3-dichloropropene	10061-02-6	70-130	20	0.1	0.2	10	0.2-50
trichloroethene	79-01-6	70-130	20	0.1	0.2	10	0.2-50
trichlorofluoromethane	75-69-4	60-140	20	0.1	0.2	10	0.2-50
Vinyl acetate	108-05-4	40-160	20	2.5	5	10	0.2-50
vinyl chloride	75-01-4	60-140	20	0.1	0.2	10	0.2-50
Xylenes	1330-20-7	70-130	20	0.1	0.2	10	0.2-50

3.0 6200B standards preparation:

3.1 Primary and intermediate calibration standards prepared per Section 7.0.

3.2 Working standards used for instrument calibration and calibration verification are prepared by diluting intermediate standards in DI water as follows:

Water Initial Calibration Standards, ug/L
(suggested preparation)

STOCK STANDARD CONCENTRATION	WORKING STANDARD, ug/L							
	0.2	0.4	0.5	1.0	5.0	10	20	50
Ethanol 200 ug/mL	N/A	N/A	N/A	10 x***	100 uL	250 uL	500 uL	1000 uL
5050VOA Mega mix (8260 Standard) 50 ug/mL	10 x*	1.6 uL	10 x**	10 x***	5 uL	10 uL	20 uL	50 uL
Custom Gases 50 ug/mL	10 x*	1.6 uL	10 x**	10 x***	5 uL	10 uL	20 uL	50 uL
Custom Mix 2 50 ug/mL	10 x*	1.6 uL	10 x**	10 x***	5 uL	10 uL	20 uL	50 uL
Custom Mix 3 50 ug/mL	10 x*	1.6 uL	10 x**	10 x***	5 uL	10 uL	20 uL	50 uL
Surrogate Standard 50 ug/mL	10 x*	1.6 uL	10 x**	10 x***	5 uL	10 uL	20 uL	50 uL
Final volume, DI water (mL)	50	200	50	50	50	50	50	50

10x* denotes 2.0 ug/L standard diluted 10x
10x** denotes 5.0 ug/L standard diluted 10x
10x*** denotes 10 ug/L standard diluted 10x

3.3 Working standards used for LCS/MS/ICV are prepared by diluting intermediate standards as follows:

Water Analyses

STOCK STANDARD CONCENTRATION (ug/mL)	VOLUME (uL)	FINAL VOLUME (mL)	FINAL CONCENTRATION (ug/L)
VOC mixture 8260 QC (20 ug/mL)	25	50	10
VOC additionals Adds QC (20 ug/mL)	25	50	10
8260 extra additionals (20 ug/mL)	25	50	10
Ethanol (2000 ug/mL)	250	50	500

4.0 Configure instrument per the following:

RECOMMENDED GC/MS PURGE AND TRAP PARAMETERS	
purge time	11 minutes
dry purge time	1 minute
desorb preheat	245° C
desorb	0.5 minute at 250° C
bake	14.5 minutes at 260° C
Velocity temps	valve 150° C, lines: 150° C
Archon temps	valve 95° C, lines 120° C
GAS CHROMATOGRAPH PARAMETERS	
carrier gas	helium, 99.999 %
injector temperature	220° C
oven temperature program	35° C (hold 4 minutes)
oven ramp	10° C/minute to 240° C (hold 2 minutes)
split ratio	40 : 1
MASS SPECTROMETER PARAMETERS	
beginning mass	45
ending mass	300
scan rate / sampling	8 scans/sec

5.0 Calibration standards analyzed per parameters per 4.0 above**.**

5.1 Initial Calibration acceptance criteria:

- Minimum 5 point calibration
- Average RRF used if %RSD < 20%, else curve-fitting (linear regression > 0.994, quadratic regression > 0.99).
- Residual for ICAL standards ± 20% when using linear or quadratic curve-fitting
- Initial calibration verification: ± 40% drift

5.2 Continuing calibration (CCV) acceptance criteria:

- CCV prepared at 10ug/L and analyzed per 4.0 above
- Performed daily, every 20 samples, and at the completion of analytical sequence
- Target analytes recoveries: 70%-130% (gases, ketones, ethanol, vinyl acetate and diisopropylether 60%-140%)

6.0 Quality control:

6.1 Laboratory reagent blank: Analyzed prior to samples. Target analytes must be < RL.

6.2 Laboratory-fortified blank (LFB): Laboratory reagent blank containing all target analyes at 10ug/L. Acceptance criteria 70%-130% (60%-140% for gases. The laboratory fortified blank and continuing calibration may be the same.

6.3 Laboratory-fortified blank duplicate (LFBD): Duplicate laboratory reagent blank when MS/MSD not provided by client. Acceptance criteria per 6.2 above.

7.0 Sample preparation and analysis:

7.1 Allow samples to warm to ambient.

7.2 Insert samples into auto-sampler tray.

7.3 Program auto-sampler to utilize a 25mL sample purge volume.

7.4 Record pH of samples after analysis.

7.5 Target analyte results above the upper calibration limit of the instrument require a dilution. The appropriate sample volume is diluted in a volumetric flask to obtain results within the calibrated range of the instrument.

8.0 Additional QC:

8.1 Internal standards responses for QC and samples: 70%-130% compared to the mean ICAL responses.

8.2 Surrogate recoveries for QC and samples: 70%-130%.

8.3 Laboratory-fortified sample/sample duplicate: Analyzed provided submits appropriate sample volume. Prepared and analyzed at 10ug/L.

8.4 All data undergoes thorough primary and secondary review per Section 14.0.

8.5 Refer to Section 13.11 for non-conforming data.

Appendix V
South Carolina Requirements

1.0 The following lists LCS control limits for analyses associated with South Carolina samples:

PARAMETER	WATER LCS	SOIL LCS
acetone*	60-140	60-140
benzene	70-130	70-130
bromobenzene	70-130	70-130
bromochloromethane	70-130	70-130
bromodichloromethane	70-130	70-130
bromoform*	60-140	60-140
bromomethane*	60-140	60-140
2-butanone*	60-140	60-140
n-butylbenzene	70-130	70-130
s-butylbenzene	70-130	70-130
t-butylbenzene	70-130	70-130
carbon disulfide	70-130	70-130
carbon tetrachloride	70-130	70-130
chlorobenzene	70-130	70-130
chlorodibromomethane	70-130	70-130
chloroethane*	60-140	60-140
2-chloroethyl vinyl ether*	60-140	60-140
chloroform	70-130	70-130
chloromethane*	60-140	60-140
2-chlorotoluene	70-130	70-130
4-chlorotoluene	70-130	70-130
1,2-dibromo-3-chloropropane*	60-140	60-140
1,2-dibromoethane	70-130	70-130
dibromomethane	70-130	70-130
1,2-dichlorobenzene	70-130	70-130
1,3-dichlorobenzene	70-130	70-130
1,4-dichlorobenzene	70-130	70-130
dichlorodifluoromethane*	60-140	60-140
1,1-dichloroethane	70-130	70-130
1,2-dichloroethane	70-130	70-130
1,1-dichloroethene	70-130	70-130
c-1.2-dichloroethene	70-130	70-130
t-1,2-dichloroethene	70-130	70-130

PARAMETER	WATER LCS	SOIL LCS
1,2-dichloropropane	70-130	70-130
1,3-dichloropropane	70-130	70-130
2,2-dichloropropane	70-130	70-130
1,1-dichloropropene	70-130	70-130
c-1,3-dichloropropene	70-130	70-130
t-1,3-dichloropropene	70-130	70-130
ethyl benzene	70-130	70-130
hexachlorobutadiene	70-130	70-130
2-hexanone*	60-140	60-140
isopropylbenzene	70-130	70-130
p-isoporpyltoluene	70-130	70-130
4-methyl-2-pentanone*	60-140	60-140
methylene chloride	70-130	70-130
naphthalene	70-130	70-130
n-propylbenzene	70-130	70-130
styrene	70-130	70-130
1,1,1,2- tetrachloroethene	70-130	70-130
1,1,2,2-tetrachloroethane	70-130	70-130
tetrachloroethene	70-130	70-130
toluene	70-130	70-130
1,2,3-trichlorobenzene	70-130	70-130
1,2,4-trichlorobenzene	70-130	70-130
1,1,1-trichloroethane	70-130	70-130
1,1,2-trichloroethane	70-130	70-130
trichloroethene	70-130	70-130
trichlorofluoromethane*	60-140	60-140
1,2,3-trichloropropane	70-130	70-130
1,2,4-trimethylbenzene	70-130	70-130
1,3,5-trimethylbenzene	70-130	70-130
vinyl acetate*	60-140	60-140
vinyl chloride*	60-140	60-140
o-xylene	70-130	70-130
m,p-xylene	70-130	70-130

* Denotes poor purging / poor performing compounds.

2.0 South Carolina requires all LCS compounds to meet acceptance criteria in Section 1.0 of Appendix V.

3.0 Analytes cannot be reported to South Carolina if the initial calibration fails to meet acceptance criteria.

4.0 All surrogate compounds must be within control limits for samples from South Carolina.

5.0 Based on project requirements, water samples collected for the analysis of vinyl chloride, styrene, and 2-chloroethylvinylether must be collected without acid preservation and analyzed within seven days of collection. The laboratory will send two sets of sample containers (one set (2 vials) preserved with acid, the other set (2 vials) un-preserved for the three compounds) or collect all samples without acid preservation.

Table 1
Method Analytes for MSV01

ANALYTE	CAS NUMBER
1,1,1,2-tetrachloroethane	630-20-6
1,1,1-trichloroethane	71-55-6
1,1,2,2-tetrachloroethane	79-34-5
1,1,2-trichloro-1,2,2-trifluoroethane	76-13-1
1,1,2-trichloroethane	79-00-5
1,1-dichloroethane	75-34-3
1,1-dichloroethene	75-35-4
1,1-dichloropropene	563-58-6
1,2,3-trichlorobenzene	87-61-6
1,2,3-trichloropropane	98-18-4
1,2,4-trimethylbenzene	95-63-6
1,2,4-trimethylbenzene	95-63-6
1,2-dibromo-3-chloropropane	96-12-8
1,2-dibromoethane	106-93-4
1,2-dichlorobenzene	95-50-1
1,2-dichloroethane	107-06-2
1,2-dichloropropane	78-87-5
1,3,5-trimethylbenzene	108-67-8
1,3-butadiene	106-99-0
1,3-dichlorobenzene	541-73-1
1,3-dichloropropane	142-28-9
1,4-dichlorobenzene	106-46-7
1,4-dioxane	123-91-1
1-bromopropane	106-94-5
1-butanol	71-36-3
1-chlorohexane	544-10-5
2,2-dichloropropane	594-20-7
2-butanone	78-93-3
2-chloroethylvinylether	110-75-8
2-chlorotoluene	95-49-8
2-hexanone	591-78-6
2-nitropropane	79-46-9
4-chlorotoluene	106-43-4
4-methyl-2-pentanone	108-10-1
acetone	67-64-1
acetonitrile	75-05-8
acrolein	107-02-8
acrylonitrile	107-13-1
allylchloride (3-chloroprene)	107-05-1
a-methyl styrene	98-83-9
benzene	71-43-2
bromobenzene	108-86-1
bromochloromethane	74-97-5
bromodichloromethane	75-27-4
bromoform	75-25-2
bromomethane	74-83-9
carbon disulfide	75-15-0
carbon tetrachloride	56-23-5
chlorobenzene	108-90-7
chloroethane	75-00-3
chloroform	67-66-3
chloromethane	74-87-3
-	-

ANALYTE	CAS NUMBER
chloroprene (2-chloro-1,3-butadiene)	126-99-8
cis-1,2-dichloroethene	156-59-2
cis-1,3-dichloropropene	10061-01-5
cyclohexane	110-82-7
cyclohexanone	108-94-1
dibromochloromethane	124-48-1
dibromomethane	74-95-3
dichlorodifluoromethane	75-71-8
diethyl ether	60-29-7
diisopropyl ether	108-20-3
dimethyl disulfide	624-92-0
dimethylsulfide	75-18-3
ethyl acetate	141-78-6
ethyl ether	60-29-7
ethyl methacrylate	97-63-2
ethyl t-butyl ether	637-92-3
ethylbenzene	100-41-4
hexachlorobutadiene	87-68-3
iodomethane	74-88-4
isobutanol	78-83-1
isoprene	78-79-5
isopropyl benzene	98-82-8
m+p-xylene	108-38-3 & 106-42-3
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-heptane	142-82-5
n-hexane	110-54-3
n-propyl benzene	103-65-1
o-xylene	95-47-6
paraldehyde	123-63-7
p-isopropyl-toluene	99-87-6
propionitrile (ethyl cyanide)	107-12-0
sec-butyl-benzene	135-98-8
styrene	100-42-5
t-amylmethyl ether	994-05-8
t-butanol	75-65-0
tert-butyl-benzene	98-06-6
tetrachloroethene	127-18-4
tetrahydrofuran	109-99-9
toluene	108-88-3
trans-1,2-dichloroethene	156-60-5
trans-1,3-dichloropropene	10061-02-6
trans-1,4-dichloro-2-butene	110-57-6
trichloroethene	79-01-6
trichlorofluoromethane	75-69-4
vinyl acetate	108-05-4
vinyl chloride	75-01-4

Table 2
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR VOLATILE ORGANIC ANALYSES OF GROUNDWATER

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/L)	REPORTING LIMITS (ug/L)	LCS, MS/MSD, TRUE VALUE (ug/L)	SUGGESTED CALIBRATION RANGE (ug/L)
1,1,1,2-tetrachloroethane	630-20-6	80-130	20	0.25	5	20	200-300
1,1,1-trichloroethane	71-55-6	80-134	20	0.25	5	20	200-300
1,1,2,2-tetrachloroethane	79-34-5	79-125	20	0.2	5	20	200-300
1,1,2-trichloro-1,2,2-trif	76-13-1	40-160	20	1	10	20	200-300
1,1,2-trichloroethane	79-00-5	80-125	20	0.25	5	20	200-300
1,1-dichloroethane	75-34-3	80-125	20	0.125	5	20	200-300
1,1-dichloroethene	75-35-4	80-132	20	0.5	5	20	200-300
1,1-dichloropropene	563-58-6	75-130	20	0.25	5	20	200-300
1,2,3-trichlorobenzene	87-61-6	55-140	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	65-135	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	50-130	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)	156-59-2 + 156-60-5	80-124	20	0.25	5	40	200-300
1,2-dichloropropane	78-87-5	80-120	20	0.2	5	20	200-300
1,3,5-trimethylbenzene	108-67-8	80-127	20	0.25	5	20	200-300
1,3-butadiene	106-99-0	10-200	20	1	10	20	200-300
1,3-dichlorobenzene	541-73-1	80-120	20	0.25	5	20	200-300
1,3-dichloropropane	142-28-9	80-120	20	0.2	5	20	200-300
1,4-dichlorobenzene	106-46-7	80-120	20	0.125	5	20	200-300
1,4-dioxane	123-91-1	20-160	20	50	100	200	200-300
1-bromopropane	106-94-5	50-150	20	0.5	1	20	200-300
1-butanol	71-36-3	50-150	20	50	100	200	50-800
1-chlorohexane	544-10-5	80-127	20	0.125	1	20	200-300
2,2-dichloropropane	594-20-7	80-133	20	0.25	5	20	200-300
2-butanone	78-93-3	10-170	20	2.5	10	20	200-300
2-chloroethyl vinyl ether	110-75-8	45-160	20	2.0	10	20	200-300
2-chlorotoluene	95-49-8	80-127	20	0.125	5	20	200-300
2-hexanone	591-78-6	55-130	20	2.5	10	20	200-300
2-nitropropane	79-46-9	10-150	20	5	50	100	200-300
3-chloro-1-propene	107-05-1	70-130	20	2.5	10	20	200-300
4-chlorotoluene	106-43-4	80-126	20	0.25	5	20	200-300
4-methyl-2-pentanone	108-10-1	64-140	20	2.5	10	20	200-300
acetone	67-64-1	40-180	20	2.5	10	20	200-300
acetonitrile	75-05-8	70-130	20	5	100	100	200-300
Acrolein	107-02-8	10-200	20	20	100	20	200-300
acrylonitrile	107-13-1	50-150	20	2.5	100	20	200-300
alpha-methyl-styrene	98-83-9	50-150	20	0.5	10	20	200-300
benzene	71-43-2	80-121	20	0.125	5	20	200-300
bromobenzene	108-86-1	80-120	20	0.125	5	20	200-300
bromochloromethane	74-97-5	65-130	20	0.2	5	20	200-300
bromodichloromethane	75-27-4	80-131	20	0.25	5	20	200-300
bromoform	75-25-2	70-130	20	0.5	5	20	200-300

Table 2 (continued)

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/L)	REPORTING LIMITS (ug/L)	LCS, MS/MSD, TRUE VALUE (ug/L)	SUGGESTED CALIBRATION RANGE (ug/L)
bromomethane	74-83-9	30-145	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	40-125	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	70-125	20	0.25	5	20	200-300
cis-1,3-dichloropropene	10061-01-5	70-130	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	60-135	20	0.25	5	20	200-300
dibromomethane	74-95-3	75-125	20	0.25	5	20	200-300
dichlorodifluoromethane	75-71-8	40-160	20	0.25	5	20	200-300
diethyl ether	60-29-7	70-130	20	5	10	100	200-300
diisopropyl ether	108-20-3	70-130	20	5	10	100	200-300
dimethyl disulfide	624-92-0	70-130	20	1.0	10	20	200-300
dimethyl sulfide	75-18-3	70-130	20	0.5	10	20	200-300
ethyl acetate	141-78-6	70-130	20	5	50	100	200-300
ethyl benzene	100-41-4	80-122	20	0.25	5	20	200-300
ethyl methacrylate	97-63-2	70-130	20	1.0	10	20	200-300
ethyl-tert-butyl ether	637-92-3	70-130	20	5	10	100	200-300
hexachlorobutadiene	87-68-3	72-132	20	0.25	5	20	200-300
iodomethane	74-88-4	10-160	20	0.5	10	20	200-300
Isobutanol	78-83-1	10-180	20	50	100	200	50-800
isoprene	78-79-5	70-130	20	0.53	10	20	200-300
isopropylbenzene	98-82-8	80-122	20	0.25	5	20	200-300
m+p-xylene **	179601-23-1	80-122	20	0.5	5	40	5-800
methacrylonitrile	126-98-7	70-130	20	2.5	5	100	200-300
Methyl acetate	79-20-9	50-190	20	1	10	20	200-300
Methyl cyclohexane	108-87-2	80-130	20	1	10	20	200-300
methyl methacrylate	80-62-6	70-130	20	2.5	5	100	200-300
methylene chloride	75-09-2	80-123	20	0.25	5	20	200-300
methyl-tert-butyl ether	1634-04-4	65-125	20	0.5	5	20	200-300
naphthalene	91-20-3	59-149	20	0.2	5	20	200-300
n-butylbenzene	104-51-8	80-131	20	0.25	5	20	200-300
n-heptane	142-82-5	70-130	20	2.5	5	20	200-300
n-hexane	110-54-3	74-137	20	0.56	5	20	200-300
o-xylene	95-47-6	80-122	20	0.25	5	20	200-300
p-isopropyl-toluene	99-87-6	80-122	20	0.25	5	20	200-300
propionitrile	107-12-0	50-150	20	2.5	5	100	200-300
propylbenzene	103-65-1	80-129	20	0.125	5	20	200-300
sec-butylbenzene	135-98-8	80-127	20	0.25	5	20	200-300
styrene	100-42-5	80-123	20	0.125	5	20	200-300
tert-amyl-methyl ether	994-05-8	70-130	20	5	10	100	200-300
tert-butyl alcohol	75-65-0	10-180	20	50	100	200	50-800

Table 2 (continued)

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/L)	REPORTING LIMITS (ug/L)	LCS, MS/MSD, TRUE VALUE (ug/L)	SUGGESTED CALIBRATION RANGE (ug/L)
tert-butylbenzene	98-06-6	80.126	20	0.25	5	20	200-300
tetrachloroethene	127-18-4	80-124	20	0.25	5	20	200-300
tetrahydrofuran	109-99-9	60-140	20	5	50	100	200-300
Toluene	108-88-3	80-124	20	0.25	5	20	200-300
trans-1,2-dichloroethene	156-60-5	80-127	20	0.25	5	20	200-300
trans-1,3-dichloropropene	10061-02-6	80-130	20	0.5	5	20	200-300
trans-1,4-dichloro-2-butene	110-57-6	50-150	20	2.0	10	20	200-300
trichloroethene	79-01-6	80-122	20	0.25	5	20	200-300
trichlorofluoromethane	75-69-4	62-151	20	0.25	5	20	200-300
vinyl acetate	108-05-4	10-190	20	2.5	10	20	200-300
vinyl chloride	75-01-4	50-170	20	0.25	10	20	200-300
xylenes (total)	108-38-3 + 106-42-3 + 95-47-6	80-121	20	0.5	15	60	5-1200

** Unresolvable compound

Table 3
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR VOLATILE ORGANIC ANALYSES OF SOLID WASTE

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/Kg)	REPORTING LIMITS (ug/Kg)	LCS, MS/MSD, TRUE VALUE (ug/Kg)	SUGGESTED CALIBRATION RANGE (ug/Kg)
1,1,1,2-tetrachloroethane	630-20-6	71-137	30	0.5	5	20	5-200
1,1,1-trichloroethane	71-55-6	70-135	30	0.5	5	20	5-200
1,1,2,2-tetrachloroethane	79-34-5	55-130	30	0.5	5	20	5-200
1,1,2-trichloro-1,2,2-trif	76-13-1	70-130	30	1.0	10	20	5-200
1,1,2-trichloroethane	79-00-5	60-125	30	0.5	5	20	5-200
1,1-dichloroethane	75-34-3	75-125	30	1.0	5	20	5-200
1,1-dichloroethene	75-35-4	65-135	30	0.5	5	20	5-200
1,1-dichloropropene	563-58-6	57-138	30	0.5	5	20	5-200
1,2,3-trichlorobenzene	87-61-6	60-135	30	0.5	5	20	5-200
1,2,3-trichloropropane	96-18-4	65-130	30	1.0	5	20	5-200
1,2,4-trichlorobenzene	120-82-1	65-130	30	0.5	5	20	5-200
1,2,4-trimethylbenzene	95-63-6	75-132	30	0.5	5	20	5-200
1,2-dibromo-3-chloropropane	96-12-8	40-135	30	2.0	5	20	5-200
1,2-dibromoethane	106-93-4	69-128	30	0.5	5	20	5-200
1,2-dichlorobenzene	95-50-1	70-130	30	0.5	5	20	5-200
1,2-dichloroethane	107-06-2	63-133	30	0.5	5	20	5-200
1,2-dichloroethene (total)	156-59-2 + 156-60-5	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	100	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	100	20	5-200
acetonitrile	75-05-8	50-150	30	50	100	100	5-200
acrolein	107-02-8	50-150	30	20	100	20	5-200
acrylonitrile	107-13-1	60-140	30	2.5	100	20	5-200
alpha-methyl-styrene	98-83-9	70-130	30	0.5	10	20	5-200
benzene	71-43-2	70-130	30	0.5	5	20	5-200
bromobenzene	108-86-1	72-131	30	0.5	5	20	5-200
bromochloromethane	74-97-5	70-130	30	0.5	5	20	5-200
bromodichloromethane	75-27-4	72-137	30	0.5	5	20	5-200
bromoform	75-25-2	49-136	30	0.5	5	20	5-200
bromomethane	74-83-9	37-143	30	1.0	10	20	5-200

Table 3 (continued)

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/Kg)	REPORTING LIMITS (ug/Kg)	LCS, MS/MSD, TRUE VALUE (ug/Kg)	SUGGESTED CALIBRATION RANGE (ug/Kg)
carbon disulfide	75-15-0	39-139	30	0.5	5	20	5-200
carbon tetrachloride	56-23-5	59-136	30	0.5	5	20	5-200
chlorobenzene	108-90-7	70-130	30	0.5	5	20	5-200
chloroethane	75-00-3	52-135	30	1.0	10	20	5-200
chloroform	67-66-3	74-129	30	0.5	5	20	5-200
chloromethane	74-87-3	30-131	30	2.0	10	20	5-200
chloroprene	126-99-8	50-150	30	2.5	5	100	5-200
cis-1,2-dichloroethene	156-59-2	70-130	30	0.5	5	20	5-200
cis-1,3-dichloropropene	10061-01-5	70-142	30	0.5	5	20	5-200
Cyclohexane	110-82-7	70-130	30	1.0	10	20	5-200
cyclohexanone	108-94-1	60-140	30	5	50	100	5-200
dibromochloromethane	124-48-1	59-136	30	0.5	5	20	5-200
dibromomethane	74-95-3	69-130	30	0.5	5	20	5-200
dichlorodifluoromethane	75-71-8	25-130	30	1.0	5	20	5-200
diethyl ether	60-29-7	60-140	30	5	10	100	5-200
diisopropyl ether	108-20-3	60-140	30	5	10	100	5-200
dimethyl disulfide	624-92-0	60-140	30	0.5	10	20	5-200
dimethyl sulfide	75-18-3	60-140	30	0.5	10	20	5-200
ethyl acetate	141-78-6	60-140	30	5	50	100	5-200
ethyl benzene	100-41-4	70-130	30	0.5	5	20	5-200
ethyl methacrylate	97-63-2	60-140	30	1.0	10	20	5-200
ethyl-tert-butyl ether	637-92-3	60-140	30	5	10	100	5-200
hexachlorobutadiene	87-68-3	65-135	30	0.5	5	20	5-200
iodomethane	74-88-4	20-288	30	1.0	10	20	5-200
isobutanol	78-83-1	50-150	30	50	100	200	10-400
Isoprene	78-79-5	40-140	30	2.0	10	20	5-200
isopropylbenzene	98-82-8	68-129	30	0.5	5	20	5-200
m+p-xylene **	179601-23-1	70-130	30	0.5	5	40	5-200
methacrylonitrile	126-98-7	60-140	30	2.5	5	100	5-200
methyl acetate	79-20-9	70-130	30	1	10	20	5-200
methyl cyclohexane	108-87-2	70-130	30	1	10	20	5-200
methyl methacrylate	80-62-6	70-130	30	2.5	5	100	5-200
methylene chloride	75-09-2	74-128	30	1.0	5	20	5-200
methyl-tert-butyl ether	1634-04-4	54-151	30	0.5	5	20	5-200
naphthalene	91-20-3	50-146	30	0.5	5	20	5-200
n-butylbenzene	104-51-8	70-136	30	0.5	5	20	5-200
n-heptane	142-82-5	60-140	30	2.5	5	20	5-200
n-hexane	110-54-3	58-142	30	0.5	10	20	5-200
o-xylene	95-47-6	70-130	30	0.5	5	20	5-200
p-isopropyl-toluene	99-87-6	72-128	30	0.5	5	20	5-200
propionitrile	107-12-0	60-140	30	2.5	5	100	5-200
propylbenzene	103-65-1	72-136	30	0.5	5	20	5-200
sec-butylbenzene	135-98-8	71-132	30	0.5	5	20	5-200
styrene	100-42-5	74-130	30	0.5	5	20	5-200
tert-amyl-methyl ether	994-05-8	60-140	30	5	10	100	5-200
tert-butyl alcohol	75-65-0	50-150	30	50	100	200	50-400
tert-butylbenzene	98-06-6	72-130	30	0.5	5	20	5-200

Table 3 (continued)

PARAMETER	CAS #	ACCURACY (% REC)	PRECISION (RPD)	MDL (ug/Kg)	REPORTING LIMITS (ug/Kg)	LCS, MS/MSD, TRUE VALUE (ug/Kg)	SUGGESTED CALIBRATION RANGE (ug/Kg)
tetrachloroethene	127-18-4	72-130	30	0.5	5	20	5-200
tetrahydrofuran	109-99-9	50-150	30	25	50	100	5-200
toluene	108-88-3	77-126	30	0.5	5	20	5-200
trans-1,2-dichloroethene	156-60-5	72-127	30	0.5	5	20	5-200
trans-1,3-dichloropropene	10061-02-6	65-139	30	0.5	5	20	5-200
trans-1,4-dichloro-2-butene	110-57-6	50-150	30	1.0	10	20	5-200
trichloroethene	79-01-6	72-126	30	0.5	5	20	5-200
trichlorofluoromethane	75-69-4	48-154	30	1.0	5	20	5-200
vinyl acetate	108-05-4	10-150	30	2.5	10	20	5-200
vinyl chloride	75-01-4	45-140	30	1.0	10	20	5-200
xylenes (total)	108-38-3 + 106-42-3 + 95-47-6	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
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

* Denotes suggested parameters; systems may be adjusted to improve efficiency.

Table 5
BFB Key Ion Abundance Criteria

MASS	ION ABUNDANCE CRITERIA
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95%, but less than 101% of mass 174
177	5 to 9% of mass 176

Table 6
Surrogate Standards Recovery Limits

SURROGATE COMPOUND	8260 WATER *	AFCEE 1998 QAPP WATER	8260 SOIL *	AFCEE 1998 QAPP (3.0) SOIL	2001/2005 AFCEE WATER	2001/2005 AFCEE SOIL	8260 OIL
dibromofluoromethane	86-118	75-125	80-120	65-135	85-115	65-135	52-122
Toluene-d_8	88-110	75-125	81-117	65-135	81-120	84-116	35-127
4-bromofluorobenzene	86-115	75-125	74-121	65-135	76-119	84-118	26-158
1,2-dichloroethane-d_4	80-120	62-139	80-120	52-149	72-119	52-149	43-128

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

Table 7
Quality Control Criteria
Volatile GC/MS Analysis
Method 8260B

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Mass spectral ion intensities (BFB criteria)	Every 12 hours prior to ICAL, ICV or CCV	See Table 5	Retune instrument and repeat BFB check
Initial Calibration (ICAL)	When Continuing Calibration is out of control or when system conditions have been altered.	≤ 30% RSD for CCC compounds, < 15% RSD for all target compounds, if >15% RSD, then linear regression, provided r ≥ 0.995,then quadratic regression, provided r^2 ≥ 0.990 SPCC minimum $\overline{RRF}$	Evaluate cause; repeat calibration; or qualify data and discuss in narrative (1) See section 13.7 for additional corrective action.
Second source calibration verification (ICV)	After each initial calibration	≤ 30% 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, CCC percent drift ≤ 20%; other target analytes ≤ 40% (1)	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	< ½ RL	Notify supervisor and initiate NCR; investigate; re-analyze samples
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	One per matrix/batch; maximum of 20 samples per batch	Target compounds within the designated ranges; use project QAPP or standard control criteria (1,2)	Notify supervisor and initiate NCR; investigate; re-analyze samples
Matrix Spikes/ Matrix Spike Duplicate (MS/MSD) Sample/Sample Duplicate	One per matrix/batch; maximum of 20 samples per batch	Target compounds within the designated ranges; use project QAPP or standard control criteria (1,2)	Qualify data and/or address in the report narrative
Surrogate spike	Every sample, standard, and quality control sample	Recoveries within designated ranges: use project QAPP or standard control criteria; (one surrogate outlier permitted provided % R > 10%) (1)	Notify supervisor and initiate NCR; investigate; re-analyze samples

(1) Evaluation criteria are often project specific. Check the project QAPP.
(2) Standard criteria are set at three standard deviations from the mean; 10% marginal failure allowed, otherwise re-extract and re-analyze batch; consult supervisor and project QAPP for any exceptions.

Figure 1

Checklist ID: 6687

Microbac Laboratories Inc.

Data Checklist

Date: _____
Analyst: _____
Analyst: NA
Method: _____
Instrument: _____
Curve Workgroup: NA
Runlog ID: _____
Analytical Workgroups: _____

Runlog ID number:	
System Performance Check	
BFB	
Initial Calibration	
Average RF	
Linear Reg or Higher Order Curve	
Second Source standard % Difference	
Continuing Calibration /Check Standards	
Project/Client Specific Requirements	
Special Standards	
Blanks	
TCL's	
Surrogates	
LCS (Laboratory Control Sample)	
Recoveries	
Surrogates	
MS/MSD/Duplicates	
Samples	
TCL Hits	
Spectra of TCL Hits	
Surrogates	
Internal Standards Criteria	
Library Searches	
Calculations & Correct Factors	
Dilutions Run	
Reruns	
Manual Integrations	
Case Narrative	
Results Reporting/Data Qualifiers	
KOBRA Workgroup Data	
Check for Completeness	
Primary Reviewer	
Secondary Reviewer	
Check for compliance with method and project specific requirements	X
Check the completeness of reported information	X
Check the information for the report narrative	X
Check the reasonableness of the results	X

Primary Reviewer: Secondary Reviewer:

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Microbac

Figure 2
VOA Preparation/Preservation and Extraction Log

Microbac Laboratories Inc.
VOA Preparation/Preservation/Extraction Log

Workgroup (AAB#):WG270339
Method:8260
Reagent ID:RGT10001

Analyst:DGB
Run Date:06/05/2008 09:50

SAMPLE #	Fraction	Collected	Preserved	PCT-S	Tare Wt	Total Wt	Sample Wt	Water	MeOH	Vt	Comments
L08020355-08	A	04/20/08 11:12	06/05/08 09:50	79.86			30.12	5		5	5
L08020355-09	A	04/20/08 11:25	06/05/08 09:50	77.08			30.12	5		5	5
L08020355-09	B	04/20/08 11:25	06/05/08 09:51	77.08	5.12	30.1	24.98	5		5	5
L08020355-09	C	04/20/08 11:25	06/05/08 09:52	77.08			25.08		10	15.748816	5

Comments: 1 = improperly sealed cap
2 = preserved out of hold
3 = effervesced
4 = preserved with NaHSO4
5 = preserved by freezing
6 = preserved in field

Analyst: Douglas Butcher

VOA_EXT - Modified 05/22/2008
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MICROBAC

STANDARD OPERATING PROCEDURE
ORGANIC CARBON, TOTAL (OXIDATION)
EPA 415.1/SW846 9060A/SM5310C-2000
(2011 EDITORIAL REVISION)

Issue/Implementation Date: 15 January 2014

Last Review Date: 20 March 2014

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

Approved by:

Deanna I. Hesson, Conventionals Supervisor — Date: 3/17/14

Wade T. DeLong, Quality Assurance Officer — Date: 3-17-14

Leslie S. Bucina, Laboratory Manager — Date: 3/17/14

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This method is applicable for the determination of Total Organic Carbon (TOC) in drinking, surface and saline waters, domestic and industrial wastes. It is also applicable for leachable organic carbon on a 1:10 soil to acid mixture. **It is not applicable for total organic carbon on a soil**. TOC is defined as all of the organic carbon present in the sample that can be determined by this method. All analyses are performed within the Shimadzu TOC-VWP analyzer.

1.2 TOC is determined by the difference of Total Carbon (TC) and Inorganic Carbon (TIC). For the TC analysis, gas flows at a controlled rate through the TC reactor, which is comprised of a UV lamp and heater. When sample is injected along with the oxidizing reagent (containing sodium persulfate and phosphoric acid) into the TC reactor which has been heated to 80° C, the TC in the sample is oxidized and decomposed to form carbon dioxide. This carbon dioxide is swept via the carrier gas from the reaction tube to a dehumidifier for cooling and dehydration. These products then pass through a halogen scrubber to reach the cell of a Non-Dispersive Infrared Detector (NDIR), where the carbon dioxide is detected. The analog detection signal of the NDIR forms a peak, and the area of this peak is measured by a data processor. The peak area is proportional to the TC concentration of the sample. Inorganic carbon (IC) refers to carbon contained in the carbon dioxide dissolved in water and that found in carbonates. By acidifying the sample with a small amount of phosphoric acid to obtain a pH less than 3, all the carbonates produce carbon dioxide (CO_2). The carbon dioxide and dissolved carbon dioxide in the sample are volatilized by bubbling (sparging) gas through the sample. Sparge gas consisting of tiny bubbles flows at a controlled rate through the IC reactor. When sample is injected along with phosphoric acid into the IC reactor, only the IC component of the sample is converted to carbon dioxide, which is subsequently detected by the NDIR. The IC concentration in the sample is then measured in the same way as in the TC measurement process.

1.3 This method references EPA Method 415.1, and SW846 Method 9060A, and Standard Method 5310C-2000 (2011 Editorial Revision).

1.4 To comply with SW846 Method 9060A, each sample must be injected in quadruplicate.

1.4.1 The LIMS product TOC-14 is used when one sample is injected 4 times and one average result is reported (1 sample * 4 injections).

1.4.2 The LIMS product TOC-44 is used when the sample was sampled in the field in quadruplicate and each one is injected 4 times. This gives 16 results. The average of each bottle (four injections) is reported (4 samples * 4 injections).

1.4.3 The LIMS product TOC-1 is used to show that the sample gets 1 injection. The sample is reported as Method 9060 modified since 4 injections are not analyzed.

1.5 Method EPA 415.1 requires only 1 injection. SM5310C requires duplicate injections (average reported).

1.5.1 If samples are logged as a matrix 2, SM5310C must be followed. All other liquid matrices may follow Method 415.1.

1.5.2 The LIMS product TOC is used for both methods. One result is reported.

1.5.3 The LIMS product TOC-4 is used when one sample is sampled in the field in quadruplicate. Four results are reported.

1.6 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

CCB	Continuing calibration blank
CCV	Continuing calibration verification
COA	Certificate of analysis
DI water	Deionized water
DUP	Duplicate
IC	Inorganic carbon
ICB	Initial calibration blank
ICV	Initial calibration verification
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
LIMS	Laboratory Information Management System
LOD	Limits of Detection
LOQ	Limits of Quantitation
LQAP	Laboratory Quality Assurance Program
MB	Method blank
MDL	Method detection limit
MS	Matrix spike
MSD	Matrix spike duplicate
NCR	Nonconformance report
NDIR	Non-Dispersive Infrared Detector

QA	Quality assurance
QC	Quality control
RGT	Reagent
RL	Reporting limit
RPD	Relative Percent Difference
SDS	Safety Data Sheet
SOP	Standard Operating Procedure
STD	Standard
TC	Total Carbon
TIC	Total Inorganic Carbon
TOC	Total Organic Carbon

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

2.0 SAFETY PRECAUTIONS

2.1 Standard laboratory safety procedures must be followed when working with unknown samples. Lab coats and safety glasses with sideshields are required.

2.2 When doing maintenance on the TOC instrument, the electricity must be turned off to prevent electrocution.

2.3 The following chemicals have the potential to be toxic or hazardous. Consult the SDS for further information.

2.3.1 Sodium persulfate: This is a strong oxidizing agent and must not be stored near strong reducing agents.

2.3.2 Phosphoric acid: This is a strong and corrosive reagent.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Samples should be collected in a glass container and preserved with sulfuric acid (pH < 2). **NOTE**: HCL should not be used as preservative, because chloride is an interference and the result could be biased.

3.2 Samples must be kept refrigerated at ≤ 6° C until time of analysis, which is not to exceed 28 days from the time of collection. After analysis, samples are stored in a designated area in the archive room. See Section 16.0 for sample disposal.

3.3 Samples should be kept from sunlight and atmospheric oxygen.

3.4 A minimum of 40 mL is required for one analysis. More volume is required for reanalysis and QC requirements.

3.5 If less than the recommended volume is available the analysis will be performed on a dilution, and the RL will be elevated proportionately.

4.0 METHOD PERFORMANCE

4.1 This method uses an RL or LOQ of 1 mg/L. RLs are nominal laboratory values, but project RLs may vary. The LOQ are nominal limits and were established per Microbac SOP 45. Upon request a RL of 0.5 mg/L can be reported.

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. The laboratory verified MDL or LOD is 0.5 mg/L. The MDL/LOD is verified annually (using 7 replicates), or when necessary and verified quarterly. Verification consists of analyzing a fortified blank (MDL check standard) spike at a concentration one to three times the concentration of the MDL/LOD. The MDL/LOD check standard is used to verify that the laboratory MDL is routinely achievable over the course of time. Additional details on MDL studies may be found in Microbac SOP 45.

4.3 The linear range of the method depends on the range selected. The overall range is 1 mg/L to 50 mg/L total carbon. The upper limit may be extended by diluting the sample.

4.4 The current limits are 85-115% for the LCS and MS/MSD and the RPD is 15%. Precision and accuracy data were derived from an initial demonstration of capability using spiked control samples. The laboratory uses results from LCS to assess the precision and accuracy and to annually evaluate the associated control limits.

4.5 Each analyst is required to perform an initial demonstration of precision and accuracy. Annually, each analyst is recertified by performing four LCS. See Microbac SOP 45, which describes the procedure in detail.

4.6 AFCEE and other specific QA objectives may be found in the appropriate statement-of-work or QUAPP.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 The TOC value is determined by the difference in TC and IC analysis, which includes errors associated with both TC and IC analyses.

5.2 Large particles that cannot enter the sipper tube cannot be analyzed. This can result in low recovery.

5.3 Filtration can result in low recovery (removal of carbon-containing particles) or high recovery (carbon from filter paper).

5.4 Very high chloride content results in low recovery. Adding mercuric nitrate to the sample reduces this interference. Dilution also reduces the interference. The instrument can tolerate up to approximately 3000 mg/L CL.

6.0 EQUIPMENT AND SUPPLIES

6.1 TOC Analyzer: Shimadzu TOC-VWP with autosampler, computer, and printer

6.1.1 Software version 1.06.00.

6.1.2 Software versions and serial numbers of the instrument are recorded in the maintenance logbook.

6.2 Several 40 mL VOA vials

6.3 Mechanical shaker

6.4 All glassware is washed with soap and water as described in Microbac SOP K0001.

7.0 STANDARDS AND REAGENTS

NOTE: See Section 16.0 for reagent disposal.

NOTE: All standards and reagents are prepared using class A volumetric glassware.

NOTE: All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or

RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.1 TOC Stock Standard Solution (1000 mg/L): purchased. Discard by manufacturer's printed expiration date.

7.2 TOC LCS Stock Solution (1000 mg/L): Dissolve 2.1254g KHP DI water. Mix well. Preserve with 5 mL of 1:1 H_2SO_4. Dilute to 1 L. Discard after 6 months. This is a second source standard.

7.3 TIC Calibration Stock Solution (1000 mg/L): Dissolve 4.41 g sodium carbonate and 3.5 g sodium bicarbonate in DI water and dilute to 1 L.

7.4 TIC ICV Stock (1000 mg/L): Make exactly like calibration stock (7.3) except use different lots.

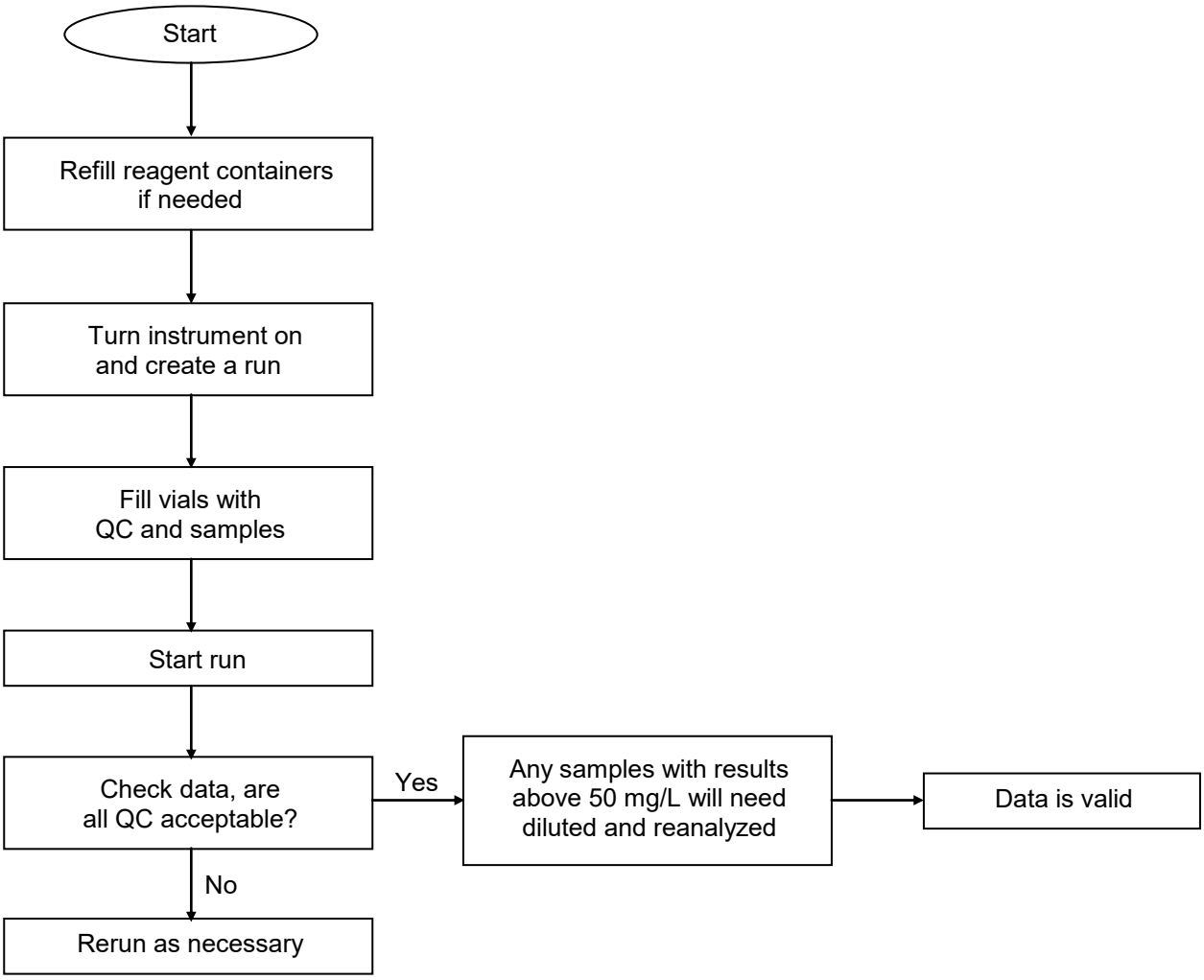
7.5 Sodium Persulfate Solution: Dissolve 120 g ultra pure sodium persulfate and 30 mL phosphoric acid in DI water and dilute to 1 L. This solution should sit for a day to allow oxidation of trace impurities. This reagent stable for at least a month when stored in a cool dark location.

7.6 Phosphoric Acid: Dilute 100 mL of concentrated phosphoric acid with DI water to a final volume of 500 mL with DI water. Discard after 6 months.

7.7 Sulfuric Acid (20%): Add 200 mL concentrated sulfuric acid to 800 mL DI water. Mix well.

7.8 Ultra pure Nitrogen gas.

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



9.0 SAMPLE PREPARATION

9.1 If the sample is a solid and leachable organic carbon is needed, a leachate of the solid will need prepared.

9.2 Add 10 mL of 20% sulfuric acid (7.5) to 10.0 g of soil. Mix well.

9.3 Add 90 mL of DI water and shake on a mechanical shaker for 30 minutes.

9.4 Let the sample settle.

9.5 Decant the solution and analyze the solution for TOC. This is considered Leachable Organic Carbon.

9.6 If dissolved organic carbon is needed, filter the sample through a 0.45 um membrane filter (previously soaked in 1:1 nitric acid) and analyze as below. A filtered DI water blank should also be analyzed.

9.7 If NPOC (non-purgeable TOC) is requested, purge the sample with nitrogen for 15 minutes. Analyze the sample the same as all others.

10.0 CALIBRATION PROCEDURES

10.1 The instrument is calibrated as needed (when CCV's fail or project defined). Two calibration curves are needed: TC and TIC.

10.1.1 The following standards are analyzed to determine the calibration curves. The TC and TIC curves are prepared identically from respective stocks (7.1 and 7.3).

STANDARD	USED (mL)	STOCK	TOTAL VOLUME	CONCENTRATION (mg/L)
1	10	1000	200	50
2	5	1000	200	25
3	2	1000	200	10
4	1	1000	200	5
5	10	10	100	1
6	-	-	-	0

10.2 R must be > 0.995 for each curve.

10.3 TOC ICV: (25 mg/L) Prepare a 25 mg/L standard using the LCS stock (7.2), and a dilute 5 mL of stock (7.2) to 200 mL DI water to make a 25 mg/L standard. The

result must be within ± 10% of true value, or the instrument will need recalibrated.

10.4 TIC ICV: (25 mg/L) Prepare a 25 mg/L standard using the LCS stock (7.4), and a dilute 5 mL of stock (7.4) to 200 mL DI water to make a 25 mg/L standard. The result must be within ± 10% of true value, or the instrument will need recalibrated.

10.5 To analyze a calibration curve use the following procedure:

- Choose NEW FILE
- Calibration curve
- Choose system (TOCVW ASI)
- Edit points manually
- Choose analysis (TC or IC): a curve will be ran for both.
- Choose linear regression
- UNCHOOSE zero shift
- Type the name of the file (ex: TCCURVE-8-28-2003 or TICCURVE-8-28-2003)
- Choose units, TWO injections, ONE wash then next page
- Enter each calibration point by pressing ADD and typing concentration then OK. Enter from blank to highest concentration.
- Create a run as in Section 11.0.
- Analyze the curves
- Analyze an ICV for both curves
- Before analyzing samples, a method must be created using the new curves.
  1. New file
  2. Method
  3. Pick system
  4. Next
  5. Choose analysis: TOC
  6. Choose a name for method (ex: TOC-9-30-2003)
  7. Next
  8. Pick calibration curve for TC analysis
  9. Next
  10. Enter number of injections: (1 for 415.1, 4 for 9060A, 2 for SM5310C)
  11. Enter washes: 1, UV oxid vol: 1.5
  12. Next
  13. pg. 5 Next
  14. Repeat steps 8-13 for TIC analysis (wash: 1, acid add :10)
  15. pg. 5 Next
  16. Next

10.6 A CCV and CCB are analyzed every 10 samples. See Section 13.0 for criteria.

10.7 Calibration training materials are available on the intranet home page in the "General" links section, "Calibration Training". Review of "Calibration Models" and "The Effect that Saturation of the Detector has Upon Calibration" are recommended training for all new analysts. There are additional calibration training materials available through the same link on the homepage.

11.0 ANALYTICAL PROCEDURES

11.1 Fill reagent containers with the persulfate (7.5) and acid reagents (7.6), and also the three DI water containers.

11.2 Make sure gas is on. The gas is left on at the tank at all times. The instrument turns the gas on and off within the instrument. Turn the instrument on.

11.3 Create a run as follows:

- TOC icon
- Sample table editor
- OK
- New file
- Sample run
- System: TOCVW ASI
- OK
- Save with file name (ex: 9-30-2003-dih-TOC)
- Save
- At position 1 press "autogenerate" icon
- Choose method from list (choose from three methods depending on how many injections are needed).
- Next
- Enter number of samples to be run
- Enter start position
- Next
- pg. 3 Next
- pg.4 Next
- At sparge/acid window fix vial numbers if needed
- OK
- Continue with other methods if needed
- Enter the sample ID and dilution in the sample ID box.

- Press the “connect” icon
- The instrument will “warm up and stabilize”.

11.4 Fill vials with at least 20 mL of samples (or 20 mL of a diluted portion) and place in the appropriate position.

11.4.1 Dilutions are made using disposable 1 mL, 5 mL or 10 mL pipets, and graduated cylinders.

11.4.2 If the DI water to be used is greater than 15 mL, the water is measured with a graduated cylinder, otherwise a pipet is used.

11.4.3 If less than 2 mL of the sample is needed, a pipet is used, otherwise a graduated cylinder is used.

11.4.4 See examples in Table 2.

11.5 The run should be as follows:

- TOC CCV (25 mg/L)
- TIC CCV (25 mg/L)
- CCB / Blank
- TOC LCS
- TOC LCS Duplicate
- 8 samples
- CCV
- CCB
- Continue run with samples and all required QC.
- Run a CCV and CCB every ten samples.
- Finish the run with a CCV / CCB.

11.6 When the instrument is stable press the “START” icon.

11.7 Fix any vial numbers in the sparge window.

11.8 Uncheck the “ADD ACID” box

11.9 The run will begin

11.10 Any sample with a TC value over 50 mg/L will need diluted and reanalyzed.

12.0 DETAILS OF CALCULATIONS

12.1 The instrument calculates and prints out TC, TIC, and TOC results in mg/L. These results do not take into account dilution factors.

12.2 To correct for the dilution, the following formula is used:

$$(instrument\,readout) * (dilution) = mg/L\ TOC$$

where:

readout = Answer obtained from instrument
dilution = Dilution of the sample (ex: *A 1/5 Dilution = 5*)

12.3 If leachable organic carbon is needed, the formula is:

$$[instrument\,readout/(dilution)] * (volume/weight) = mg/Kg\ LOC$$

where:

weight - weight in mg of sample volume: volume in mL of water used to shake
volume - volume in mL of water used to leach the sample.

12.4 The following equation is used to calculate the LCS recovery:

$$\%R = \left(\frac{C_x}{C_t}\right)100$$

where:

C_x = the concentration of the analyte in the LCS
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

12.5 The following equation is used to calculate the spike recovery:

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

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 The following equation is used to calculate the duplicate 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 If Method 9060A is required, the average of the four results is reported.

13.0 QUALITY CONTROL (QC) REQUIREMENTS

NOTE: See project QAPP's for more specific QC requirements.

NOTE: See Table 1 for QC summary

13.1 Separate Blanks, LCS, Dup, MS are run for each method.

13.2 TOC CCV (25 mg/L): Dilute 5 mL of the standard stock (7.1) to 200 mL and analyze exactly like the samples. The result should be ± 15% of true value, or the affected samples will need reanalyzed and/or a calibration curve ran.

For North Carolina, the CCV must be +/- 10%.

13.3 TIC CCV (25 mg/L): Dilute 5 mL of TIC stock (7.3) to 200 mL. Criteria is the sample as step 13.1.

13.4 CCB / Method blank: Analyze DI water exactly like the samples. Results will be evaluated down to one half of the RL. The analyst will prepare an NCR to determine the appropriate correction if the blank exceeds this level.

13.5 LCS/LCSD (25 mg/L): Dilute 5 mL of TOC LCS stock (7.2) to 200 mL and analyze exactly like the samples in duplicate. The recovery must be within the

control limits (see Table 1), otherwise the analyst will prepare an NCR to determine the appropriate correction.

13.6 MS/MSD: Add 0.4 mL of TOC LCS stock (7.2) to 40 mL of sample and analyze. The recovery must be within the statistically determined limits (see Section 4.0) or a case narrative will be written. An MS/MSD is required every 10 samples for method 9060A.

13.7 Duplicate: Analyze any sample in the batch in duplicate at the same dilution. The RPD should be less than the statistically determined RPD (see Table 1) or the run will be examined and reanalyzed as necessary. No duplicate is necessary for Method 9060A.

13.8 A batch (workgroup) is defined as a blank, LCS, LCS Duplicate, Spike, duplicate, and up to 20 samples. Several batches may be run in sequence on any particular day.

13.9 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.9.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Steps 13.2-13.8, does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Steps 13.2-13.8.

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)

13.9.2 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.

13.9.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 QA/QC data is reviewed and reported to the client upon request. Data review uses the attached checklist (Figure 1). All data is calculated and reviewed by the analyst. The supervisor (or designated person) performs a second review before final uploading into the LIMS system.

14.2 TOC is reported in mg/L TOC using a maximum of three significant figures. The analyst, date and time are reported.

15.0 PREVENTATIVE MAINTENANCE

15.1 Daily

- Verifiy dilution water is sufficient
- Verifiy sufficient reagents for analyses
- Verify drain vessel is full
- Verify waste container is not full
- Verify there is sufficient gas
- Check for leaks

15.2 Periodically (as required)

- Replace CO_2 absorber
- Replace Halogen scrubber
- Replace Syringe plunger tip
- Wash the TC reactor
- Wash the IC reactor

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.

Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33, Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA, and Ohio VAP rules allow the suspension of our certification for failure to comply with these laws.

16.2 Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.3 All reagents and samples and instrument waste are disposed of down the sink (near or in a hood) after neutralization and flushed with a large amount of water.

16.4 Upon completion of the analysis, the samples are stored in a designated area of the archive storage room to await proper disposal by the waste management team.

17.0 REFERENCES

17.1 "Methods for Chemical Analysis of Water and Wastes", EPA/600/4-79/020, Method 415.1 (Combustion or Oxidation), 1983.

17.2 SW846 Method 9060A

17.3 Standard Method 5310C, 2000 (2011 Editorial Revision).

17.4 Shimadzu TOC-VWP user's manual

17.5 40 CFR Part 136

17.6 Microbac SOP LQAP "Laboratory Quality Assurance Plan"

17.7 Microbac SOP 45 "Method Validation Procedures"

17.8 Microbac SOP 33 "Laboratory Waste Management"

17.9 Microbac SOP GP-CAPA "Corrective Action/Preventive Action: Initiating, Tracking and Monitoring"

17.10 Microbac SOP GP-RCA "Root Cause Analysis"

17.11 Microbac SOP K0001 "Glassware Washing"

Table 1
Quality Control Criteria
Total Organic Carbon (TOC)

CONTROL ITEM	FREQUENCY	ACCEPTABLE CRITERIA	CORRECTIVE ACTION
Initial Calibration Curve	When needed or project defined	R ≥ 0.995	Reanalyze calibration curve
Initial Calibration Verification (ICV)	After calibration	± 10%	See Sections 10.3 and 10.4
Continuing Calibration Verification (CCV)	Every 10 analyses	± 15% NC: ± 10%	See Sections 13.1, 13.2, and 13.3
Continuing Calibration Blank (CCB)	After CCV	≤ ½ RL	See Section 13.4
Method Blank	One per batch (20 samples maximum per batch)	≤ ½ RL	See Sections 4.1, 13.4
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	One per batch (20 samples maximum per batch)	85 – 115% or project specified	See Section 4.4, 13.5
Matrix Spikes/ Matrix Spike Duplicate (MS/MSD) (MSD if required)	One MS per batch of 20 9060A: MS/MSD every 10 samples	Same as LCS criteria	See Sections 4.4, 13.6
Sample Duplicate	One per batch (20 samples maximum per batch)	RPD ≤ 15%	See Sections 4.4, 13.7

Table 2
Examples of Common Dilutions

Dilution	Total Volume Needed	Sample (mL)	Deionized water (mL)
1/2	40	20	20
1/3	30	10	20
1/4	20	5	15
1/10	20	2	18
1/50	50	1	49
1/100	100	1	99

*Dilutions over 1/100 will need to be made in a series of dilutions. Ex: 1/5000 would be made as a 1/50 on a 1/100.

Figure 1

Checklist ID: 27469

Microbac Laboratories Inc.

Data Checklist

Date: 14-APR-2008
Analyst:
Analyst: NA
Method:
Instrument:
Curve Workgroup: NA
Runlog ID:
Analytical Workgroups:

Calibration/Linearity	
Second Source Check	
ICV/CCV (std)	
ICB/CCB	
Blank	
LCS/LCS Dup	
MS/MSD	
Duplicate	
Upload Results	
Client Forms	
QC Violation Sheet	
Case Narratives	
Signed Raw Data	
STD/LCS on benchsheet	
Check for compliance with method and project specific requirements	
Check the completeness of reported information	
Check the information for the report narrative	
Primary Reviewer	
Secondary Reviewer	DIH
Comments	

Primary Reviewer:
14-APR-2008

Secondary Reviewer:
14-APR-2008

CHECKLIST1 - Modified 03/05/2008
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Microbac

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STANDARD OPERATING PROCEDURES
ORGANIC ANALYSIS OF METABOLIC ACIDS
METHOD 830MBA

Issue/Implementation Date: 23 July 2014

Last Review Date: 23 July 2014

Microbac Laboratories, Inc.
Ohio Valley Division
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Approved by:

Michael D. Cochran, Semi-Volatile Supervisor — Date 7/22/14

Wade T. DeLong, Quality Assurance Officer — Date 7-22-14

Leslie S. Bucina, Laboratory Manager — Date 7/23/14

Document Control # 279

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TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This method is used for the analysis of certain organic acids in water. Sample are filtered and injected into a High Performance Liquid Chromatograph (HPLC). A complete list of the applicable compounds and their reporting limits can be found in Table 1.

1.2 This method is restricted to use by or under the supervision of analysts experienced in the use of HPLC and interpretation of chromatographs. Each analyst must demonstrate the ability to generate acceptable results with this method.

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.

CCV	Continuing calibration verification
COA	Certificate of analysis
DI water	Deionized water
HPLC	High performance liquid chromatography
ICAL	Initial calibration
ICV	Initial calibration verification
LCS	Laboratory control sample
LCV	Low calibration verification
LOD	Limit of Detection
LOQ	Limit of Quantitation
LQAP	Laboratory Quality Assurance Program
MDL	Method detection limit
MS	Matrix spike
MSD	Matrix spike duplicate
NCR	Nonconformance report
RL	Reporting limit
RSD	Relative standard deviation
SOP	Standard operating procedure
QC	Quality control

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

1.4 Updates that effect concentration, vendor choices, reagents, MDLs, RLs and QC limits are subject to change without notice.

2.0 SAFETY PRECAUTIONS

2.1 Compounds and reagents used in this method are acidic. Proper laboratory safety procedures must be followed. This includes but is not limited to the use of lab coats, gloves, fume hoods, and protective eyewear.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Samples are collected in a 250 mL amber glass bottle with 5 mL of 5% H_3PO_4 as a preservative to adjust the pH to < 2. Once collected, samples are stored at ≤ 6° C. Samples have a hold time of 28 days from the date of collection.

4.0 METHOD PERFORMANCE

4.1 Table 1 summarizes the performance data for water analysis. This includes the analyte list, ranges for accuracy and precision, nominal laboratory RLs, and the current laboratory MDLs.

4.2 The laboratory performed an initial assessment of the MDLs 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 1 and 2 and were established using verification procedures outlined in Microbac SOP 45.

4.4 The LOQ are the nominal laboratory RLs and were established as per Microbac SOP 45.

4.5 Precision and accuracy data were derived from an initial demonstration of capability using spiked control samples. The laboratory uses results from LCS to assess precision/accuracy and to annually evaluate the associated control limits.

4.6 AFCEE and other specific QA objectives may be found in the appropriate statement-of-work or QAPP.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Other organic acids and matrix artifacts may interfere. These artifacts may show up in the chromatograms as peaks, and may interfere with the compounds of interest. Care is taken to keep all contamination from entering into the sample

preparation procedures via glassware or through solvents. This in no way will prevent all contamination from occurring. If a method blank finds any of the target analytes at a level at or above the RL, the entire batch of samples are re-analyzed.

6.0 EQUIPMENT AND SUPPLIES

6.1 HPLC and Related Equipment:

Hewlett Packard 1050 HPLC System or equivalent
HPLC pump
Autosampler unit
UV Detector
Vacuum Degasser
HP3365 Series 2 Chem Station
Supelco Supelcogel H Column, 25 cm x 4.6 mm ID

6.2 Filter - 0.45 um

6.3 Syringe (gas tight) - 10 uL, 100 uL, 1 mL

7.0 STANDARDS AND REAGENTS

7.1 All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.2 HPLC grade water (J.T. Baker 4218-03 or equivalent)

7.3 phosphoric acid (J.T. Baker 0260-05 or equivalent)

7.4 Stock standards are prepared from neat materials by weighing 0.1 g each of lactic acid, 85+%, solution in water (Sigma-Aldrich 252476-100G) , acetic acid (Chem Service O-4) , propionic acid (Chem Service O-25), and butyric acid (Chem Service O-5) into a 100 mL volumetric flask and filling to volume with 0.1% phosphoric acid. Pyruvic acid (Chem Service O-56) is prepared in the same manner in a separate 100 mL volumetric flask. These stock standards contain the analytes at a 1000 mg/L and are stored at room temperature for up to six (6) months.

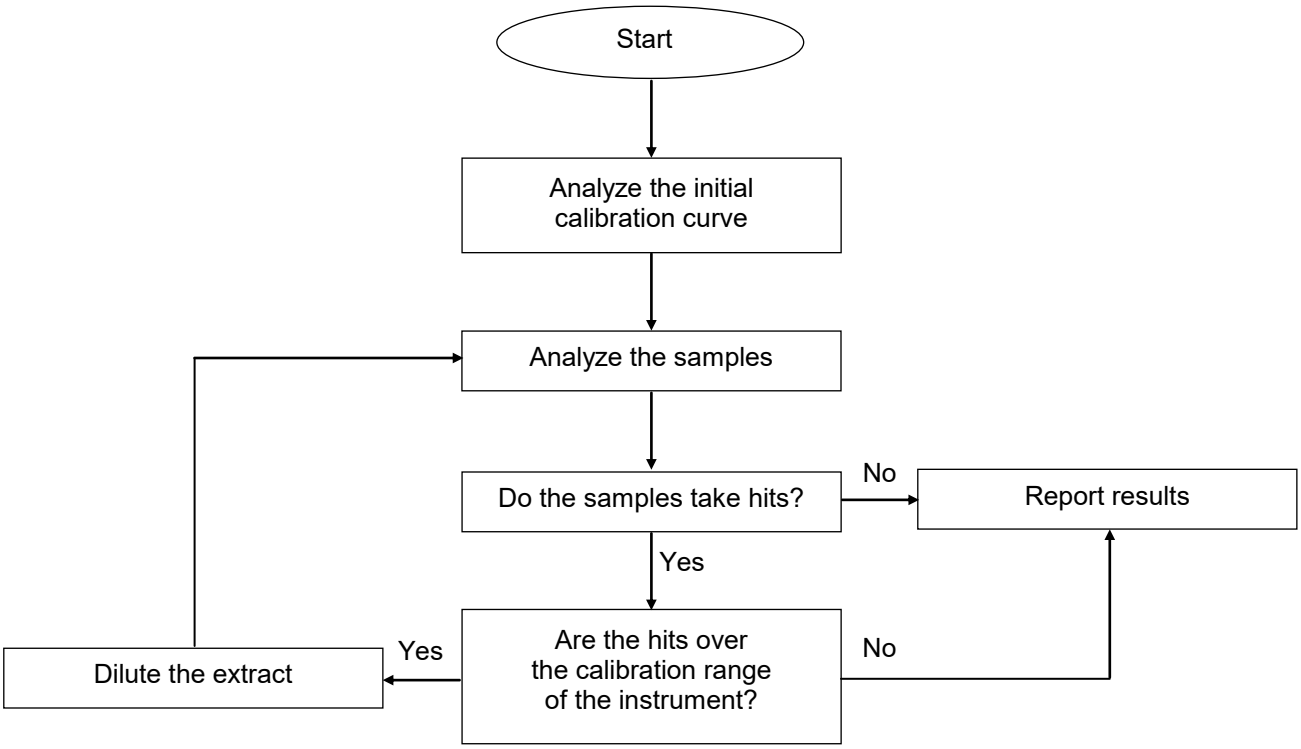
7.5 An intermediate standard solution is prepared by placing 1000 uL of the mixed acid stock solution and 100 uL of the pyruvic acid stock solution into a 10 mL volumetric flask and filling to volume with 0.1% phosphoric acid. The intermediate standard contains lactic acid, acetic acid, propionic acid, and butyric acid at 100 mg/L and pyruvic acid at 10 mg/L.

7.6 Calibration standards are prepared in vials from the intermediate standard using the following scheme:

Calibration STD #	Concentration	Preparation
6	100 mg/L	Intermediate solution 7.4
5	50 mg/L	500 uL of std. 6 plus 500 uL 0.1% H_3PO_4
4	20 mg/L	200 uL of std. 6 plus 800 uL 0.1% H_3PO_4
3	10 mg/L	100 uL of std. 6 plus 900 uL 0.1% H_3PO_4
2	5 mg/L	100 uL of std. 5 plus 900 uL 0.1% H_3PO_4
1	1 mg/L	100 uL of std. 3 plus 900 uL 0.1% H_3PO_4

7.7 ICV: A 20 mg/L ICV standard is prepared independently from the initial calibration standards using the procedure in Sections 7.4, 7.5, and 7.6.

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



9.0 SAMPLE PREPARATION

9.1 All samples are filtered at 0.45 micron and analyzed with no extraction.

10.0 CALIBRATION PROCEDURES

10.1 Recommended HPLC conditions:

Injection Volume:	80 uL
Flow Rate:	0.25 mL/min
Eluent (isocratic):	0.1% phosphoric acid
UV Detector:	210nm
Temperature:	35° C
Run Time:	approximately 40 minutes

10.2 For the initial calibration 80 uL of each calibration standard is injected and a linear curve is determined for concentration versus peak area count of each analyte. The correlation coefficient must be ≥ 0.995.

10.3 An ICV must be performed. Immediately after the analysis of the calibration a second source standard at the continuing calibration level is analyzed. The ICV must have a %D less than 20 (Section 12.4). Inability to achieve this level of verification indicates a problem and will necessitate discerning and resolving the problem and performing a new calibration.

10.4 A CCV must be injected after every ten (10) samples have been analyzed. This is done to determine if the response of the analytical system has changed. Details for calculating the %D for the CCV can be found in Section 12.4 of this document. A %D of less than 20 must be achieved to permit continuing to analyze samples. If a CCV fails to yield a %D of less than 20 then a second CCV may be analyzed from the same source as the first. Failure of two consecutive CCV's requires that the problem be discerned and recalibration may be required. Any samples not bracketed between two passing CCV's must be re-analyzed.

10.4.1 It is possible that certain sample matrices may cause a cumulative baseline rise that will interfere with CCV responses. If this effect is observed, the runtime may be doubled (i.e.: 80 minutes) to remove the interferences. The entire analytical sequence must adhere to this runtime modification.

10.5 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 The instrument(s) are calibrated and the analytical sequence proceeds as follows:

Analytical batch sequence:
ICAL or CCV
Method blank
LCS, LCSD
7 samples (8 if no LCSD; including MS/MSD, sample/sample duplicate)
Solvent rinse (optional)
CCV
10 samples
CCV standard
etc., with a CCV as the last injection of the sequence

11.2 A report of ND (not detected) is a form of quantitation and must therefore adhere to these rules. The list of compounds requested by the client will constitute the target compounds to be analyzed. Calibration standards which fail criteria for compounds not on the target compound list, will not be considered when interpreting the check standard. Only target compounds, as defined by the clients request will be reported.

11.3 Retention time windows are established in accordance with SW-846 Method 8000B. They are established at initial method setup and repeated when there is a column change or a major modification to the method. The width of retention time windows are defined as ± three times the standard deviation of the retention time of a standard analyzed on three consecutive days. The retention time windows are calculated by analyzing three injections of a standard over the course of a 72 hour period, calculating the standard deviation of the target analytes' retention time, and multiplying each of the standard deviations by 3. The retention time window for a particular analyte then is ± this value. The center of the retention time window is the retention time of each analyte taken from the continuing calibration verification standard run at the beginning of a daily sequence.

11.4 After sample analysis, the data system produces a quantitation report listing target analytes and concentrations found in the sample. A compound is

considered tentatively identified if it falls within the calculated retention time window.

11.5 If the response for any target analyte exceeds the initial calibration range, the sample must be diluted. Dilutions are prepared so that the majority of compounds above the calibration range fall near the midpoint of the calibration. Dilutions are prepared by using syringes to transfer aliquots of extract into appropriate amounts of solvent in autosampler vials. Examples are presented below:

Dilution	Amount Sample Extract (uL)	Amount 0.1% H_3PO_4 (uL)	Final dilution volume (uL)
2x	200	200	400
5x	100	400	500
10x	100	900	1000
20x	50	950	1000

Higher dilutions are prepared by performing serial dilutions, e.g. for a 100x dilution, a 10x dilution is diluted again by a factor of 10.

11.6 After the raw data is processed, it is uploaded into the LIMS.

11.7 The analyst will perform a primary review and data verification, followed by a secondary review by a peer for quality and completeness.

12.0 DETAILS OF CALCULATIONS

12.1 Calibration factor (CF) from external standard calibration:

$$CF = \frac{A_s}{C_s}$$

where:

A_S = Peak area for the analyte or surrogate in the external standard
C_S = Concentration of the external standard (ug/L)

12.2 Mean calibration factor (CF) = $\overline{CF}$

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

12.3 Calculation of standard deviation. The standard deviation is calculated using the formula:

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

12.4 The % difference or drift is calculated using the formula:

$$\%D = \left| \frac{(C_t - C_x)}{C_t} \right| 100$$

where:

C_t = True concentration of the analyte or surrogate in the standard
C_x = Measured concentration of analyte or surrogate in the standard

12.5 The % RSD is calculated using the formula:

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

where:

s = Standard deviation
x = Average

12.6 Coefficient of correlation

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

where:

X = individual values of the independent variable, i.e. concentration
Y = individual values of the dependent variable, i.e. response
n = number of pairs of data

12.7 The LCS recovery is calculated as follows:

$$\%R = \left(\frac{C_x}{C_T}\right)100$$

where:

C_x = the concentration of the analyte in the LCS.
C_t = the theoretical spike concentration.
$\%R$ = percent recovery

12.8 Calculation of % Recovery

$$\%R = \left[\frac{\left(C_{spk} - C_x\right)}{C_t}\right]100$$

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

12.9 Calculation of RPD

$$RPD = \left[\frac{\left|C_1 - C_2\right|}{(C_1 + C_2)/2}\right]100$$

where:

C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

12.10 Calculation of concentration of analyte using calibration factor

$$C_x = \frac{(A_x)(D)}{(\overline{CF})}$$

where:

A_x = Peak area for the analyte or surrogate
D = Dilution factor for the samples (1/10 = 10)
CF = Mean CF for the analyte or surrogate from
C_x = Calculated concentration of the analyte or surrogate (ug/L)

12.11 Linear calibration calculations (external standard method)

12.11.1 The response ratio is plotted vs. the concentration ratio giving a linear equation:

$$y = mx + b$$

where:

y = Response(area) = R
x = Concentration = C_i

And m and b are the slope and intercept from the regression equation

For a given response ratio we can solve for C_i:

12.11.2 $$C_i = [R - b]/m$$

Use equations 12.4 or 12.5 to calculate the sample concentration, C_x.

12.12 Solving for the concentration in water samples using linear.

For a given concentration, compute the unknown, C_x

$$C_x = (C_i)(V_f/V_i)(D)(1000)$$

where:

C_i = concentration in the extract (ug/mL)
V_f = final sample (extract) volume (mL)

V_i = initial sample volume (mL)
D = dilution factor
C_x = concentration in the sample (ug/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 Method blank, LCS, MS/MSD. 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 3. The MS/MSD samples may be waived if insufficient sample is available.

13.2 A batch is defined as a group of samples, which are prepared together. A batch contains a maximum of 20 samples. With each batch of samples extracted, a LCS and a Method blank must also be extracted. It is recommended that at least one sample for the batch be extracted three times. The last two extractions are fortified with a spiking solution to provide an MS and MSD. All QC samples must undergo the identical extraction and cleanup procedures as each sample in the batch. A standard containing the analytes of interest is used as the spike in the LCS, MS, and MSD. The LCS, MS and MSD are spiked so as to yield a concentration that falls within the mid-point of the curve.

13.3 The Method blank cannot contain amounts of any target analytes, which are over the RL. If any target analytes are found in the method blank with concentrations higher than the RL, the entire batch must be re-extracted and the analysis performed again. All blanks are evaluated down to the current MDL for the presents 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.

13.4 The LCS must be evaluated for the acceptance criteria listed in Table 1 and for any project specific criteria which may be more, or less, stringent. 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 Table 1 is used in the absence of a project QAPP, or program specified control limits. If any 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. Corrective action will consist of re-extraction and re-

analysis of the affected samples, unless the client's representative and the Quality Assurance Officer (QAO) approve of another course of action.

13.5 Retention time studies are performed at initial method setup and are repeated after each column change or change in the instrument run condition.

13.6 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).

13.6.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Table 3 does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Table 3.

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)

13.6.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RCA, corrections, corrective action(s) and evidence of effectiveness.

13.6.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank is greater than or equal to the reporting limit, but the corresponding sample results are non-detect.
- One or more surrogates are high and the samples are non-detect.
- The continuing calibration verification (bracketing) is biased high and the samples are non-detect.
- There is insufficient sample volume or weight to perform a corrective action.
- The sample has exceeded holding time.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data Review

Prior to data entry into the LIMS (either manual or automatic), all data must undergo two (2) levels of review in the department. The primary review is performed by the analyst and the secondary review is performed by either the department supervisor (or a designee) or another qualified analyst.

14.2 Data Reporting

The reporting requirements depend upon the need of the client. Microbac offers four levels of data reporting which are described in some detail below.

14.2.1 Level 1 reporting provides the client with the results for all samples submitted for analysis. No other documents or raw data are provided with this level of report.

14.2.2 Level 2 reporting provides the client with all of the information contained in a Level 1 report plus a summary of all of the QC analysis associated with the samples submitted by the client.

14.2.3 Level 3 reporting is essentially a custom report provided to the client that contains any additional data from the analysis that the client might request.

14.2.4 Level 4 reporting is provided in those cases where the client wishes to perform full data validation. All raw data, lab generated logs, and other associated data are provided.

14.2.5 Results for water samples are reported in mg/L to three significant figures.

15.0 PREVENTIVE MAINTENANCE

15.1 System pressures are monitored daily to indicate any changes in the HPLC which can include leaks, restrictions, temperature and solvent fluctuations, and gas bubbles.

15.2 A guard column to protect the system and the analytical column may be used.

15.3 Use only HPLC grade solvents and filter all samples at 0.45 um to prevent introducing materials, which can lead to restrictions in the system.

15.4 Follow the vendor's instruction manual for other preventive maintenance indicated for each HPLC module.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste, which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2 This method generates wastes in the form of sample extracts in vials, which are placed in the satellite waste container labeled for Waste Vials/Sample Extracts (D001, F002).

16.3 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33, Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

17.0 REFERENCES

17.1 Test Methods for Evaluating Solid Waste - Physical/Chemical Methods (SW-846) Third Edition, US-EPA - Method 8000.

17.2 Microbac SOP 33, “Laboratory Waste Management”

17.3 Microbac SOP LQAP, “Laboratory Quality Assurance Plan”

17.4 Microbac SOP 45, “Method Validation Procedures”

17.5 Microbac SOP GC-CAPA, “Corrective Action/Preventive Action: Initiating Tracking and Monitoring”

17.6 Microbac SOP GC-RCA, “ Root Cause Analysis”

TABLE 1
MICROBAC'S QA OBJECTIVES AND ANALYTICAL METHODS FOR METABOLIC ACID ANALYSES OF GROUNDWATER

PARAMETER	CAS #	ACCURACY (% RECOVERY)*	PRECISION (% RPD)*	MDL WATER (mg/L)	REPORTING LIMITS WATER (mg/L)
acetic acid	64-19-7	90-110	0-30	0.5	1.0
butyric acid	107-92-6	90-110	0-30	0.5	1.0
lactic acid	50-21-5	90-110	0-30	0.5	1.0
propionic acid	79-09-4	80-115	0-30	5.0	10.0
pyruvic acid	127-17-3	90-110	0-30	0.05	0.1

* Values are statistically derived from laboratory control samples and are updated annually. Actual control limits may vary.

TABLE 2
CALIBRATION STANDARDS (mg/L)

ANALYTE	STANDARD					
	# 1	# 2	# 3	# 4	# 5	# 6
acetic acid	1	5	10	20	50	100
butyric acid	1	5	10	20	50	100
lactic acid	1	5	10	20	50	100
propionic acid	NA	5	10	20	50	100
pyruvic acid	0.1	0.5	1	2	5	10

TABLE 3
QUALITY CONTROL CRITERIA
METABOLIC ACIDS
METHOD 830-MBA

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration (ICAL)	Initially and upon failure of two consecutive CCV's	Linear regression with $r \geq 0.995$ for each analyte	Evaluate cause; Repeat calibration; or Qualify data and discuss in narrative (1)
Second source calibration verification (ICV)	After each initial calibration	≤ 20% drift for each analyte (1)	Re-analyze ICV; upon second failure, repeat initial calibration (1)
Continuing calibration verification (CCV)	Daily, before sample analysis, every ten samples, and at the end of the analysis sequence	≤ 20% drift for each analyte (1)	Re-analyze CCV, upon second failure, repeat initial calibration (1)
Method Blank (MB)	One per matrix/batch: Maximum of 20 samples	< project report limit for each target analyte (1)	Notify supervisor and initiate NCR; Investigate; repeat samples or qualify data and address in the report narrative (3)
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; repeat samples or qualify data and address in the report narrative (3)
Matrix Spikes/ Matrix Spike Duplicate (MS/MSD)	One per matrix/batch: Maximum of 20 samples per batch	Target compounds within the designated range (1)	Qualify data and/or address in the report narrative

(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.
(3) Data will be qualified if sample volume is insufficient for re-extraction/re-analysis.

Figure 1
Semi-Volatile HPLC Checklist

Checklist ID: 28180

Microbac Laboratories Inc.

Data Checklist

Date: 05-MAY-2008
Analyst: HAV
Analyst: NA
Method: 830-MBA
Instrument: HPLC3
Curve Workgroup: NA
Runlog ID: 22009
Analytical Workgroups: L08040001 (QMDL), L08040002 (QMDL), L08040808

ANALYTICAL	
System Performance Check	X
DFTPP (MS)	NA
Endrin/DDT breakdown (8081/MS)	NA
Pentachlorophenol/benzidine tailing (MS)	NA
Eluent check (IC)/system pressure (HPLC)	X
Window standard (FID)	NA
Initial Calibration	NA
Average RF	NA
Linear regression or higher order curve	NA
Alternate source standard (ICV) % Difference	NA
Continuing Calibration (CCV)	X
% D/% Drift	X
Minimum response factors (MS)	NA
Continuing calibration blank (CCB) (IC)	NA
Special standards	NA
Blanks	X
TCL hits	X
Surrogate recoveries	NA
LCS/LCSD (Laboratory Control Sample)	X
Recoveries	X
Surrogate recoveries	NA
MS/MSD/Sample duplicates	NA
Recoveries	NA
%RPD	NA
Samples	X
TCL hits	X
Mass spectra (MS/HPLC)/2nd column confirmations (ECD/FID/HPLC)	NA
Surrogate recoveries	NA
Internal standard areas (MS)	NA
Library searches (MS)	NA
Calculations & correct factors	X
Compounds above calibration range	NA
Reruns	NA
Manual integrations	NA
Project/client specific requirements	X
REPORTING	
Upload batch form	X
KOBRA workgroup data/forms/bench sheets	X
Case narratives	X
Check for completeness	X
Primary Reviewer	HAV
SUPERVISORY/SECONDARY REVIEW	
Check for compliance with method and project specific requirements	X
Check the completeness/accuracy of reported information	X
Data qualifiers	X
Secondary Reviewer	MDC

Primary Reviewer:
06-MAY-2008

Secondary Reviewer:
06-MAY-2008

CHECKLIST1 - Modified 03/05/2008
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Microbac

Figure 2
Semi-Volatile HPLC Runlog

Run Log ID: 22009

Microbac Laboratories Inc.
Instrument Run Log

Instrument: HPLC3 Dataset: 050508
Analyst1: HAV Analyst2: NA
Method: 830-MBA SOP: HPLC03 Rev: 1

Maintenance Log ID: 23999

Column 1 ID: SUPELCOGEL H Column 2 ID: NA
Workgroups:
Internal STD: NA Surrogate STD: NA Calibration STD: STD24689
Comments:

Seq.	File ID	Sample Information	Mat	Dil	Reference	Date/Time
1	3L009168.F	SOLV BLK	1	1		05/05/09 12:09
2	3L009169.F	SOLV BLK	1	1		05/05/09 12:46
3	3L009170.F	WG270362-01 CCV	1	1		05/05/09 13:23
4	3L009171.F	WG270393-01 BLK	1	1		05/05/09 14:46
5	3L009172.F	L08040808-01	1	1		05/05/09 15:25
6	3L009173.F	L08040808-02	1	1		05/05/09 16:02
7	3L009174.F	L08040808-03	1	1		05/05/09 16:38
8	3L009175.F	L08040808-04	1	1		05/05/09 17:15
9	3L009176.F	L08040001-01 QMD	1	1		05/05/09 17:52
10	3L009177.F	L08040002-01 QMD	1	1		05/05/09 18:29
11	3L009178.F	L08040001-01 QMD	1	1		05/05/09 19:05
12	3L009179.F	L08040002-01 QMD	1	1		05/05/09 19:42
13	3L009180.F	WG270393-02 LCS	1	1		05/05/09 20:19
14	3L009181.F	WG270362-02 CCV	1	1		05/05/09 20:55
15	3L009182.F	WG270393-03 LCS	1	1		05/05/09 21:32
16	3L009183.F	WG270362-03 CCV	1	1		05/05/09 22:09

Comments

Seq.	Rerun	Dil.	Reason	Analytes
9				
	MDL was only for propionic @10ppm			
10				
	MDL was only for propionic @10ppm			

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MICROBAC®

STANDARD OPERATING PROCEDURES
ANALYSIS OF DISSOLVED GASES
IN GROUND WATER
EPA RSKSOP-175

Issue/Implementation Date: 26 June 2014

Last Review Date: 26 June 2014

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

Approved by:

Michael D. Albertson, Volatiles Supervisor — Date: 6/26/14

Wade T. DeLong, Quality Assurance Officer — Date: 7-8-14

Leslie S. Bucina, Laboratory Manager — Date: 7/8/14

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 Microbac Standard Operating Procedure RSK01 pertains to the analysis of light gases dissolved in an aqueous matrix. A list of target analytes can be found in Table 1.

1.2 The concentration of dissolved gases in the aqueous sample is determined by analysis of an aliquot of headspace. The aliquot is injected onto the column and response is measured by a Flame Ionization Detector and a Thermal Conductivity Detector on a Gas Chromatograph.

1.3 This method references RSKSOP-194, Revision 3, January 2005, US-EPA, and RSKSOP-175, Revision 2, May 2004, US-EPA, however, those procedures have been modified as described herein. Microbac SOP RSK01 applies to all headspace analysis of dissolved gases, except where client's specific Quality Assurance Project Plan overrides this method's quality assurance plan.

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.

AFCEE	Air Force Center for Environmental Excellence
CCV	Continuing calibration verification
COD	Coefficient of Determination
DI water	Deionized water
FID	Flame Ionization Detector
GC	Gas Chromatography
ICAL	Initial calibration
ICV	Initial calibration verification
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
LIMS	Laboratory Information System
LOD	Limits of Detection
LOQ	Limits of Quantitation
MB	Method blank
MDL	Method detection limit
MS	Matrix spike
MSD	Matrix spike duplicate
NCR	Nonconformance report
PPE	Personal Protective Equipment
QAPP	Quality Assurance Project Plan

QA/QC	Quality Assurance/Quality Control
RGT	Reagent
RL	Reporting limit
RSD	Relative Standard Deviation
RT	Retention time
SDS	Safety Data Sheets
STD	Standard
TCD	Thermal Conductivity Detector
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compounds

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

2.0 SAFETY PRECAUTIONS

2.1 Standard laboratory safety procedures must be followed when working with unknown samples. Gloves should be worn while handling any chemicals, standards, or samples. Other required PPE include lab coats and safety glasses with sideshields.

2.2 **WARNING:** The following VOC's have been tentatively classified as known or suspected human or mammalian carcinogens:

benzene	carbon tetrachloride
chloroform	vinyl chloride

The toxicity or carcinogenicity of the other reagents and analytes used in this method have not been precisely defined, therefore, each chemical should be treated as a potential health hazard. Exposure to the compounds should be reduced to the lowest possible level. Procedures involving concentrated samples and primary standard preparation should be performed in a fume hood.

2.3 SDS for standards and reagents used within the laboratory are available to all employees. SDS should be consulted prior to handling chemicals.

2.4 **CAUTION/WARNING:** Precaution must be used when preparing standards for analysis. Should a gas cylinder leak uncontrollably, serious injury or death by asphyxiation or fire and burns may occur. Inspect each septa in the gas cylinder's syringe adapter daily. Replace any septa exhibiting excessive puncture marks. Cylinder release valves must be closed after each use. The

gases used in this method are flammable at higher concentrations. Use caution when handling the cylinders and keep them away from heat and flame. Refer to Microbac SOP CHP04 for more information regarding the handling of compressed gases.

2.5 **CAUTION/WARNING:** Should the column be disconnected for any reason, the hydrogen supply must be turned off at the instrument and/or the gas supply itself. Alternatively, the column fitting at the detector may be capped off with the appropriate fitting. This will only be performed if the column is disconnected for a short period of time. If hydrogen flows into the heated oven through the disconnected column fitting an explosion may occur.

2.6 Over-pressurization of auto-sampler vials during manual sample preparation may cause the vials to explode.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Pre-cleaned 20 mL glass headspace screw top vials with Teflon-faced silicon septa must be used for samples.

3.2 Sample vials must be filled without headspace. Each sample should be collected in triplicate to ensure proper sample volume for analysis.

3.3 Samples preserved with HCl (pH $\leq$ 2) must be analyzed within 14 days of sample collection. Unpreserved samples (pH $>$ 2) must be analyzed within 7 days of sample collection. All samples stored at ≤ 6° C.

3.4 Upon completion of sample analysis and hold time expiration, samples are removed from storage units and returned to sample archive. Samples requiring internal chain-of-custody are returned to sample receiving custodian to complete the chain.

3.5 At a minimum 20 mL of sample is required for analysis.

3.6 Samples collected in vials other than 20 mL glass screw cap vials will be transferred to a 20 mL screw cap vial prior to making head-space.

4.0 METHOD PERFORMANCE

4.1 Table 2 summarizes the performance data for water analysis. This table includes the analyte list, ranges for accuracy and precision, nominal laboratory RLs, and true values. The laboratory evaluates standard control limits for accuracy through annual review of LCS. Project-specific control limits may supersede the standard limits in Table 2.

MDLs are derived in accordance with 40 CFR Part 136 for Method RSK175. MDLs presented in Table 2 are updated, or verified, annually. Verification consists of analyzing a fortified blank (MDL check standard) spiked at a concentration near the concentration of the reported laboratory MDL. This MDL check standard is used to verify that the laboratory MDL is routinely achievable over the course of time. The actual instrument detection limit may be lower. Precision and accuracy data were derived from LCS results from the previous year and verified annually. RLs are nominal laboratory values, but project RLs may vary.

4.2 The laboratory performed an initial assessment of the MDLs using the procedures outlined in 40 CFR Part 136. Results are filed electronically at H:\DATA\COMMON\MDL.

4.3 The LOD, or verified MDL, are presented in Table 2 and were established using verification procedures outlined in Microbac SOP 45.

4.4 The LOQ are the nominal laboratory RLs and were established per Microbac SOP 45. Actual project reporting limits may be higher.

4.5 Precision and accuracy data were derived from an initial demonstration of capability using spiked control samples. The laboratory uses results from LCS to assess precision/accuracy and to annually evaluate the associated control limits.

4.6 AFCEE and other specific QA objectives may be found in the appropriate statement-of-work or QAPP.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Samples for VOA are susceptible to laboratory contaminants. To eliminate the potential for interferences from other areas of the laboratory, the volatiles laboratory has an independent air intake system and positive air pressure is maintained in the laboratory.

5.2 Prior to sample analysis, a method blank is analyzed to demonstrate the system is free of contaminants and interferences.

5.3 Samples may become contaminated by diffusion of volatile organics through the septum into the sample during shipment and storage. A trip blank prepared from organic-free reagent water is carried through the sampling and handling process to serve as a check on such contamination.

5.4 Contamination by carryover can occur whenever high concentration and low concentration samples are sequentially analyzed. Contamination by carryover during a non-monitored run may require the re-analysis of the affected sample(s).

5.5 To minimize potential contamination from organic hydrocarbons in the carrier gas a hydrocarbon trap is installed.

6.0 EQUIPMENT AND SUPPLIES

6.1 Agilent 6890N GC equipped with an Agilent FID/TCD, and Teledyne Tekmar HT3 Headspace Autosampler.

6.2 Varian Archon autosampler for sample preparation (i.e.: create headspace)

6.3 Front column with FID: Restek Rt-Q BOND column 30 m .53 mm ID
Back column with TCD: Restek Rt-Q BOND column 15 m .53 mm ID

6.4 Syringes: Hamilton Samplelock 1 mL, 25 mL, 50 uL, 100 uL, 500 uL;
SGE Analytical Science 10 uL

6.5 Class “A” volumetric flask: 50 mL

6.6 Disposable Pasteur pipets (Kimble): 1 mL, 10 mL

6.7 20 mL glass headspace screw top vials with Teflon-lined screw caps (SUN-SRi)

6.8 pH paper (Macherey Nagel, EMD): Ranges 0-14, 0.3-2.3

6.9 Equivalent equipment and supplies may be used.

6.10 Refer to Table 3 for operating parameters

6.11 Computer, software, hardware:

Instrument	Operation System	Computer Name	Connection Type	Instrument Software
HP16	Windows XP Professional	C10050	1 Gbps	Enviroquant Chemstation C.00.00

7.0 STANDARDS AND REAGENTS

7.1 All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.2 Primary Calibration Standards:

0.1% Standard Mix: 1000 umol/mol mix of methane, ethene, acetylene, ethane and propane (Scotty Analyzed Gases)

1% Standard Mix: 10,000 umol/mol gas mix of methane, ethene, acetylene, ethane and propane (Air Gas)

100% Carbon Dioxide (Air Gas)

7.3 Primary LCS / MS / Alternate Source (ICV) Standards:

1% Standard Mix: 10,000 umol/mol gas mix of methane, ethene, acetylene, ethane and propane (Scotty Analyzed Gases)

100% Carbon Dioxide (Scotty Analyzed Gases)

7.4 Following lists initial calibration standards concentrations:

Initial Calibration Standard Concentration

ANALYTE	ug/L						
methane	-	2.86	28.6	114	228	570	913
ethene	-	5.00	49.9	200	398	997	1596
acetylene	-	4.64	46.4	185	370	926	1482
ethane	2.14	5.36	53.5	214	427	1069	1711
propane	3.14	7.86	78.5	314	626	1568	2510
carbon dioxide	-	9398	23472	31326	62654	156639	250623

7.5 Preparation of working standards:

A 20 mL screw top autosampler vial is filled completely with DI water and capped with no headspace. Headspace is then created per Section 9.2. The desired volume of primary standard is injected into the autosampler vial below the surface of the liquid. The standard is placed on the autosampler where it is heated to 40° C while it equilibrates, vents, and injects. 3 mL of the headspace is then injected onto the GC by the autosampler. **NOTE:** It is assumed the volume of added standard is inconsequential in comparison to the volume of headspace desired. Working standards prepared as follows:

Primary STD Volume (uL)

ANALYTE	0.1% Mix			1% Mix			
methane	-	25	500	100*	200**	500	800
ethene	-	25	500	100*	200**	500	800
acetylene	-	25	500	100*	200**	500	800
ethane	10	25	500	100*	200**	500	800
propane	10	25	500	100*	200**	500	800
100% CO_2							
carbon dioxide	-	30	65	100*	200**	500	800

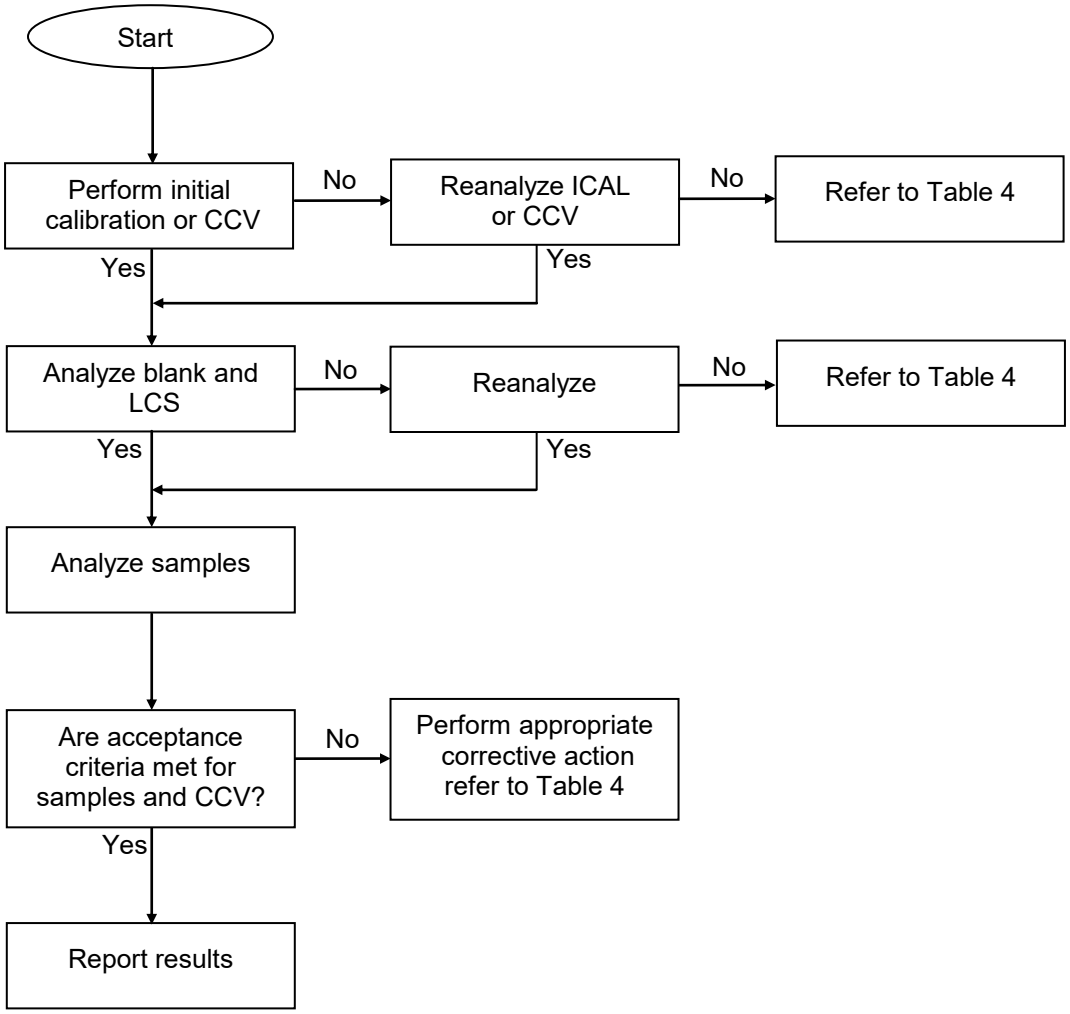
* denotes LCS, MS/MSD preparation
** denotes CCV, ICV preparation

7.6 Water: UV-treated DI water (or equivalent)

7.7 Air Gas helium, hydrogen 99.999%; compressed air.

7.8 Equivalent standards and reagents may be used

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



9.0 SAMPLE PREPARATION

9.1 Sample vials are removed from refrigerated storage and allowed to warm to ambient.

9.2 Sample preparation (create headspace): The preferred mode of sample preparation is the "automated" mode (Section 9.2.1) while "manual" mode is reserved as a contingency preparation mode.

9.2.1 Automated: A 20 mL screw top autosampler vial containing the sample vial is placed on the Archon Autosampler and 15 mL of sample is displaced by the Archon.

9.2.2 Manual: 15 mL of sample is displaced from a 20 mL screw top autosampler vial containing the sample by slowly injecting 15 mL of helium (displaced sample is evacuated via a 3 inch or longer needle attached to the tubing). The displaced sample volume is collected and discarded.

NOTE: Helium should not be rapidly added to the vial, as the vial may become pressurized and/or explode. The vial should also not be under-pressurized during sample preparation resulting in a loss of gases. Under-pressurization would be observed by varying volumes of sample remaining in the vial after preparation.

9.3 The sample vial is placed on the headspace autosampler where 3 mL of headspace is injected onto the GC.

9.4 After analysis is complete, the sample pH is measured and recorded.

10.0 CALIBRATION PROCEDURES

10.1 Configure instrument per Table 3

10.2 External calibration is performed by analyzing no fewer than 5 calibration standards at varying concentrations (refer to Section 7.0). The lowest calibration level must be equal to or below the required reporting limit for each analyte.

10.3 Upon completion of the initial calibration standards analyses, the average calibration factors ($\overline{CF}$) and the percent relative standard deviation (% RSD) are calculated for all target analytes. % RSD must be ≤ 20.

10.4 Alternatively, linear regression or quadratic regression maybe used for the calibration curve. For linear or quadratic calibration, an acceptable curve must have a minimum coefficient of determination (COD) (r^2) of 0.99, for quadratic regression and r ≥ 0.995 for linear regression.

10.5 Upon completion of the initial calibration, an ICV will be analyzed. The preparation and analysis of the ICV will be performed in a separate batch and on a separate day than the initial calibration if the ICV is of the same source as the ICAL Standards. ICV results must be ± 15% of the expected value for all target analytes.

10.6 A CCV is analyzed at the beginning of each analytical batch, every tenth sample thereafter, and at the end of the analytical batch. CCV results must be ± 15% of the expected value for all target analytes (% drift).

10.7 Refer to Table 4 for corrective action.

11.0 ANALYTICAL PROCEDURES

11.1 Configure instrument using parameters found in Table 3 and perform ICAL or CCV per Section 8.0.

11.2 Once calibration requirements are met, a method blank is analyzed to verify that the analytical system is free of interferences.

11.3 Following the analysis of a method blank a LCS is analyzed.

11.4 After the blank and LCS criteria are met samples may be analyzed. Samples may be analyzed until 10 analyses are performed (blanks, LCS's, and MS/MSD's count as analyses, rinses do not). After 10 analyses a CCV is analyzed. Sample analysis may continue as long as the CCV standard meets criteria in Section 8.0. The final analysis of each analytical batch must be a CCV standard. **NOTE:** Blank and LCS analyses must be repeated after 20 sample analyses.

The following illustrates an example analytical batch sequence:

ICAL or CCV
Blank
LCS, LCSD
7 samples (including MS/MSD)

CCV standard
10 samples
CCV standard
etc.
end of run: CCV standard

11.5 All analyses (standards, QC samples, environmental samples) undergo the same sample preparation procedures.

11.6 Failure of the above warrants corrective action including, but not limited to, reanalysis and/or dilution analysis. If reanalysis and/or dilution analysis is required, an unopened vial is used when possible.

11.7 Target analyte results that exceed the upper calibration standard require dilution. A dilution analysis may be performed by taking an aliquot of sample and diluting into DI water using volumetric flasks.

11.8 Analytical batches must also contain an LCS and LCSD or a MS/MSD. Sample/Sample duplicate analyses are not performed to preserve sample volume unless the client specifies sample/sample duplicate and provides enough sample.

11.9 Refer to Section 13.0 for additional QC requirements and corrective action.

12.0 DETAILS OF CALCULATIONS

12.1 Calibration factors (CF) for each analyte are calculated:

$$CF = \frac{A_s}{C_s}$$

where:

A_s = Peak area for the analyte in the external standard
C_s = Concentration of the external standard (ug/L)

12.2 Standard deviation(s):

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

where:

x = CF

12.3 Percent relative standard deviation (%RSD):

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

where:

s = standard deviation
$\bar{x}$ = mean calibration factor

12.4 Mean calibration factor (CF) = $\overline{CF}$

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

12.5 The percent (%) difference or drift is calculated using the formula:

$$\%D = \left[\frac{(C_t - C_x)}{C_t}\right]100$$

where:

C_t = True concentration of the analyte in the standard
C_x = Measured concentration of analyte in the standard

12.6 Instrument results are “in-extract” (umol/mol) concentration and are converted to “in-sample” concentration (ug/L) as follows:

$$ug/L = C_{ex}\left(\frac{MW}{T_F}\right)\left(\frac{HS}{S}\right)DF$$

where:

C_{ex} = headspace concentration, umol/mol
MW = molecular weight of the analyte, ug/umol
T_F = temperature factor = (22.45 L/mol)[(40°C +273)/273]
HS = headspace volume, L
S = sample volume remaining after headspace creation, L

DF = dilution factor

12.7 Laboratory control sample (LCS) percent recovery (% R) calculated as:

$$\%R = \left(\frac{C_x}{C_t}\right)100$$

where:

C_x = the concentration of the analyte in the LCS
C_t = the theoretical spike concentration.
%R = percent recovery

12.8 Matrix spikes (MS/MSD) % R calculated as:

$$\%R = \left[\frac{(C_{spk} - C_x)}{C_t}\right]100$$

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

12.9 Relative percent difference (RPD) calculated as:

$$RPD = \left[\frac{|C_1 - C_2|}{(C_1 + C_2)/2}\right]100$$

where:

C_1 = Concentration of the first sample
C_2 = Concentration of the second sample

12.10 Example calculations for sample are presented in Figure 2.

13.0 QUALITY CONTROL REQUIREMENTS

13.1 This section cites the procedures and analyses required to fully document the quality of data generated by the method. The required components of the laboratory's QA program and specific QC analyses are described in this section. For each QC analysis, the complete analytical procedure, the frequency of required analyses, and interpretation of results are specified.

13.2 Retention time (RT) windows are calculated annually. Three CCV standards are analyzed within 72 hours. Serial injections are not used and each must pass CCV criteria. For each parameter the retention time is recorded and the average and standard deviation are calculated. RT windows are ± 3 standard deviations from the average RT and adjusted daily by centering on the CCV.

13.3 Workgroups are analytical batches that contain calibration verification standards (ICAL, ICV, CCV), QC samples, and client samples.

Workgroups are comprised of:

- 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: (also serves as helium blank).
- LCS/LCSD: verify precision and accuracy of system
- MS/MSD: measure matrix effect of environmental sample on target analytes; measure precision
- Sample/sample duplicate: dual analysis of environmental sample to measure precision
- Environmental sample: samples submitted for analysis

13.4 Method reagent blank preparation: Per Section 9.2 with the exception of DI water in place of a sample. Method blank analyzed per method requirements. Target analytes must be ≤ MDL (see Table 4 for method blank criteria). 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 LCS preparation: Per Section 7.0 criteria: The LCS must be evaluated using acceptance criteria listed in Table 2, 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 Table 2 are used in the absence of a project QAPP or program specified control limits.

13.6 MS/MSD preparation: Per Section 7.0 with the exception of a sample used in place of DI water. 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. In obvious cases of error, reanalysis would be performed.

13.7 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.7.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Table 4, does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Table 4.

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)

13.7.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event

requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RCA, corrections, corrective action(s) and evidence of effectiveness.

13.7.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

13.8 LCS control limits are reviewed annually.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data review:

All data undergoes a 100% primary review to ensure method and project specific compliance, reduction of the data into reportable results, and generation of appropriate QC forms. All items in Figure 1 (data review checklist) are reviewed and results are uploaded to the LIMS.

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 may be performed by the supervisor or designee.

14.2 Data reporting:

Following peer review all uploaded results are reviewed, verified, and qualified.

Default reporting units are "ug/L". **NOTE:** Project specific QAPP may supercede default reporting units.

All results are uploaded to a maximum number of significant figures dictated by the LIMS. The number of significant figures in the final report may vary per project requirements.

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 A target analyte is reported when the peak is within the retention time window, the concentration is at or above the reporting limit, and within the calibrated range of the instrument.

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 checked daily and other maintenance (e.g. clip column, injector port maintenance, clean detector) performed as needed.

15.2 Instrument configuration and maintenance is recorded in the instrument maintenance log-book. Changes to instrument configuration are also recorded in the maintenance log book.

15.3 Trouble-shooting involves, but is not limited to, monitoring chromatography, contamination, standards recoveries, injection port maintenance, and pressurized checks.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2 The waste streams are as follows:

- non-halogenated solvents

16.3 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33 Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous 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, Methods 5021 and 8000.

17.2 RSKSOP-175, USEPA “Sample Preparation and Calibration for Dissolved Gas Analysis in Water Samples Using a GC Headspace Equilibration Technique” Revision No. 2, May 2004

17.3 RSKSOP-194, US-EPA, “Gas Analysis by Micro Gas Chromatographs” Revision No. 3, January 2005

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 GC-CAPA, “Corrective Action / Preventative Action: Initiating, Tracking, and Monitoring”

17.9 Microbac SO GC-RCA, “Root Cause Analysis”

17.10 Microbac SOP CHP04 “Ohio Valley Chemical Hygiene Plan”

17.11 Analysis of Dissolved Methane, Ethane, and Ethylene in Ground Water by a Standard Gas Chromatographic Technique, Journal of Chromatographic Science, Vol. 36, May 1998, Don H Kampbell and Steve A. Vandegrift

Table 1
RSK01 Target analytes

SIGNAL 1 - FID	
Analyte	**CAS Number**
methane	74-82-8
ethene	74-85-1
acetylene	74-86-2
ethane	74-84-0
propane	74-98-6
SIGNAL 2 - TCD	
carbon dioxide	124-38-9

Table 2
QA Objectives for SOP RSK01

PARAMETER	ACCURACY (% REC)	PRECISION (% RPD)	MDL (ug/L)	REPORTING LIMIT (ug/L)	LCS, MS/MSD, TRUE VALUE (ug/L)
methane	56 – 140	0 – 40	1.0	5.0	114
ethene	56 – 140	0 – 40	1.0	5.0	200
acetylene	60 – 140	0 – 40	2.5	5.0	185
ethane	56 – 137	0 – 40	1.0	5.0	214
propane	60 - 140	0 – 40	2.5	5.0	314
carbon dioxide	10 – 200	0 – 40	2500	10,000	31,300

Table 3
Parameters

GC PARAMETERS*	
Injection port	200° C
TCD	200° C
FID	200 °C
Oven program	30° C, hold 2 min., 30° C/min. to 110° C hold 0.5 min.
AUTO-SAMPLER PARAMETERS*	
Constant heat time	OFF
Standby flow rate	50 mL/min.
Platen temperature equilibration time	0.50 min.
Sample equilibration time	1.00 min.
Mixer	OFF
Pressurize	8 PSIG
Pressurize time	2.00 min.
Pressurize equilibration time	0.20 min.
Loop fill pressure	5 PSIG
Loop fill time	2.00 min.
Inject time	1.00 min.
Valve oven temperature	100° C
Transfer line temperature	125° C
Platen/sample temperature	40° C
ARCHON HEAD SPACE PREPARATION PARAMETERS*	
Sample volume	13 mL
Rinses	0
Standards	0
Syringe flushes	1
Operation mode	LOCAL

* Recommended parameters; may be adjusted to improve performance.

Table 4
Quality Control Criteria
Dissolved Gases - RSK01

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration (ICAL)	Initially and upon failure of two consecutive CCV's	RSD ≤ 20%, $r \geq 0.995$ or $r^2 \geq 0.99$	Evaluate cause; repeat calibration; or qualify data and address in narrative (1)
Initial Calibration Verification (ICV)	After each initial calibration	% Drift ± 15%	Re-analyze ICV; upon second failure, repeat initial calibration (1)
Continuing calibration verification (CCV)	Prior to sequence, every 10 samples, at end of sequence	% Drift ± 15%	Re-analyze CCV; upon second failure, repeat initial calibration (1)
Method Blank (MB)	One per matrix/batch; maximum of 20 samples per batch	Target analytes ≤ MDL (methane and carbon dioxide < RL)	Notify supervisor and initiate NCR; investigate; repeat samples or qualify data and address in narrative (1)
Laboratory Control Samples/ 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: repeat samples or qualify data and address in narrative (1)
Matrix Spikes/Matrix Spike Duplicate (MS/MSD)	One per matrix/batch; maximum of 20 samples per batch	Target compounds within the designated range	Qualify data and/or address in narrative

(1) Evaluation criteria are often project specific. Check the project QAPP.
(2) Data will be qualified if sample volume is insufficient for re-extraction/re-analysis.

Figure 1

Checklist ID: 48358

Microbac Laboratories Inc.

Data Checklist

Date: _____
Analyst: _____
Analyst: NA
Method: RSK175
Instrument: HP16
Curve Workgroup: NA
Runlog ID: _____
Analytical Workgroups: _____

System Performance Check	
Initial Calibration	
Average RF	
Linear Reg or Higher Order Curve	
Second Source standard % Difference	
Continuing Calibration /Check Standards	
Project/Client Specific Requirements	
Blanks	
TCL's	
LCS (Laboratory Control Sample)	
Recoveries	
MS/MSD/Duplicates	
Samples	
TCL Hits	
Calculations & Correct Factors	
Dilutions Run	
Reruns	
Manual Integrations	
Narrative Summary	
Results Reporting/Data Qualifiers	
Client Data Package Assembly	
Check for Completeness	
Primary Reviewer	
Secondary Reviewer	
Check for compliance with method and project specific requirements	
Check the completeness of reported information	
Check the information for the report narrative	
Check the reasonableness of the results	

Primary Reviewer: Secondary Reviewer:

CHECKLIST1 - Modified 03/05/2008
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Figure 2
Example Calculations

RSK-175 - Example Calculation for Carbon Dioxide

ICAL Plot - Quadratic Regression (y = Ax^2 + Bx +C)

Ax^2 + Bx + (C - y) = 0

Step 1 - Calculate the concentration in extract, x:

Data from quadratic regression plot:		
Value of A from plot:	**0.916**	
Value of B from plot:	**1540**	
Value of C from plot:	**0**	
Response for carbon dioxide from quantitation report (y):	**8763828**	
Value of C - y	**-8763828**	
Solving for x using the quadratic formula:		
Root 1 - Computed x1:	**2364.716284**	umol/mol
Root 2 - Computed x2:	**-4045.938991**	

Step 2 - Calculate the concentration in sample, Cs:

Cs = x (MW/Tf) (HS/S) (DF)

Where:

x = Concentration in extract :	**2364.716284**	umol/mol
MW = molecular weight of analyte:	**44**	ug/umol
TF = temperature factor = (22.45)(313/273):	**25.68**	L/mol
HS = initial headspace volume (extraction log):	**0.015**	L
S = final volume (extraction log):	**0.00547**	L
DF = dilution factor:	**1**	
Cs = calculated sample concentration:	**11110.6798**	ug/L

Note: Temperature = 40 C = 313 K

MICROBAC

STANDARD OPERATING PROCEDURE
PERKIN ELMER OPTIMA 4300
INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROSCOPY SW-846 6010/EPA 200.7

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Microbac Laboratories, Inc.
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Approved By:

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Wade T. DeLong — Wade T. DeLong, Quality Assurance Officer — 4-15-14 — Date

Leslie S. Bucina — Leslie S. Bucina, Laboratory Manager — 4/15/14 — Date

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1.0 SCOPE AND APPLICATION

1.1 This standard operating procedure covers the operation of a Perkin Elmer Optima 4300 Inductively Coupled Argon Plasma spectrometer according to methods 6010B, 6010C, and 200.7 for the analysis of metals in digested soils, sludges, wastes, extracts and waters. Filtered waters preserved in acid may also be analyzed by this method. Samples originating from the states of North and South Carolina will be analyzed by Method 6010C in lieu of Method 6010B.

1.2 For elements for which this method is applicable see Table 1.1.

Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices. The data shown in Section 4.0 provides concentration ranges for clean aqueous samples. Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

1.3 Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods (see Microbac SOPs ME401A, ME406 and ME407). The instrument is prepared for operation and stabilized through a warmup period. Once stabilized, the instrument is calibrated and initial quality control elements are analyzed. When initial quality control requirements have been met, sample analysis begins. Samples are analyzed for metals content by optical emission spectroscopy. The Perkin Elmer Optima 4300 optical design combines an eschelle polychromator with a solid state detector in an integrated system optimized for ICP-OES. The polychromator allows for simultaneous multi-element analysis. The Perkin Elmer Optima 4300 contains a custom, two dimensional SCD array detector and RF generator. Perkin Elmer software is completely Windows based and extremely easy to use. A basic understanding of windows operation enables the analyst to move throughout the software.

1.4 Background correction points are selected according to compared background scans of standards, blanks, and samples.

1.5 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
COC	Coefficient of Correlation
DI water	Deionized Water
HCl	Hydrochloric Acid

HNO_3	Nitric Acid
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICS	interference Check Sample
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
IECs	Interelement Corrections
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LLCCV	Low Level Continuing Calibration Verification
LLICV	Low Level Initial Calibration Verification
LOD	Limit of Detection
LOQ	Limit of Quantitation
MB	Method Blank
MDL	Method Detection Limit
MS	Matrix Spike
MSA	Method of Standard Additions
MSD	Matrix Spike Duplicate
NCR	Nonconformance Report
OES	Optical Emission Spectroscopy
QC	Quality Control
RGT	Reagent
RL	Reporting Limit
SCD	Segmented-array Charge-coupled Device
SOP	Standard Operating Procedure
STD	Standard

Calibration blank – a calibration standard prepared with DI water (7.8) used in establishing the calibration curve.

1.6 For a comprehensive list of common terms and definitions, consult Appendix A in SOP LQAP.

2.0 SAFETY PRECAUTIONS

2.1 The Optima 4300 is equipped with safety interlocks to protect the analyst and instrument from harm if something out of the ordinary happens. The system checks water flow, argon pressures, sample compartment door interlocks, and plasma stability. These interlocks are constantly monitored and displayed on the screen. If any interlock is interrupted, the plasma is automatically shut down. Never attempt to defeat any interlocks.

2.1.1 The following interlocks must be satisfied in order to ignite the plasma:

- Front and side doors on the sample compartment must be closed
- Argon pressures for the torch must be correct
- Cooling water must be flowing to the RF coil

2.1.2 The following interlocks must not be interrupted while the system is operating:

- Purge gas for the detectors and the spectrometer optics must be functioning properly.
- The temperature of the optics housing must be 36-40° C
- The temperature of the detector must be less than -30° C

2.2 **WARNING:** Due to the high voltages and temperature, caution must be used in maintenance and troubleshooting.

2.3 A red Emergency Plasma Off switch located on the left front of the instrument allows the analyst to shut off the plasma in an emergency.

2.4 **WARNING:** Use gloves, safety glasses, lab coats and/or other appropriate safety precautions when handling samples and reagents.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1

Measurement	Digestion Vol./Wt.Req*	Collection Vol./Wt.	Preservative	Holding Time**
Total recoverable	50 mL	250 – 1000 mL p	HNO3 to pH <2	6 months
Dissolved	50 mL	250 – 1000 mL p	HNO3 to pH <2 Filter on-site	6 months
Suspended	50 mL	250 – 1000 mL p	Filter on-site	6 months
Total	50 mL	250 – 1000 mL p	HNO3 to pH <2	6 months
Soil	1 g	200 g glass	≤6° C	6 months

p = Plastic

* If insufficient sample volume is received a smaller volume of sample will be used and the reagents ratio will be reduced accordingly except for soils.

** Storage time allowed between sample collection and analysis when properly preserved and stored.

3.2 Water samples received unpreserved will be acid preserved in the laboratory and must rest for at least 24 hours prior to digestion.

4.0 METHOD PERFORMANCE

Instrument Detection Limits (IDLs)

4.1 IDLs are calculated by multiplying the standard deviation obtained from analysis of a reagent blank solution with seven consecutive measurements by the one-sided 98% confidence level t-statistic (3.14 is the t-statistic for seven samples). Each measurement is performed as though it were a separate sample (i.e, with rinsing in between).

IDLs must be determined quarterly. The IDL is required to be numerically less than the associated MDL.

Method Detection Limits (MDLs)

4.2 The laboratory performed an initial assessment of the MDL using the procedures outlined in 40 CFR Part 136. Results are filed electronically at H:\DATA\COMMON\MDL.

4.3 The LOD, or verified MDL, are presented in Table 4-1. These limits were established using verification procedures outlined in Microbac SOP 45.

4.4 The LOQ are the nominal laboratory RLs and were established per Microbac SOP 45. Actual project reporting limits may be higher.

4.5 Precision and accuracy data in Tables 4.2a and 4.2b were derived from an initial demonstration of capability using spiked control samples. The laboratory uses results from LCS to assess precision/accuracy and to annually evaluate the associated control limits.

4.6 MDLs must be re-determined whenever there is any change to the sample preparation procedure, or any significant change to the instrument

Linear Calibration Ranges

4.7 Calibrate the instrument, as described in Section 10.0.

4.8 Run a series of increasing concentration standards close to the upper linear range of the instrument. It is suggested that multi-element standards be used for the procedure whenever possible.

4.9 The linear range is defined as the highest concentration where the measured value is within 10% of the actual prepared value of the standard. The values reported in Table 4-1 are 90% of the verified upper linear range.

4.10 Linear Dynamic Range verifications are analyzed quarterly and edited in KOBRA and in the instrument software.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 IEC's (InterElement Corrections) are minimized by the use of the eschelle grating. However this grating does not eliminate all interferences due to spectral overlap. Table 5-1 lists the approximate IEC's necessary of analyte per unit (mg/L) of interferant for the Optima 4300. These IEC's are subject to change with modifications made to operating conditions, such as changes in coolant flow, power, nebulizer or even new torches.

5.2 The use of a peristaltic pump reduces physical interferences. However, samples with high dissolved solids, high acid concentration or high viscosity may need to be diluted.

6.0 EQUIPMENT AND SUPPLIES

6.1 Perkin Elmer Optima 4300 equipped with a CETAC ASXpress-520 Autosampler.

6.2 Argon gas supply (liquid).

6.3 Dell Pentium 4 computer with Microsoft Windows 2000 Professional and Perkin Elmer WinLab32 ICP Continuous Software Version 4.0.0.0303.

6.4 ESI Microflow PFA-ST3-84 Nebulizer

6.5 Peristaltic pump tubing

6.5.1 Black/Black – 0.32 mL/min (Sample and flush station).

6.5.2 Yellow/Orange – 0.51 mm I.D (Internal Standard)

6.5.3 Red/Red – 1.14 mm I.D. (drain)

6.6 Mixing block

6.7 Calibrated mechanical pipettes:

6.7.1 10 -100 uL

6.7.2 100-1000 uL

6.7.3 1000-5000 uL

6.8 Metal-free plastic pipette tips (for the pipettes) specified in 6.7

6.9 Metal-free 15 mL plastic test tubes

6.10 Metal-free 50 mL plastic test tubes

6.11 Class A glass pipettes – for preparation of standard solutions

7.0 STANDARDS AND REAGENTS

7.1 Acids used in the preparation of standards and for sample processing must be reagent grade or better. Redistilled acids may be used.

All purchased stock standards and reagents are logged into the LIMS System and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.2 Concentrated hydrochloric acid (HCl). Baker Instra Analyzed Grade or better.

7.3 Concentrated nitric acid (HNO_3). Baker Instra Analyzed grade or better.

7.4 DI water – ASTM Type II or equivalent (ASTMD 1193)

Calibration Solutions

7.5 Mixed calibration stock standards are purchased from Inorganic Ventures as KEM-CONC-1,2,3 standards set. From this the High Standard and CCV are made for two different matrices.

7.6 Working Calibration Solutions – For the High Standard water matrix, dilute 5 mL from each bottle of stock into 500 mL of 5% HCl and 2% HNO_3 in DI water. For the soil matrix, dilute 5 mL from each bottle of stock into 500 mL 5% HCl and 5% HNO_3 in DI water. From the High Standard the following dilutions are made with the appropriate matrix matched acid water:

For Optima 4300:

S1: 250X dilution of high standard
S2: 125X dilution of high standard
S3: 2X dilution of high standard
S4: High Standard

Standard S0 is a matrix matched blank (7.8). Concentrations of each element in the calibration standards are given in Table 7.4.1 for Optima 4300.

7.7 Working CCV Solutions – The CCV is prepared for 2 different matrices. For the water matrix dilute 5 mL from each bottle of calibration stock into 1000 mL of 5% HCl and 2% HNO_3 in DI water. For the soil matrix, dilute 5mL from each bottle of calibration stock into 1000 mL 5% HCl and 5% HNO_3 in DI water. Concentrations for each element are listed in Table 7.5.1.

7.8 Working Calibration Blank Solution - A calibration blank of 5% HNO_3 , 5% HCl for soil and 2% HNO_3, 5% HCl for water is used to establish the analytical curve and is analyzed following each initial and continuing calibration standard analysis.

Initial Calibration Verification Solutions

7.9 The ICV stock solution is purchased from SPEX as XKES-5 and SCP Science as Plasmacal1 and 3. The ICV is at the same level as the midpoint standard used in the calibration and is a separately prepared, quality control analyzed and certified source from that of the calibration standards.

7.10 1000 ug/mL silicon – Inorganic Ventures

7.11 The ICV is prepared for 2 different matrices. For the water matrix dilute 1 mL from each bottle of ICV stock into 200 mL of 5% HCl and 2% HNO_3 in DI water. For the soil matrix, dilute 1 mL from each bottle of ICV stock into 200 mL 5% HCl and 5% HNO_3 in DI water. Concentrations for each element are listed in Table 7.5.1.

Interference Check Sample Solutions

7.12 ICS Stock – An ICS A (ICSA) stock solution is purchased from Inorganic Ventures as CLPP-ICS-A.

ICS AB (ICSAB) standards are purchased from Inorganic Ventures as CLPP-ICS-A, CLPP-ICS-B, and KEM-ICS-B-1A.

7.13 Working ICS Solutions – The ICSA is prepared for 2 different matrices. For the water matrix dilute 10 mL of the ICSA stock solution into 200 mL 5% HCl and 2%

HNO_3 in DI water. For the soil matrix dilute 10 mL of the ICSA stock solution into 200 mL 5% HCl and 5% HNO_3 in DI water. The ICSA solution will contain only aluminum, calcium, iron and magnesium in high concentrations (see Table 7.7.1).

The ICSAB is made for 2 different matrices. For the water matrix dilute 10 mL of CLPP-ICS-A, 1 mL of CLPP-ICS-B and 1 mL of KEM-ICS-B-a into 200 mL 5% HCl and 2% HNO_3 in DI water. For the soil matrix dilute same volumes of the ICSAB into 200 mL 5% HCl and 5% HNO_3 in DI water. The ICSAB will have the same concentrations for aluminum, calcium, iron and magnesium as the ICSA with additional metals spiked at detectable levels (see Table 7.7.1).

Internal Standard Solutions

7.14 10,000 ug/mL Yttrium – Inorganic Ventures

7.15 10,000 ug/mL Gallium – Inorganic Ventures

7.16 Working Internal Standard Solution – a 5 mg/L Y and 25 mg/L Ga internal standard solution for Optima 4300, prepared by diluting 5 mL gallium and 1 mL yttrium into 2000 mL of the appropriate matrix matched acid water.

Low Level Calibration Verification Solutions

7.17 The low level ICV and low level CCV (LLICV and LLCCV) are analyzed for 6010C only. The LLICV and LLCCV are analyzed using the same solution with concentrations at or below the RL.

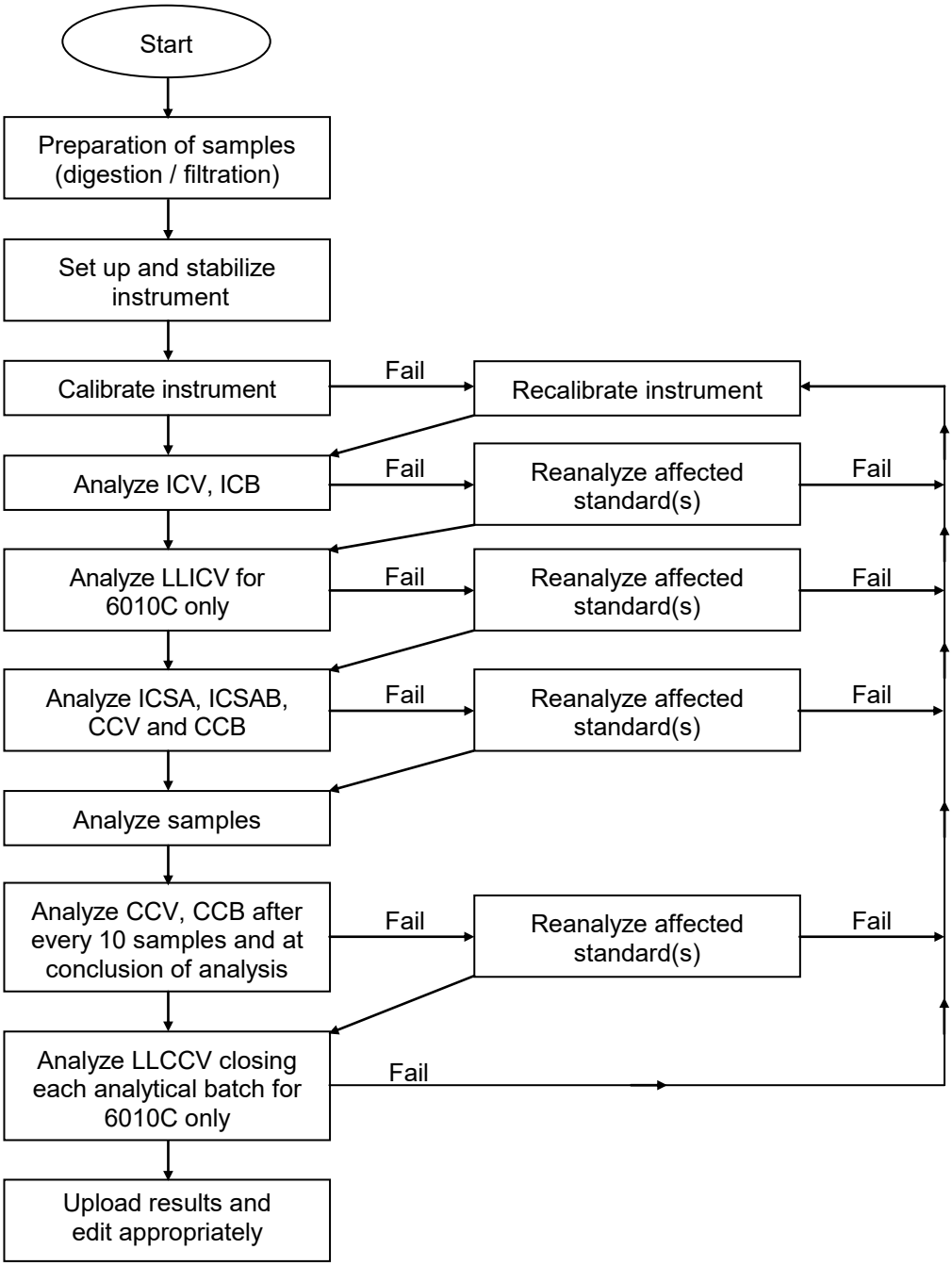
7.18 The LLICV/LLCCV solution is custom prepared from single element standards (Inorganic Ventures or equivalent) or by appropriate dilution of the CCV solution (7.7) into 5% HCl and 2% HNO_3 for the water matrix and 5% HCl and 5% HNO_3 for the soil matrix.

7.19 Concentrations of each target metal must be at or below the respective RL for the metal.

7.20 4 % HCl Solution – To approximately 500 mL of DI water add 40 mL conc. HCl. Dilute to 1000 mL final volume with DI water.

7.21 Custom Multielement Solution MIC-SPK-1A from Inorganic Ventures or equivalent.

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



9.0 SAMPLE PREPARATION

9.1 Sample preparation is dependent on matrix and digestion type. Refer to the following methods:

ME401A – Microwave Digestion – Dissolved Aqueous, SW-846 Method 3015/EPA 200.7

ME406 – Microwave Digestion of Sediments/Sludges/Soils/Oils, SW-846 Method 3051

ME407 – Microwave Digestion – Aqueous, SW-846 Method 3015

10.0 CALIBRATION PROCEDURES

10.1 Initial Calibration – The instrument is calibrated before analysis of any samples with a blank and four calibration standards or alternatively with a blank and the high standard, S4 from 7.6 (6010C only). Calibration Standards are prepared from Inorganic Ventures Stock. The dilutions of the calibration standards are listed in Section 7.6. The concentrations are listed in Table 7.4.1.

For the multipoint calibration, the instrument performs a linear or a weighted linear regression for all elements except potassium and sodium which have a non-linear curve fit. For the single point calibration the instrument performs a linear regression with a calculated intercept for all analyzed elements. (See Appendix A for calibration algorithms.) The correlation coefficients are printed out when calibration is complete. The first standard run must be the calibration blank, followed by standards of increasing concentration in order to minimize cross-contamination and carryover. The prepared calibration standards are analyzed in three replicates with the reported results being the arithmetic mean (average) of the three replicate readings.

When performing the multipoint calibration the low calibration standard must contain the elements of interest at concentrations at or below the reporting limit or a low level calibration check standard at or below the reporting limit must be analyzed after calibration and before sample analysis. The LLICV must always be analyzed for 6010C. See Tables 13.1, 13.2 and 13.3 for acceptance criteria and corrective action for the curve and low level calibration verification standard.

10.2 ICV Analysis – ICV analysis must be performed immediately after calibration standards to verify calibration. See Tables 13.1, 13.2 and 13.3 for acceptance criteria and corrective action.

10.3 CCV Analysis – The CCV is required to be run after every 10 samples, at the end of the analysis and prior to sample analysis. See Tables 13.1, 13.2 and 13.3 for acceptance criteria and corrective action.

10.4 ICB and CCB Analysis – The solution used is the calibration blank (7.8). See Tables 13.1, 13.2 and 13.3 for acceptance criteria and corrective action.

10.5 ICSA and ICSAB Analysis – Required at the beginning of analytical run. See Tables 13.1, 13.2 and 13.3 for acceptance criteria and corrective action.

10.6 Low Level Calibration Verification (LLICV and LLCCV) Analysis – For 6010C only is required at the beginning of the run (LLICV) and at a minimum after every analytical batch (LLCCV). See Table 13.3 for acceptance criteria and corrective action.

10.7 Calibration training materials are available on the intranet home page in the "General" links section, "Calibration Training". Review of "Calibration Models" and "The Effect that Saturation of the Detector has Upon Calibration" are recommended training for all new analysts. There are additional calibration training materials available through the same link on the homepage.

11.0 ANALYTICAL PROCEDURES

11.1 Preliminary treatment of all matrices is always necessary because of the complexity and variability of sample matrices.

11.2 Startup procedures

11.2.1 Pump tubing on instrument must be pliable and have only slight discoloration. If tubing is worn, replace.

11.2.2 Torch must be clean and dry.

11.2.3 The instrument's main power source, RF generator, water recirculator and vent must be on at all times unless there is an emergency situation or for maintenance purposes.

11.2.4 Turn on the computer and login to the system. Click on WinLab 32. While the instrument goes through its systems check, attach all tubing to the pump and engage tension arms. Double click on Xpress Config 3.0 located on the desk top to open up the autosampler software.

Click on connect to ASXpress and minimize the window.

11.2.5 Click on plasma icon at the top of the screen. This opens up the plasma control window. Click on box that says pump. This starts the sample, rinse and internal standard flows.

11.2.6 To ignite plasma, click on the "on" button in the plasma control window, close window.

11.3 Set up the instrument with the proper operating conditions. These conditions are found under Plasma control.

- Power 1300 – 1450 W
- Plasma 15 -17 L/min
- Nebulizer 0.45 – 0.6 L/min
- Auxiliary 0.2 – 0.8 L/min
- Pump rate 2.0 - 2.5 mL/min for samples 4.0 mL/min rinse
- Water circulation on

11.4 The ICP must be allowed to become thermally stable before beginning analysis. (This usually requires approximately 30 minutes of operation prior to calibration.)

11.5 Pumps must be on before beginning calibration.

11.6 Set up of analytical sequence

11.6.1 After igniting plasma click on file (top left of screen) open, sample info File, click on Default .SIF, open. To call up method click on Method (top right of screen) click on method desired for current analysis, ok. Click on Auto, Open Results Data Set Name, change date, click on ok.

11.6.2 To enter the analytical sequence click on SamInfo. Enter all sample labels.

11.6.3 Enter 2,3,4,5,6,7 into the "Analyze QCs Before" Column and 6,7 in the same column after every 10 samples. This tells the instrument to read check standards at the beginning and check standards after every 10 samples.

11.6.4 To have the instrument calculate percent difference and percent recoveries, click on "Matrix Check Samples" column. Click on Duplicate for a duplicate sample, Recovery Set Number for LCS, MS/MSD and post digestion spike. Click on Remarks and enter 5 for the dilution factor for a serial dilution. Reference the

correct sample for calculation of recoveries and RPD. Enter dilution factor in the remarks column.

11.6.5 To save all run information, click on file (top left of screen), Save As, Sample info File, type in run date and click on Save.

11.7 Calibration

11.7.1 Standards are prepared from Inorganic Ventures stock. The concentrations are listed in Tables 7.4.1.

11.7.2 For the Optima 4300 model, the prepared blank and standards are placed in positions one through five in the standard rack (larger tubes). The ICV solution is located in position 11. The ICSA and ICSAB are in positions 12 and 13 respectively. The CCV is placed in position six following calibration.

11.7.3 Prior to calibration you must align the Hg lamp. To do this click on Tools, Spectrometer Control, Hg Realign. This is done axially and radially. Record the results for entry into the maintenance log. You must also perform the Background Equivalent Concentration test (BEC).

To run the BEC Test:

1) Warm-up the instrument for at least 5 minutes
2) Open method **MnBEC**
3) Open the workspace by clicking on: File – Open – Workspace – MnBEC
   This will bring up the Manual Analysis Control Window, Results Window and Spectrometer Control Window.
4) In the Manual Analysis Control Window, enter a **Results Data Set Name** (e.g. date + BEC)
5) Click on ASXpress Configuration to open aurosampler window.
6) Switch the valve to load position.
7) Connect the probe to the peripump channel leading to the rinse port on the valve.
8) Put probe into a cup of deionized water.
9) Disable ASXpress operation through xpress configuration by unclicking the top check box.
10) Click save configuration to ASXpress.
11) Aspirate the blank, click on **Analyze Blank** button
12) Aspirate the 1 mg/L Mn standard, click on **Analyze Standard** button RSD <1%
13) Aspirate rinse solution
14) In the Spectrometer Control Window select **Shutter: Closed**

15) In the Manual Analysis Control Window type in **BEC** next to ID, click on the **Analyze Sample** button
16) The absolute value of the result is the **BEC**
17) Compare **BEC** value to the acceptable value of < 0.04 mg/L
18) In the Spectrometer Control Window select **Shutter: Auto**
19) Record BEC value for entry-in the maintenance log and close the windows.
20) Enable ASXpress operation through xpress configuration by unclicking the top check box.
21) Click save configuration to ASXpress.

11.7.4 To initiate calibration, click on Auto, Analyze and Analyze All.

11.7.5 The prepared calibration standards ICV, CCV, ICB/CCB. ICSA/ICSAB and LLICV/LLCCV (if running 6010C) are analyzed in three replicates with the reported results being the arithmetic mean (average) of the three replicate readings.

11.7.6 For a multipoint calibration, the instrument software performs a linear or a weighted linear regression with a calculated intercept for all analyzed metals except potassium and sodium which exhibit a nonlinear response. The software performs a nonlinear regression with a calculated intercept for potassium and sodium. The instrument software performs a linear regression with a calculated intercept for single point calibration. (See Appendix A for the calibration algorithms). Upon completion of the calibration, check printout to verify that all correlation coefficients are 0.995 (0.998 for 6010C) or better before continuing.

11.8 Upon completion of the calibration begin following the analytical sequence as described below. For acceptance criteria and corrective actions, see Tables 13.1, 13.2 and 13.3:

Calibration
ICV
ICB
LLICV (6010C)
ICSA
ICSAB
CCV
CCB
10 or less samples
CCV
CCB
Continue with the last three (3) steps until the end of run,
always ending with a CCV/CCB and LLCCV (6010C).

11.8.1 The sample results are an arithmetic mean (average) of three replicate readings per analyte. For any analyte with a result greater than the reporting detection limit, the % RSD between the replicate readings must be less than ten.

11.9 Flush the system with the rinse solution for at least one (1) minute before the analysis of each sample. Analyze the CCV and CCB after each 10 samples at a minimum, and at the end of each analytical run.

11.10 Calculations: If dilutions were performed, the appropriate factors must be applied to sample values.

11.11 Dilute and reanalyze samples that are more concentrated than the linear dynamic range.

11.12 After analysis, let the instrument rinse for fifteen minutes then extinguish plasma and release pump tension. For the Optima 4300 model, press F11 to raise probe.

11.13 Exit software and turn off computer.

12.0 DETAILS OF CALCULATIONS

12.1 All calculations for samples and standards are computed from the mean of three exposures. Each metal has a specified linear range. Refer to Table 4-1 for upper limits.

12.2 After the multipoint calibration is complete, the software performs a linear or a weighted linear regression with a calculated intercept for all analyzed metals except potassium and sodium which exhibit nonlinear responses.

The software performs a nonlinear regression with a calculated intercept for potassium and sodium. The instrument calculates the correlation coefficients for each metal and the analyst can view each curve for acceptance.

Following a single point calibration, the software performs a linear regression with a calculated intercept. Calculated correlation coefficients in this case are all equal to 1.0. (See Appendix A for the calibration algorithm). All sample results are calculated from the calibration curve.

12.3 Dilution factors and preparation factors are calculated into the final result which is computed from the mean of three exposures.

12.3.1 For Liquid Samples:

$$mg/L\ metal\ in\ sample = mg/L\ in\ digestate * \frac{Final\ Prepared\ Volume(mL)}{Inital\ Volume(mL)} * \frac{Total\ Diluted\ Volume}{Sample\ aliquot}$$

12.3.2 For Solid Samples:

$$mg/Kg\ metal\ in\ sample = mg/L\ in\ digestate * \frac{Final\ Prepared\ Volume(mL)}{Inital\ Volume(g)} * \frac{Total\ Diluted\ Volume}{Sample\ aliquot}$$

12.3.3 LCS/LCS Duplicate % Recovery

$$\%\ recovery = \frac{C_s}{C_t} * 100\%$$

where:

C_s = the LCS sample result
C_t = the LCS true value

12.3.4 MS/MSD % Recovery

$$\%\ recovery = \frac{(C_s - C_a)}{C_t} * 100\%$$

where:

C_s = the MS/MSD sample result
C_a = the reference sample result
C_t = the MS/MSD true value

12.3.5 Post Digestion Spike Recovery

$$\%\ recovery = \frac{(C_s - BC_a)}{C_t} * 100\%$$

where:

C_s = the spike sample result
C_a = the reference sample result
B = a factor to account for the dilution of the spiked sample relative to the reference sample (usually B = 0.9)
C_t = the spike true value

12.3.6 Duplicate RPD

$$RPD = \frac{|C_a - C_b|}{(C_a + C_b)/2} * 100\%$$

where:

C_a = the reference sample result
C_b = the duplicate sample result

12.3.7 Serial Dilution % Difference

$$\%D = \frac{|C_a - 5C_b|}{C_a} * 100\%$$

where:

C_a = the reference sample result
C_b = the diluted sample result

12.4 Calcium and magnesium results can be used to calculate water hardness results. The LIMS will use the uploaded calcium and magnesium results to calculate the hardness results. The automated hardness calculation is manually verified annually.

$$Hardness(mg/L) = 2.497[Ca(mg/L)] + 4.118[Mg(mg/L)]$$

12.5 Silicon results can be used to calculate silica results.

The calculation assumes all silicon recovered from 6010 is in the form Si02. KOBRA converts the results as follows:

$$silica(mg/L) = silicon(mg/L) * 2.13923$$

12.6 See Figure 12.1 for a sample calculation summary

13.0 QUALITY CONTROL REQUIREMENTS

Overview

13.1 Refer to Microbac SOP 45 for Details of Demonstration of Capability. (Method Validation Procedures).

13.2 Each preparation batch (or workgroup) consists of a maximum of twenty (20) samples plus QC samples (see Section 13.3 for list of required QC). The QC samples are prepared and digested identically to the analytical samples.

13.3 The following QC are digested with every preparation batch. The frequency, acceptance criteria and corrective action for this QC is listed in Tables 13.1, 13.2 and 13.3.

Batch Quality Control

13.3.1 Method blank (MB) an aliquot of DI water that is digested with the sample batch and contains all reagents identical with the samples.

13.3.2 LCS - for water matrix, a spiked DI water that is digested with the sample batch. The LCS is prepared by diluting 5 mL of KEM-SPK-1F (see Table 13.3.2a for initial concentrations) and 0.025 mL each of single element tin, phosphorus and zirconium into a 50 mL volume of DI water.

For soil matrix; 1.0 g of PTFE Boiling Stones (Teflon) Chemware is spiked with 5 mL of KEM-SPK-1F and 0.025 mL each of single element tin, phosphorus and zirconium prior to digestion.

A LCSD may also be analyzed with the batch and is prepared identically to the LCS.

(**NOTE:** Both the LCS and LCSD must pass acceptance criteria or the samples will have to be re-digested/re-analyzed for the analyte in question.)

The final concentration of the aqueous and soil LCS are given in Table 13.3.2b:

13.3.3 Sample duplicate - a sample prepared in duplicate, both carried through the digestion procedure. (By client request only)

13.3.4 MS and MSD - a sample that is spiked in duplicate and then digested with the sample batch. It is prepared by taking 3 aliquots of sample, 2 of which are spiked with 5 mL of KEM-SPK-1F and 0.025 mL each of single element tin, phosphorus and zirconium for each 50 mL of sample. The final concentration spiked into the two spiked samples will be the same as the aqueous and soil LCS level in Table 13.3.2.

Interference Tests – Post Digestion

13.4 With every analytical batch a post digestion spike and a serial dilution must be analyzed. If the matrix spike fails the 75 - 125% acceptance criteria, one of the following interference tests must pass for the analyte outlier:

13.4.1 Serial dilution: If the analyte concentration is sufficiently high (at least a factor of 50 above the method detection limit before dilution), analysis of a 1:5 dilution should agree within ± 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

13.4.2 Post Digestion Spike: an analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75 to 125% (80-120% for 6010C) of the known value. The spike addition should produce a minimum level of 10 times the instrument detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

13.4.3 Method of standard additions: This method must be employed for certain TCLP extracts and can also be used to compensate for sample constituents which enhance or depress the analyte signal producing a slope different from that of the calibration standards. It will not correct for additive interferences which cause a base line shift.

The simplest version of these techniques is the single-addition method, in which two identical aliquots of the sample, each of volume Vx are taken. To the first (A) a small volume (V_s) of a standard analyte concentration (C_s) is added. To the second (B) a volume (V_s) of the solvent is added. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration (C_x) is calculated:

$$C_x = (S_b)(V_s)(C_s)/(S_a - S_b)(V_x)$$

where:

S_a and S_b are the analytical signals of A and B respectively. V_s and C_s should be chosen so that S_a is roughly twice S_b on the average. It is best if V_s is made much less than V_x, and thus C_s is much greater than C_x, to avoid excess dilution of the sample matrix. Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected concentration from the endogenous analyte in the sample.

Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample concentration. The concentration of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero concentration, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

13.5 Linear Range Analysis/High-Level Standard

A linear range analysis is performed quarterly to determine the highest concentrations for each analyte at which the instrument yields a result within 10% of the true concentration. When use of the Department of Defense Version 3 QAP is indicated, a high-level check standard with analyte concentrations at the linear range of the instrument may be analyzed subsequent to sample analysis for any analyte which exceeds the calibration range. The recovery must be within 10% of the true concentration.

13.6 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.6.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Tables 13.1, 13.2 and 13.3, does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Tables 13.1, 13.2 and 13.3.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch (Figure 14.1). A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval

- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.6.2 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.

13.6.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that may be invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate an NCR to record the event.

- An ICV, CCV or LCS recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank, ICB or CCB exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank, ICB or CCB exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

13.7 All data is scrutinized by the analysts for method and project specific compliance. Check lists are utilized and accompany each data batch (Figure 14.1).

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Data Review

Data is archived from the instrument computer to the LIMS where it is stored in a CSV format. When analysis is complete the analyst must upload the relevant CSV files including calibration, check standards, QA/QC samples and client

samples into KOBRA. This is done via MICROBAC's customized upload program.

When the upload is complete, the analyst must check the sample data for correct digestion factors, dilutions and RLs. Any elements that are not to be reported must be checked as excluded. This will be determined by the primary analyst through real time review of all quality control elements as summarized in Tables 13.1, 13.2 and 13.3. The analyst must certify that this primary review has been carried out by completing the Data Review Checklist (Figure 14.1), signing and dating. The analyst generates batch QA/QC summary forms automatically through an oracle program. The analyst must then assemble the QA/QC summaries, batch upload reports, digestion logs and runlogs, case narratives if required and KOBRA workgroup reports. The Data Review Checklist acts as a cover page and will be archived with the hardcopy data. The completed package is then submitted for secondary review.

The secondary review consists of an additional 100% review of the hardcopy data for QA/QC compliance. This review consists of a double check of the batch QA/QC summary and associated post spikes and serial dilutions. Sample results are reviewed for completeness, reasonableness and compliance with any special project or client requirements. The case narrative, if any, is also checked for accuracy and completeness. The supervisor (or designate) also signs and dates the Data Review Checklist.

When all levels of review have been completed. The elements being reported on each sample are taken to a done status in KOBRA.

15.0 PREVENTIVE MAINTENANCE

15.1 Check the torch and nebulizer every day and clean when needed depending on sample load.

15.2 Tubing needs to be changed when it loses pliability and is worn.

15.3 Drain compressor daily.

15.4 The instruments are under service contracts so that every year a service representative will perform a systems check.

15.5 The water in the recirculator/cooler must be changed yearly.

15.6 Troubleshooting procedures for the Perkin Elmer Optima 4300 can be found in Chapter 6 of the Optima 4000 Series Hardware Guide, Document Control ID #65, and in Chapter 10 of the WinLab32 Instrument Control Software Guide, Document Control ID #67.

15.7 Performance problems (such as loss of signal or poor precision) are often related to the sample introduction system.

15.7.1 Perform the sodium bullet test to monitor sample flow.

15.7.2 Check pump tubing for flat spots, leaks or discoloration.

15.7.3 Check pump rollers for grooves or binding.

15.7.4 Check pump tubing for crimps, pinching and clogging.

15.7.5 Check nebulizer and spray chamber for leakage and proper drainage.

15.7.6 Check nebulizer spray pattern. The spray must not be uneven or sputtering.

15.7.7 Check to torch and injection for deposits, deformation and security of fit. O-rings must not be cracked or worn.

15.7.8 Check that the purge window is not cloudy or dirty.

15.8 The instrument configuration for the OPTIMA 4300 can be found in the electronic maintenance log. The template maintenance log is found at number 13195 in KOBRA. Replacement of instrument components will be recorded in the relevant maintenance log and updated in the log header as needed.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33 – Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

16.2 Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are

responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2.1 The waste streams are as follows:

The metals laboratory waste is neutralized with sodium bicarbonate and flushed down the drain with tap water as per agreement with the Marietta Department of Water and Waste Water.

17.0 REFERENCES

17.1 Inductively Coupled Plasma – Atomic Emission Spectrometry, US EPA SW-846 Method 6010B, Revision 2, December 1996, EPA Publication SW-846

17.2 Us Environmental Protection Agency, EPA Method 200.7, Revision 4.4, 1994, 40CFR pt.136.

17.3 Inductively Coupled Plasma-Atomic Emission Spectrometry, US EPA SW-846 Method 6010C, Revision 3, February 2007, EPA publication SW-846.

17.4 Microbac SOP LQAP “Laboratory Quality Assurance Plan”

17.5 Microbac SOP 45 “Method Validation Procedures”

17.6 Microbac SOP ME406 “Microwave Digestion of Sediments, Sludges, Soils and Oils (3051)”.

17.7 Microbac SOP ME407 “Microwave Digestion – Aqueous SW846-3015”.

17.8 Microbac SOP 33 “Laboratory Waste Management”

17.9 Microbac SOP GP-CAPA “Corrective Action/Preventive Action: Initiating, Tracking and Monitoring”

17.10 Microbac SOP GP-RCA “Root Cause Analysis”

17.11 Microbac SOP ME401A “Preparation of Dissolved Metals in Waters for Direct Analysis by Inductively Coupled Plasma Spectroscopy (EPA 200.7)”

Appendix A

Linear Calibration Algorithm

This calibration curve is established by assuming that the relationship between concentration (the X values) and intensity (the Y values) is linear and that the following equation describes this relationship:

$$Y=MX+B$$

where:

X = concentration
Y = intensity
M = slope of the calibration curve
B = y-axis intercept

Given 2 or more data points, the values for M and B are calculated using the following equations [1, 2].

N = number of standards (includes the blank)

In this equation, the blank is subtracted from all solutions and included in the calculation of the calibration curve.

[1] $$M = \frac{(n)\sum_{n}^{i=1}(X_i Y_i) - \sum_{n}^{i=1}(X_i)\sum_{n}^{i=1}(Y_i)}{(n)\sum_{n}^{i=1}\left(X_i^{\,2}\right) - \left(\sum_{n}^{i-1} X_i\right)^2}$$

[2] $$B = \frac{\sum_{n}^{i=1}\left(X_i^{\,2}\right)\sum_{n}^{i=1}(Y_i) - \sum_{n}^{i=1}(X_i Y_i)\sum_{n}^{i=1}(X_i)}{(n)\sum_{n}^{i=1}\left(X_i^{\,2}\right) - \left(\sum_{n}^{i-1} X_i\right)^2}$$

Linear Calibration Algorithm (continued)

$$COC = \frac{\sum xy - \frac{\sum x \sum y}{n}}{\sqrt{\left(\sum x^2 - \frac{(\sum x)^2}{n}\right)\left(\sum y^2 - \frac{(\sum y)^2}{n}\right)}}$$

where:

x = standard concentration
y = mean intensity
n = number of standards

Weighted Linear Calibration Algorithm

When working at low level concentrations it provides an alternative calibration scheme that weighs the low standards to a greater degree.

Determine the slope, intercept, and correlation coefficient for the equation:

$$y = b_0 + b_i x$$

Where y is the measured net corrected intensity (blank subtracted). The weights are applied by multiplying the intensity by the weighting factor for each standard. In this calibration the weighting factor is the reciprocal of the square of the user entered concentration value for each standard.

where:

x = concentration value of the standard
y = measured intensity of the standard
n = number of standards
i = index for the standards
b_0 = intercept
b_1 = slope
COC = correlation coefficient

$$w_1 = \sum \frac{1}{x_i^2} \qquad w_4 = \sum \frac{y_i}{x_i^2}$$

$$w_2 = \sum \frac{x_i}{x_i^2} \qquad w_5 = \sum \frac{y_i^2}{x_i^2}$$

$$w_3 = \sum \frac{x_i^2}{x_i^2} = n \qquad w_6 = \sum \frac{x_i y_i}{x_i^2}$$

Intercept:

$$b_0 = \frac{(w_4 w_3) - (w_6 w_2)}{(w_1 w_3) - w_2^2}$$

Slope:

$$b_1 = \frac{(w_1 w_6) - (w_4 w_2)}{(w_1 w_3) - w_2^2}$$

Weighted Linear Calibration Algorithm (continued)

Correlation Coefficient: $$COC = \frac{(w_1 w_6) - (w_2 w_4)}{\sqrt{\left[(w_1 w_3) - w_2^{\,2}\right]\left[(w_1 w_5) - w_4^{\,2}\right]}}$$

Non-Linear Calibration Algorithm

This calibration curve is established by assuming that the relationship between concentration (the X values) and intensity (the Y values) is a curvilinear one that can be described by the following polynomial equation:

$$Y = AX^2 + BX + C$$

where:

X = concentration
Y = intensity
A = curvature
B = slope
C = y-axis intercept

Given 3 or more data points, the values for A, B and C are calculated using the following equations [1, 2, 3].

n = number of standards (includes the blank)

In this equation, the blank is subtracted from all solutions and included in the calculation of the calibration curve.

[1] $$A = \frac{\left[\frac{a_2}{a_0} - \frac{a_5}{a_3}\right]}{\left[\frac{a_1}{a_0} - \frac{a_4}{a_3}\right]}$$

[2] $$B = \frac{a_5}{a_3} - \frac{(A \times a_4)}{a_3}$$

[3] $$C = \frac{c_2}{n} - \frac{(B \times c_0)}{n} - \frac{(A \times c_1)}{n}$$

where:

Non-Linear Calibration Algorithm (continued)

$$a_0 = \frac{c_0}{n} - \frac{c_1}{c_0} \qquad c_0 = \sum_{n}^{i=1}(X_i)$$

$$a_1 = \frac{c_1}{n} - \frac{c_3}{c_0} \qquad c_1 = \sum_{n}^{i=1}(X_i^{\,2})$$

$$a_2 = \frac{c_2}{n} - \frac{c_4}{c_0} \qquad c_2 = \sum_{n}^{i=1}(Y_i)$$

$$a_3 = \frac{c_1}{c_0} - \frac{c_3}{c_1} \qquad c_3 = \sum_{n}^{i=1}(X_i^{\,3})$$

$$a_4 = \frac{c_3}{c_0} - \frac{c_5}{c_1} \qquad c_5 = \sum_{n}^{i=1}(X_i^{\,4})$$

$$a_5 = \frac{c_4}{c_0} - \frac{c_6}{c_1} \qquad c_6 = \sum_{n}^{i=1}(X_i^{\,2}Y_{ii})$$

Appendix B

Simultaneously Extracted Metals (SEM)

SEM are operationally defined as metals (arsenic, cadmium, copper, lead, mercury, nickel, silver and zinc for example) that form sulfides less soluble than those of iron or manganese and which are at least partially released from the sediment under the conditions for generating Acid Volatile Sulfides (AVS) (see Microbac SOP KAVS). Since sulfide is a precipitant of heavy metals under anoxic conditions, the SEM to AVS ratio obtained by the method is an indicator of heavy metal bioavailability in the sediment sample.

AVS Generation and Filtration

AVS is generated in a reaction cell per the procedure in Microbac SOP KAVS. Once sulfide generation is complete the sediment and water from the reaction cell (Microbac SOP KAVS Section 11.10) is vacuum filtered through Whatman TCLP Glass microfiber Filters Acid Treated Low Metal (or equivalent) into a filtration apparatus that has been washed, rinsed in 0.1 M HNO_3 and then DI water rinsed prior to use.

The filtrate is transferred to B-D 60 mL sterile luer-lok syringes equipped with Corning 0.2 um membrane syringe filters (or equivalent) and filtered into an acid washed, DI water rinsed graduated cylinder.

The filter flask is rinsed with DI water and the rinsates are similarly 0.2 um filtered and added to the cylinder.

DI water is then used to bring the filtrate to a final volume of 250 mL. The sample is transferred to a labeled, certified precleaned 250 mL Nalgene bottle C and G Containers (or equivalent) and analyzed within two weeks.

Metal concentrations are determined by ICP-OES.

Batch QA/QC Samples

Method Blank – An aliquot of 4% HCl in DI water is filtered in the same manner as the samples. This filtrate will be used as the method blank.

LCS – In a digestion tube containing about 20 mL 4% HCl spike 5 mL of spike solution (7.25) and bring to a 50 mL final volume with 4% HCl. Filter the LCS in the same manner as the samples.

MS/MSD – If there are no client designated spike samples, choose one sample from the batch. Into two digestion tubes pour approximately 20 mL of the filtered sample. Add 5 mL of spike solution (7.25) and bring to final 50 mL volume with the additional filtered sample.

Interference Tests – A post digestion spike will be prepared as in Section 13.8. Additionally, if appropriate, a serial dilution will be analyzed as in 13.7.

Calibration Solutions

Calibration Solutions, ICV and Interference Check Solutions are prepared as in Sections 7.2 through 7.13 and 7.17 through 7.18 except that the diluents used in 4% HCl (7.24).

The working calibration blank solution used is a 4% HCl (7.24).

Analysis and Reporting

Metal concentrations are determined by ICP-OES following the procedures in Section 11.0.

The instrument is calibrated as in 10.1 followed by ICV/ICB, ICSA/ICSAB and CCV/CCB analysis. Acceptance criteria are found in Table 13.1. Samples are analyzed in groups of ten or less followed by CCV/CCB analyses. Concentrations of the metals are reported as umole per gram dry sediment (umole/g).

SEM is then calculated as the sum of the metal concentrations:

$$SEM = \sum[metals]$$

Table 1.1
Method Analytes

Name	Symbol	Wavelength (4300 Model)	Cas Number
Aluminum	Al	396.1	7429-90-5
Antimony	Sb	206.8	7440-36-0
Arsenic	As	189.0	7440-38-2
Barium	Ba	233.5	7440-39-3
Beryllium	Be	234.9	7440-41-7
Boron	B	249.7	7440-42-8
Cadmium	Cd	228.8	7440-43-9
Calcium	Ca	227.5	7440-70-2
Chromium	Cr	267.7	7440-47-3
Cobalt	Co	228.6	7440-48-4
Copper	Cu	327.4	7440-50-8
Iron	Fe	239.6	7439-89-6
Lead	Pb	220.4	7439-92-1
Lithium	Li	670.7	7439-93-2
Magnesium	Mg	279.1	7439-95-4
Manganese	Mn	257.6	7439-96-5
Molybdenum	Mo	202.0	7439-98-7
Nickel	Ni	231.6	7440-02-0
Phosphorus	P	214.9	7723-14-0
Potassium	K	766.5	7440-09-7
Selenium	Se	196.0	7782-49-2
Silicon	Si	251.6	7440-21-3
Silver	Ag	328.1	7440-22-4
Sodium	Na	589.6	7440-23-5
Strontium	Sr	407.8	7440-23-5
Thallium	Tl	190.8	7440-28-0
Tin	Sn	189.9	7440-31-5
Titanium	Ti	334.9	7440-32-6
Vanadium	V	290.9	7440-62-2
Zinc	Zn	206.2	7440-66-6
Calculated			
Hardness, Calculated (as $CaCO_3$)	$CaCO_3$	NA	72608-12-9
Silica (as SiO_2)	SiO_2	NA	99439-28-8

Table 4-1
Optima 4300 MDLs, RLs and Linear Ranges for 6010/200.7

Metal	Upper Linear Range (mg/L)	Verified MDL Water (mg/L)	Reporting Limit Water (mg/L)	Verified MDL Soil (mg/Kg)	Reporting Limit Soil (mg/Kg)
Aluminum	450	0.1	0.2	10	20
Antimony	45	0.05	0.1	0.5	1.0
Arsenic	9	0.005	0.01	0.5	1.0
Barium	9	0.005	0.01	0.25	0.5
Beryllium	4.5	0.001	0.002	0.0125	0.025
Boron	45	0.05	0.1	12.5	25
Cadmium	9	0.0005	0.001	0.05	0.1
Calcium	450	0.25	0.5	25	50
Chromium	45	0.0025	0.005	0.125	0.25
Cobalt	45	0.0025	0.005	0.125	0.25
Copper	45	0.0025	0.005	0.5	1.0
Iron	450	0.025	0.1	1.0	3.0
Lead	90	0.005	0.010	0.5	1.0
Lithium	0.9	0.05	0.1	2.5	5.0
Magnesium	450	.0.25	0.5	12.5	25
Manganese	27	0.005	0.01	0.25	0.5
Molybdenum	45	0.005	0.01	1.5	3.0
Nickel	45	0.01	0.04	1.	2.0
Phosphorus	180	0.5	1.0	25	50
Potassium	72	0.5	1.0	25	50
Selenium	45	0.01	0.02	0.5	1.0
Silica (as SiO_2)	77.0	1.07	2.14	NA	NA
Silicon	36	0.5	1.0	NA	NA
Silver	9	0.002	0.004	0.25	0.5
Sodium	180	0.25	0.5	12.5	25
Strontium	4.5	0.025	0.05	0.25	0.5
Thallium	45	0.1	0.2	1.0	2.0
Tin	45	0.25	0.5	12.5	25
Titanium	45	0.015	0.03	1.0	2.0
Vanadium	45	0.005	0.01	0.25	0.5
Zinc	45	0.01	0.02	0.5	1.0

Table 4.2a
Microbac's Precision & Accuracy for Inorganic Metals Analyses of Groundwater & Solid Waste for 6010B/200.7

Metal	Accuracy, Water (% Recovery)	Precision, Water (% RPD)	Accuracy, Soil (% Recovery)	Precision, Soil (% RPD)
Aluminum	85 - 115	0 - 20	80 - 120	0 - 20
Antimony	85 - 115	0 - 20	80 - 120	0 - 20
Arsenic	85 - 115	0 - 20	80 - 120	0 - 20
Barium	85 - 115	0 - 20	80 - 120	0 - 20
Beryllium	85 - 115	0 - 20	80 - 120	0 - 20
Boron	85 - 115	0 - 20	80 - 120	0 - 20
Cadmium	85 - 115	0 - 20	80 - 120	0 - 20
Calcium	85 - 115	0 - 20	80 - 120	0 - 20
Chromium	85 - 115	0 - 20	80 - 120	0 - 20
Cobalt	85 - 115	0 - 20	80 - 120	0 - 20
Copper	85 - 115	0 - 20	80 - 120	0 - 20
Iron	85 - 115	0 - 20	80 - 120	0 - 20
Lead	85 - 115	0 - 20	80 - 120	0 - 20
Lithium	85 - 115	0 - 20	80 - 120	0 - 20
Magnesium	85 - 115	0 - 20	80 - 120	0 - 20
Manganese	85 - 115	0 - 20	80 - 120	0 - 20
Molybdenum	85 - 115	0 - 20	80 - 120	0 - 20
Nickel	85 - 115	0 - 20	80 - 120	0 - 20
Phosphorus	85-115	0-20	80-120	0-20
Potassium	85 - 115	0 - 20	80 - 120	0 - 20
Selenium	85 - 115	0 - 20	80 - 120	0 - 20
Silicon	85 - 115	0 - 20	80 - 120	0 - 20
Silver	85 - 115	0 - 20	80 - 120	0 - 20
Sodium	85 - 115	0 - 20	80 - 120	0 - 20
Strontium	85 - 115	0 - 20	80 - 120	0 - 20
Thallium	85 - 115	0 - 20	80 - 120	0 - 20
Tin	85 - 115	0 - 20	80 - 120	0 - 20
Titanium	85 - 115	0 - 20	80 - 120	0 - 20
Vanadium	85 - 115	0 - 20	80 - 120	0 - 20
Zinc	85 - 115	0 - 20	80 - 120	0 - 20
Phosphorus	85 - 115	0 - 20	80 - 120	0 - 20
Zirconium	85 - 115	0 - 20	80 - 120	0 - 20

Table 4.2b
Microbac's Precision & Accuracy for Inorganic Metals Analyses of Groundwater & Solid Waste for 6010C

Metal	Accuracy, Water (% Recovery)	Precision, Water (% RPD)	Accuracy, Soil (% Recovery)	Precision, Soil (% RPD)
Aluminum	80 - 120	0 - 20	80 - 120	0 - 20
Antimony	80 - 120	0 - 20	80 - 120	0 - 20
Arsenic	80 - 120	0 - 20	80 - 120	0 - 20
Barium	80 - 120	0 - 20	80 - 120	0 - 20
Beryllium	80 - 120	0 - 20	80 - 120	0 - 20
Boron	80 - 120	0 - 20	80 - 120	0 - 20
Cadmium	80 - 120	0 - 20	80 - 120	0 - 20
Calcium	80 - 120	0 - 20	80 - 120	0 - 20
Chromium	80 - 120	0 - 20	80 - 120	0 - 20
Cobalt	80 - 120	0 - 20	80 - 120	0 - 20
Copper	80 - 120	0 - 20	80 - 120	0 - 20
Iron	80 - 120	0 - 20	80 - 120	0 - 20
Lead	80 - 120	0 - 20	80 - 120	0 - 20
Lithium	80 - 120	0 - 20	80 - 120	0 - 20
Magnesium	80 - 120	0 - 20	80 - 120	0 - 20
Manganese	80 - 120	0 - 20	80 - 120	0 - 20
Molybdenum	80 - 120	0 - 20	80 - 120	0 - 20
Nickel	80 - 120	0 - 20	80 - 120	0 - 20
Phosphorus	80 - 120	0 - 20	80 - 120	0 - 20
Potassium	80 - 120	0 - 20	80 - 120	0 - 20
Selenium	80 - 120	0 - 20	80 - 120	0 - 20
Silicon	80 - 120	0 - 20	80 - 120	0 - 20
Silver	80 - 120	0 - 20	80 - 120	0 - 20
Sodium	80 - 120	0 - 20	80 - 120	0 - 20
Strontium	80 - 120	0 - 20	80 - 120	0 - 20
Thallium	80 - 120	0 - 20	80 - 120	0 - 20
Tin	80 - 120	0 - 20	80 - 120	0 - 20
Titanium	80 - 120	0 - 20	80 - 120	0 - 20
Vanadium	80 - 120	0 - 20	80 - 120	0 - 20
Zinc	80 - 120	0 - 20	80 - 120	0 - 20
Phosphorus	80 – 120	0 – 20	80 – 120	0 - 20
Zirconium	80 – 120	0 – 20	80 – 120	0 - 20

Table 5-1
Interelement Correction Factors for Optima 4300

Factors per 100 mg/L Interferant							
Analyte	**Al**	**As**	**Ba**	**Be**	**Ca**	**Co**	**Cr**
Aluminum	-	-	-	-	-	-	-
Antimony	0.447967	-	-	-	-	-	4.19
Arsenic	-	-	.189139	-	.0981	-	-4.87
Barium	-	-	-	-	-	-	-
Beryllium	-	-	-	-	-	-	-.0120651
Boron	-	-	-	-	-	4.99	-
Cadmium	-	6.15489	-0.0191992	-	-	-6.26327	-
Calcium	-	-	-	-	-	-	-
Chromium	-	-	-	-	-	-	-
Cobalt	-	-	-1.17228	-	-	-	.159
Copper	-	-	-	-	-.0252062	.218641	-
Iron	-	-	-	-	-	4.06579	-
Lead	-.126299	-	-	-	-.0202022	.106579	-
Lithium	-	-	-	-	-	-	-
Magnesium	-	-	-	-	-	-	-
Manganese	-	-	-	-	-	-	-
Molybdenum	-	-	-	-	-	-	-
Nickel	-	-	-	-	-	.976335	-
Phosphorus		-	-	-	-	-	-
Potassium	.620975	-	-	-	.574622	-	-
Selenium	-	-	-	-	-	-.794408	-
Silicon	-	-	-	-	-	-	-
Silver	-	-	-	-	-.0118481	-	-
Sodium	.718161	-	-	-	.698715	-	-
Strontium	-	-	-	-	-	-	-
Thallium	-0.0169987	-	-	-	-.033338	-.183759	0.4
Tin	-	-	-	-	-.0529445	-	-
Titanium	-	-	-	-	-	-	.27456
Vanadium	-	-	-	-	-	-	-1.433
Zinc	0.001	-	-	-	-	-	-3.34

Table 5-1
Interelement Correction Factors for Optima 4300 (continued)

Factors per 100 mg/L Interferant						
Analyte	**Cu**	**Fe**	**Mg**	**Mn**	**Mo**	**Ni**
Aluminum	-	-	-	-	44	-
Antimony	-	-	-	-	-24.4	-.681302
Arsenic	-	-.146	-	-	.742	-
Barium	-	-	-	-	-	-
Beryllium	-	-	-	-	-	-
Boron	-	-4.35839	-	-	-	-
Cadmium	-	0.0014	-	-	.0190091	-.0568649
Calcium	-	-	-	-	-	-
Chromium	-	-.0183539	-	.328	-.26	-
Cobalt	-	.00668691	-	-	-.93	.146461
Copper	-	-.0200902	-	-	.4	-
Iron	-	-	-	-	-	-
Lead	-	.0570054	-	-	-2.56406	-
Lithium	0.0647055	-	-	-	-	-
Magnesium	-	-	-	-4.05	-1.64	-
Manganese	-	-.0170384	.0157809	-	-	-
Molybdenum	-	-0.0444556	-	-	-	-
Nickel	-	.0255009	-	-	-	-
Phosphorus	-	-	-	-	-	-
Potassium	-	.696417	.526097	-	-	-
Selenium	-	-.6	-	.405	.0945449	-
Silicon	-	-.135546	-	-	10.7478	-
Silver	-	-.208152	-	-	-.24288	-
Sodium	-	.837684	.750261	-	-	-
Strontium	-	-	-	-	-	-
Thallium	-	-0.0726809	-	-1.036	.119755	-
Tin	-	-.0519835	-.0314678	-	-	-
Titanium	-	-	.0228899	-	-.250622	-
Vanadium	-	0.1	.1	-	-40	-
Zinc	-	.001	.0001	-	-	-

Table 5-1
Interelement Correction Factors for Optima 4300 (continued)

Factors per 100 mg/L Interferant							
Analyte	**Sb**	**Se**	**Sn**	**Ti**	**Tl**	**V**	**Zn**
Aluminum	-	-	-	-	-	-	-
Antimony	-	.568779	-14.4736	2.11867	-	-2.31443	-
Arsenic	-	-	-	-	-	.214681	-
Barium	-	-	-	-	-	-2.27389	-
Beryllium	-	-	-	-.01	-	-0.0329788	-
Boron	-	-	-	-	-	-	-
Cadmium	-.0173125	-	-	-.0249829	-	.0642357	-.0109044
Calcium	-	-	-	-	-	-	-
Chromium	-	-	-	.118925	-	-.333864	-
Cobalt	-	-	-.0434213	2.34234	-	-	-
Copper	-	-	-	-.117406	-	-.21956	-
Iron	-	-	-	-	-	-	-
Lead	-	-	-	-.138344	-	-.0970818	-
Lithium	-	-	-	-	-	-	-
Magnesium	-	-	-	-2.32782	-	-	-
Manganese	-	-	-	-	-	-	-
Molybdenum	-	-	-	-	-	-.202941	-
Nickel	-.302278	-	-	-	.378754	-	-
Phosphorus	-	-	-	-	-	-	-
Potassium	-	-	-	-	-	-	-
Selenium	-	-	.185983	-	-	.250848	.0887204
Silicon	-	-	-	-	-	-	-
Silver	-	-	-	-	-	-2.88494	-
Sodium	-	-	-	-	-	-	-
Strontium	-	-	-	-	-	-	-
Thallium	-	-	-	-5.25518	-	-.406757	-
Tin	-	-	-	-3.02151	.549717	.389475	.269888
Titanium	-	-	-	-	-	-	-
Vanadium	-	-	-	-	-	-	-
Zinc	-	-	-	-	-	-	-

Table 7.4.1
Calibration Standards Concentrations in mg/L for OPTIMA 4300

Element	S0	S1	S2	S3	S4	Stock Concentration
Aluminum	0.0	0.08	0.16	10.0	20.0	2000
Antimony	0.0	0.0096	0.0192	1.2	2.4	240
Arsenic	0.0	NA	0.0064	0.4	0.8	80
Barium	0.0	0.008	0.016	1.0	2.0	200
Beryllium	0.0	0.0004	0.0008	0.05	0.1	10
Boron	0.0	NA	0.008	0.5	1.0	100
Cadmium	0.0	0.0004	0.0008	0.5	0.1	10
Calcium	0.0	NA	0.16	10.0	20.0	2000
Chromium	0.0	0.004	0.008	0.5	1.0	100
Cobalt	0.0	0.0016	0.0032	0.2	0.4	40
Copper	0.0	0.004	0.008	0.5	1.0	100
Iron	0.0	0.032	0.064	4.0	8.0	800
Lead	0.0	0.004	0.008	0.5	1.0	100
Lithium	0.0	0.008	0.016	1.0	2.0	200
Magnesium	0.0	0.08	0.16	10.0	20.0	2000
Manganese	0.0	0.004	0.008	0.5	1.0	100
Molybdenum	0.0	0.008	0.016	1.0	2.0	200
Nickel	0.0	0.004	0.008	0.5	1.0	100
Phosphorus	0.0	0.08	0.16	10	20	2000
Potassium	0.0	0.4	0.8	50.0	100.0	10000
Selenium	0.0	NA	0.0062	0.4	0.8	80
Silicon	0.0	0.04	0.08	5.0	10.0	1000
Silver	0.0	0.0032	0.0064	0.4	0.8	80
Sodium	0.0	0.4	0.8	50.0	100.0	10000
Strontium	0.0	0.008	0.016	1.0	2.0	200
Thallium	0.0	NA	0.008	0.5	1.0	100
Tin	0.0	0.008	0.016	1.0	2.0	200
Titanium	0.0	0.008	0.016	1.0	2.0	200
Vanadium	0.0	0.008	0.016	1.0	2.0	200
Zinc	0.0	0.008	0.016	1.0	2.0	200

NA = S1 is not used in construction of the calibration curve for this element.
The concentration of S2 is at or below the reporting limit for this element.

Table 7.5.1
Initial and Continuing Calibration Standards Concentration in mg/L

Element	ICV/CCV	Stock Concentration
Aluminum	10.0	2000
Antimony	1.2	240
Arsenic	0.4	80
Barium	1.0	200
Beryllium	0.05	10
Boron	0.5	100
Cadmium	0.05	10
Calcium	10.0	2000
Chromium	0.5	100
Cobalt	0.2	40
Copper	0.5	100
Iron	4.0	800
Lead	0.5	100
Lithium	1.0	200
Magnesium	10.0	2000
Manganese	0.5	100
Molybdenum	1.0	200
Nickel	0.5	100
Phosphorus	10.0	2000
Potassium	50.0	10000
Selenium	0.4	80
Silicon	5.0	1000
Silver	0.4	80
Sodium	50.0	10000
Strontium	1.0	200
Thallium	0.5	100
Tin	1.0	200
Titanium	1.0	200
Vanadium	1.0	200
Zinc	1.0	200

Table 7.7.1
ICSA and ICSAB Concentration in mg/L

Element	ICSA	ICSAB	Stock Concentration
Aluminum	250	250	5000
Antimony	-	0.5	100
Arsenic	-	0.25	50
Barium	-	0.25	50
Beryllium	-	0.25	50
Cadmium	-	0.5	100
Calcium	250	250	5000
Chromium	-	0.25	50
Cobalt	-	0.25	50
Copper	-	0.25	50
Iron	100	100	2000
Lead	-	0.5	100
Magnesium	250	250	5000
Manganese	-	0.25	50
Nickel	-	0.5	100
Potassium	-	5	1000
Selenium	-	0.25	50
Silver	-	0.5	100
Sodium	-	5.0	1000
Thallium	-	0.5	100
Vanadium	-	0.25	50
Zinc	-	0.5	100

Table 13.1
Quality Control Criteria Total Metals - ICP
Method 6010B

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial calibration	Daily at beginning Of analytical run	COC ≥ 0.995	Investigate, recalibrate
Initial Calibration Verification (ICV)	After calibration	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Verification (CCV)	Minimum every 10 samples	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Initial Calibration Blank (ICB)	After ICV	< RDL < 1/2 RDL < 3 x IDL < MDL (1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank (CCB)	minimum every 10 samples	< RDL < 1/2 RDL < 3 x IDL < MDL (1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB
Method Blank	One per batch (20 samples maximum per batch)	< RDL or < MDL or < MDL x 2 (1)	Stop analysis, investigate, reanalyze. If still > limit, qualify data and address in narrative
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One per batch (20 samples maximum per batch)	Control Limits 80 - 120% Soil 85 – 115 % Water	Stop analysis, investigate, reanalyze. If still outside limits, qualify data and address in narrative
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per batch (20 samples maximum per batch)	80 - 120% recovery RPD ≤ 20%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified
Duplicate	One per batch (20 samples maximum per batch)	RPD ≤ 20%	Qualify data and address in narrative if client specified
ICP interference check	Run at beginning of each run	80 - 120% of true value for EPA check sample element. < RL or project specific criteria for non-spiked elements	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Post digestion spike	5%, or minimum of 1 per batch	75 - 125% recovery	Serial dilution if applicable or dilute and repeat post digestion spike.
Serial Dilution	If post digestion spike fails	± 10% of original determination	Dilute and repeat Post digestion spike

(1) Acceptance criteria are analyte specific and instrument specific

Table 13.2
Quality Control Criteria Total Metals - ICP
Method 200.7

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA (1)	CORRECTIVE ACTION
Initial calibration	Daily at beginning Of analytical run	COC ≥ 0.995	Investigate, recalibrate
Initial Calibration Verification (ICV)	After calibration	95 – 105% RSD < 3%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Verification (CCV)	Minimum every 10 samples	90 – 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCV
Initial Calibration Blank (ICB)	After ICV	< RDL < 1/2 RDL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate
Continuing Calibration Blank (CCB)	minimum every 10 samples	< RDL < 1/2 RDL < 3 x IDL < MDL	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, reanalyze all samples run after the last compliant CCB
Method Blank	One per batch (20 samples maximum per batch)	< RDL or < MDL	Stop analysis, investigate, reanalyze. If still > limit, qualify data and address in narrative
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One per batch (20 samples maximum per batch)	Water 85 – 115 % (STD) 80 – 120 % (DoD) Soil 80-120 %	Stop analysis, investigate, reanalyze. If still outside limits, qualify data and address in narrative
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per ten samples (20 samples maximum per batch)	80- 120% recovery RPD ≤ 20%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified
Duplicate	One per batch (20 samples maximum per batch)	RPD ≤ 20%	Qualify data and address in narrative if client specified
ICP interference check ICS – A ICS -AB	Run at beginning of each run	Spiked Elements 80 – 120 % Nonspiked Elements <RL (STD) < 2 x MDL (DoD)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Post digestion spike	5%, or minimum of 1 per batch	75 – 125% recovery	Serial dilution if applicable or dilute and repeat post digestion spike.
Serial Dilution	If post digestion spike fails	± 10% of original determination	Dilute and repeat Post digestion spike

(1) Acceptance criteria are project specific. Consult QAPP

Table 13.3
Quality Control Criteria Total Metals – ICP
Method 6010C

CONTROL ITEM	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Initial Calibration	Daily, at beginning of run	COC ≥ 0.998	Investigate, recalibrate.
Initial Calibration Verification (ICV)	After calibration	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Continuing Calibration Verification (CCV)	After ICSA/ICSAB and minimum every 10 samples	90 - 110%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, and reanalyze all samples analyzed after last compliant CCV.
Initial Calibration Blank (ICB)	After ICV	< RL < ½RL < 3X IDL < 2X MDL(1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Continuing Calibration Blank (CCB)	After every CCV, minimum every 10 samples	< RL < ½RL < 3X IDL < 2X MDL(1)	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, and reanalyze all samples analyzed after last compliant CCB.
Low Level Initial Calibration Verification (LLICV)	After ICB	70 - 130%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Low Level Continuing Calibration Verification (LLCCV)	Minimum following closing batch CCB	70 - 130%	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate, and reanalyze all samples analyzed after last compliant LLCCV.
Method Blank	Once per batch (20 samples maximum per batch)	< RL or < MDL < ½ RL < 2X MDL(1)	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch or qualify data and address in narrative
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LCSD)	Once per batch (20 samples maximum per batch)	80 - 120%	Stop analysis, investigate, reanalyze. If still outside limits, redigest batch or qualify data and address in narrative
Matrix Spike/ Matrix Spike Duplicate (MS/MSD)	Once per batch (20 samples maximum per batch)	75 - 125%	Perform post digestion spike and/or serial dilution. Qualify data and address in narrative if client specified.
Duplicate (DP)	Once per batch (20 samples maximum per batch) upon client request	RPD ≤ 20%	Qualify data and address in narrative if client specified.
ICP Interference Check Samples (ICSA/ICSAB)	After ICB, prior to sample analysis	80 - 120% for spiked analytes; < RL or other project specific criteria for nonspiked analytes	Stop analysis, investigate, reanalyze. If still outside limits, recalibrate.
Post Digestion Spike (PS)	One per batch	80 - 120%	Serial dilution if applicable or dilute and repeat post digestion spike.
Serial Dilution (DL)	One per batch, if sample concentration > 50X MDL	± 10% of original determination	Dilute and repeat post digestion spike.

(1) Acceptance criteria are analyte specific and instrument specific.

Table 13.3.2a
Purchased Standard KEM-SPK-1F from Inorganic Ventures with the following concentrations:

Element	Concentration (mg/L)
Potassium	250
Sodium	250
Aluminum	50
Calcium	50
Magnesium	50
Silicon	25
Iron	20
Antimony	6
Barium	5
Lithium	5
Molybdenum	5
Strontium	5
Titanium	5
Vanadium	5
Zinc	5
Boron	2.5
Chromium	2.5
Copper	2.5
Manganese	2.5
Nickel	2.5
Lead	2.5
Thallium	2.5
Arsenic	2
Selenium	2
Silver	25
Cobalt	1
Beryllium	0.25
Cadmium	0.25

Table 13.3.2b
Final LCS/MS Concentrations

ELEMENT	WATER (mg/L)	SOIL (mg/Kg)
Potassium	25	1250
Sodium	25	1250
Aluminum	5	250
Calcium	5	250
Magnesium	5	250
Silicon	2.5	NA
Iron	2	100
Antimony	0.6	30
Barium	0.5	25
Lithium	0.5	25
Molybdenum	0.5	25
Strontium	0.5	25
Titanium	0.5	25
Vanadium	0.5	25
Zinc	0.5	25
Boron	0.25	12.5
Chromium	0.25	12.5
Copper	0.25	12.5
Manganese	0.25	12.5
Nickel	0.25	12.5
Lead	0.25	12.5
Thallium	0.25	12.25
Arsenic	0.2	10
Selenium	0.2	10
Silver	0.2	10
Cobalt	0.1	5
Beryllium	0.025	1.25
Cadmium	0.025	1.25
Tin*	0.5	25
Phosphorus*	5	250
Zirconium*	0.5	25

*Added as needed from single element solutions.

Figure 12.1

Example 6010 Calculations
Perkin Elmer Optima 4300 DV

1.0 Initial Calibration (ICAL) Parameters
The system performs linear regression from data consisting of a blank and three standards.

2.0 Calculating the concentration © of an element in water using data from prep log, run log, and quantitation report (NOTE: the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Vi} \times D$$

Where:	**Example:**
Cs = Concentration computed by the data system in ug/mL (ppm)	0.08
Vf = Final volume (mL)	50
Vi = Initial volume (mL)	40
D = Dilution factor as a multiplier (10X = 10)	1
Cx = Concentration of element in ug/mL (mg/L)	0.1

3.0 Calculating the concentration © of an element in soil using data from prep log, run log, and quantitation report (NOTE: the data system performs this calculation automatically when correction factors have been entered):

$$Cx = Cs \times \frac{Vf}{Vi} \times D$$

Where**:**	**Example:**
Cs = Concentration computed by the data system (mg/L) (ppm)	0.1
Vf = Final volume (mL)	50
Vi = Initial weight (g)	1
D = Dilution factor as a multiplier (10X = 10)	1
Cx = Concentration of element in ug/g (mg/kg)	5

4.0 Adjusting the concentration to dry weight:

$$Cdry = \frac{Cx \times 100}{Px}$$

Where**:**	**Example:**
Cx = Concentration calculated as received (wet basis)	5
Px = Percent solids of sample (% wt)	80
Cdry = Concentration calculated as fry weight (mg/kg)	6.25

Figure 14.1

Checklist ID: 62336

Microbac Laboratories Inc.

Data Checklist

Date: 12-DEC-2011
Analyst: KHR
Analyst: NA
Method: 6010
Instrument: PE-ICP2
Curve Workgroup: 384458
Runlog ID: 44204
Analytical Workgroups: 384170, 383839, 384366, 383507, 384440

Calibration/Linearity	X
ICV/CCV	X
ICV RSD <= 3% (EPA 200.7 only)	X
ICB/CCB	X
ICSA/ICSAB	X
CRI	
Blank/LCS	X
MS/MSD	X
Post Spike/Serial Dilution	X
Upload Results	X
Data Qualifiers	
Generate PDF Instrument Data	X
Sign/Annotate PDF Data	X
Upload Curve Data	X
Workgroup Forms	X
Case Narrative	X
Client Forms	X
Level X	
Level 3	208
Level 4	760, 028, 230
Check for compliance with method and project specific requirements	X
Check the completeness of reported information	X
Check the information for the report narrative	X
Primary Reviewer	KHR
Secondary Reviewer	SLP
Comments	

Primary Reviewer:
13-DEC-2011

Secondary Reviewer:
14-DEC-2011

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ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AIR SAMPLES COLLECTED IN SPECIALLY PREPARED CANISTERS AND GAS COLLECTION BAGS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS)
REFERENCED METHOD:	EPA TO-15
SOP ID:	VOA-TO15
REV. NUMBER:	21.0
EFFECTIVE DATE:	02/15/2014

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ALS Environmental

STANDARD OPERATING PROCEDURE

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

EPA TO-15

SOP ID:	VOA-TO15	Rev. Number:	21.0	Effective Date:	02/15/2014

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

1) Scope and Applicability

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

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

Note 1: A number of compounds selected as target analytes are not included in the TO-15 Method; therefore the Florida Department of Health (FL DOH) required the laboratory to reference this Standard Operating Procedure (SOP) when reporting results for these analytes. However, the FL DOH disapproved of the SOP Code for this purpose, so CASS TO-15/GC-MS shall be referenced. Consequently, VOA-TO15 is otherwise known as CASS TO-15/GC-MS and shall be referenced, where necessary (on quantitation reports).

2) Summary of Procedure

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

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

(programmed) increases and the VOCs elute and are detected by the mass spectrometer.

Mass spectra for individual peaks in the total ion chromatogram are examined with respect to the fragmentation pattern of ions corresponding to various VOCs including the intensity of primary and secondary ions. The fragmentation pattern is compared with stored spectra taken under similar conditions, in order to identify the compound. For any given compound, the intensity of the primary fragment is compared with the system response to the primary fragment for known amounts of the compound. This method utilizes the internal standard calibration technique; refer to Section 3.16 for a complete definition.

3) Definitions

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

3.21 SOP Standard Operating Procedure

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

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

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

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

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

4) Health and Safety Warnings

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

4.2 Safety Data Sheets (SDS)

The analyst should consult SDS for compounds being handled in the course of this procedure, and be familiar with proper safety precautions to be followed when handling hazardous chemicals. Care should be taken when handling standard material in a neat or highly concentrated form.

4.3 Liquid Nitrogen

Liquid nitrogen can cause serious tissue damage (frostbite) with only a few seconds of contact. The valves on the cryogen dewars should be opened slowly so leaky fittings can be identified. Neoprene or leather gloves should be worn when turning valves and handling tubing and fittings that have been in contact with the cryogen.

4.4 Protective Clothing

Personal protective clothing (safety glasses, gloves and lab coat) are required when preparing standards and handling standard material in neat form.

4.5 Pressurized Gases

The use of pressurized gases is required for this procedure. Care should be taken when moving cylinders. All gas cylinders must be secured to a wall or an immovable counter with a chain or a cylinder clamp when not in use. The regulator should never remain on small "D" size cylinders following use. Sources of flammable gases (i.e. pressurized hydrogen) should be clearly labeled.

4.6 Syringes

The proper use of syringes should be part of employee training for this SOP. Care should be taken to avoid personal injury as a result or improper handling techniques.

4.7 Pollution Prevention and Waste Management

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

5) Cautions

5.1 A maintenance log will be kept documenting maintenance performed on each analytical system. The serial numbers of each instrument shall be recorded, and each

log entry must include a description of the maintenance performed and be initialed by the analyst performing or observing/authorizing maintenance by an outside contractor.

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

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

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

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

5.2 Concentrating Trap

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

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

5.3 GC System

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

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

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

5.4 Mass Spectrometer

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

5.5 Instrument Tuning

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

5.6 Computer Troubleshooting

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

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

Basic Troubleshooting Outline:

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

6) Interferences

6.1 Summa Canisters

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

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

Current laboratory practice entails the segregation of 6L canisters into ambient (low) level and source levels. All the ambient canisters are used for low level (indoor air, ambient air) projects and not intentionally for soil gas, SVE monitoring, or other higher level applications. It may be necessary to "retire" an ambient canister and re-assign for source level use if high concentrations are encountered. This decision will be made by management based on analytical concentrations and what compounds were encountered at these levels. If the level of any analyte is detected above 5,000ug/m*3* in the ambient can, then the supervisor/team leader must be contacted to determine if the canister(s) is to be retired. If retirement is decided upon, make a notation on the sample tag (or other color coded tag) of each canister in question. The notation must contain the analyte, threshold levels and retirement from ambient use (initial and date notation) so that the canister conditioning/management department may properly execute the retirement.

6.2 Analytical System

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

6.3 Carbon Dioxide

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

6.4 Gas Collection Bags

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

6.5 Glassware

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

7) Personnel Qualifications and Responsibilities

7.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP may perform analysis, interpretation and peer review of the results. Data reduction and/or peer review may be performed by another qualified employee. This employee

must be familiar with the analytical technique and have completed a data review training plan to ensure familiarity with specific analysis and requirements.

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

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

7.4 Demonstration of Capability

All analysts must be trained in accordance with the guidelines detailed in the *SOP for Training Policy*. Demonstrations shall also be performed in accordance with the 2009 TNI Standards (Volume 1 Module 4 Section 1.6) and DoD Quality Systems Manual 5.0. Attachment 1 shall be used to document the training plan for new analysts' initial demonstration. Additionally, these demonstrations are performed anytime there is a change in instrument type, personnel or method.

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

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

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

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

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

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

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

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

8) Sample Collection, Handling, and Preservation

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

Controllers and Critical Orifices) utilizing the canister vacuum to draw the sample. Bags require the use of an upstream pump or a "lung machine."

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

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

9) Equipment and Supplies

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

9.2 Gas Chromatograph (GC)

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

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

9.3 Autosampler

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

9.4 Mass Spectrometer (MS)

A MS capable of scanning from 34 to 350 amu every second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for Bromofluorobenzene

(BFB) which meets all of the criteria when 50ng or less of BFB is injected onto the GC/MS system.

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

9.4.1 Ionization Gauge Controller

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

9.5 Analytical Column

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

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

  OR

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

9.6 Data Systems

IBM-compatible PC with Windows 95/98/NT/XP/7 (Microsoft Office EXCEL version 2003 or newer) and Hewlett Packard Chemstation software including EnviroQuant with Extracted Ion Current Profile (EICP), National Institute of Standards and Technology (NIST) library (2002 version or newer) or equivalent.

9.7 Canister Pressurization Station

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

9.8 Canister Sampling Devices

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

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

9.9 Gas Collection Devices

- Lab Commerce, Aerosphere Model S6L, 6.0L Summa Passivated Canisters or equivalent
- Lab Commerce, Stabilizer Model 22.4L, 2.4L Canisters or equivalent
- Restek Corporation, #24203, 3.0L Silco Canisters or equivalent

- Tedlar bags – 0.5L, 1L, 3L, 5L, 10L, 25L, and 40L (other sizes are available; however, the volumes that are listed encompass the majority of the bags supplied and the samples submitted to the laboratory).

9.10 Dynamic Dilution System

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

10) Standards and Reagents

10.1 Reagents and Equipment

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

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

10.1.3 UHP/Zero Grade Air (canister pressurization)

10.1.4 ASTM Type II Water, DI water or equivalent

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

10.2 Standards

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

10.2.1 Instrument Performance Check, Internal Standard and Surrogate Spiking Mixture Prepare a standard solution of p-Bromofluorobenzene (BFB-used as both a tune check and surrogate compound), bromochloromethane, chlorobenzene-d5, and 1,4-difluorobenzene, 1,2-dichloroethane-d4(surrogate), and toluene-d8(surrogate) at 500µg/m³ each in humidified zero air (Section 9.2.1.2). Prepare this standard according to the procedure outlined in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note. This standard may also be prepared from a neat cocktail as in Section 10.2.2.2.1 or as stated in Section 10.2.1.3.

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

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

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

Where:

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

Example:

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

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

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

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

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

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

PV = PDF(V) (Equation 2)

Where:

PV Pressurized canister volume (L)

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

P_f Final Canister Pressure

P_i Initial Canister Pressure

V Volume of canister at 1atm

P_{atm} Atmospheric Pressure = 14.7psig

Example:

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

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

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

Where:

F Desired concentration of working standard (ng/L)

V Pressurized Volume of Canister (L)

C Concentration of prepared SDB (µg/mL)

A Amount of standard (mL) of the SDB required to obtain the desired working standard concentration

Example:

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

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

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

10.2.1.3.2 For SIM analyses, the working standard is diluted and pressurized with humid zero air to the desired concentration

using Equation 2 in Section 10.2.1.2. Typical concentrations will be 20ng/L, 40ng/L or 50ng/L.

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

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

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

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

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

Step1: Determine the required flow rate of the stock standards (positions #2 and #3). The range must be from 5 to 50sccm (standard cubic centimeters per minute, same as ml/min). The flows listed below are guidelines to be used for the default standard flow (based on the desired standard concentration) and were chosen based on the ultimate final dilution required and limitations of the Dynamic Diluter (flows must be from 150 to 2000ml/min.).

Desired Standard Conc.	Default Standard Flow
200ng/L	50ml/min
100ng/L	50ml/min
20ng/L	20ml/min
5.0ng/L	10ml/min
4.0ng/L	8ml/min

1ng/L	50ml/min; 20ml/min (See Note 1 below)
0.2ng/L	10ml/min; 20ml/min (See Note 1 below)

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

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

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

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

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

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

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

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

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

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

2nd Dilution Factor Needed = Total Dilution/1st Dilution
2nd Dilution Factor = 10000/200(1st dilution) = 50

Total Flow (Dilution 2) = 20ml/min*50 = 1000ml/min

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

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

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

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

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

Cocktail Preparation:

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

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

Where:

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

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

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

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

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

Where:

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

Example:

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

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

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

Where:

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

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

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

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

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

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

Example:

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

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

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

10.2.3 Initial Calibration Verification (ICV) - (Laboratory Control Sample - LCS) Prepare a secondary source standard (either a different manufacturer or different lot from the same manufacturer as the initial calibration standard) using the same procedures as the primary source. The ICV/LCS working standard should

contain each target analyte present in the calibration working standard. Prepare the ICV/LCS working standard at a concentration of 200ng/L. Differing injection volumes account for the allowed concentrations listed in Table 4 for SCAN and 4A for SIM. The preparation of this standard shall follow the instructions detailed in Section 10.2.2, using the certified second-source standard cylinder.

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

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

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

10.3 Storage and Expiration Dates

All standards that are to be stored in a freezer shall be stored at ≤-10˚C for DoD projects.

- Neat Stock Liquids are stored at < -10˚C (-10˚C to -20˚C) as specified by the manufacturer or for a period of five years.
- Equi-Mass Primary Stock Standard is a cocktail or soup of neat compounds (containing compounds in equal mass amounts) used to in preparing intermediate gas phase standards and shall be stored in the freezer at < -10˚C (-10˚C to -20˚C) for up to six months. This is assuming that the soup is sealed with a septum-containing screw cap or Mininert™ valve. The selection of the compounds for the soup should be performed in accordance with the guidelines in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.
- Purchased Stock Standards Cylinders must be stored at laboratory temperature for a period of 2 years or as specified by the manufacturer before vendor re-certification or purchase of new standards.
- Intermediate Calibration Standards prepared by static dilution must be stored in an oven at a temperature of approximately 60°C to ensure analyte vaporization. Every time a standard is prepared from the static dilution bottle (SDB), the concentration changes. To increase the useful lifetime of an SDB standard, remove volumes of 25mL or less. The volume removed can be manipulated by increasing the SDB concentration or by adjusting the canister final volume/pressure. Depending upon the volume removed, an SDB intermediate standard is stable for approximately two months as long as new working standards made from this standard continue to meet acceptance criteria. These bottles must be in the oven for a minimum of one hour prior to use in preparing working standards. The guidelines for the storage and expiration date for the intermediate calibration standards are stated in Volume 6.5 of the *Tekmar*-DOHRMANN Application Note.
- Prepared Stock / Intermediate Calibration Standards prepared in Summa canisters (1000ng/L) may be stored at laboratory conditions for up to three months in an atmosphere free of potential contaminants. Upon preparation, canister standards should be allowed to sit for approximately 24 hours prior to use in order for equilibration to take place. Shorter equilibration periods may be necessary and acceptable as long as performance criteria are met.

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

11) Method Calibration

11.1 Initial Calibration

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

Initial calibration requirements are as follows:

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

the ICAL documentation must state the revised calibration range if the MRL must be adjusted or the calibration range is lowered for a particular compound. This must be documented on the ICAL Review Checklist.

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

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

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

SCAN

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

11.1.2 Recalibration Each GC/MS system must be recalibrated following any instrument maintenance which may change or effect the sensitivity or linearity of the instrument, if the continuing calibration verification acceptance criteria are not met and at least annually. The following procedure must be followed when updating an initial calibration method.

1. Open the most recent method.
2. Save the method with the new ICAL method ID using the "Save Method As" option. Date used in the method ID must be the date files were analyzed.

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

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

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

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

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

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

11.1.4.2 LOQ Establishment, Verification and Acceptance Criteria

1. The LOQ must be set within the calibration range (≥ low std. of the current passing ICAL) prior to sample analysis.
2. The LOQ for each analyte must be ≥ the analyte's LOD.
3. Initially a passing demonstration of precision and bias must be performed at the LOQ.
4. Run CCV 2 times at LOQ and:
   a. Generate a duplicate report for precision using ±25% as the criteria.
   b. Check the %Rec using laboratory generated control limits.
   c. Check the signal to noise ratio (S/N) using the software. The S/N ratio must be at least 3:1 for each analyte.
   d. All ion abundances must be acceptable per the requirements set forth in this document.
5. If any compounds fail, verify at a higher level and notify reporting. Also, make a note in the ICAL documentation.
6. Turn in all LOQ verification data (quant reports and software reports/checks) to QA (regardless of pass/fail).
7. Verify the LOQ on each instrument quarterly.

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

Perform a review of specific aspects of the calibration which might compromise data quality such as inappropriate extension of the calibration range with detector saturation and/or a lack of sensitivity for any analyte. Analyte concentrations which do not meet the signal to noise ratio or exhibit saturation are not to be reported and must be eliminated from the initial calibration. These instances should be followed by a short explanation regarding the reason for the omission.

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

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

11.2 Initial Calibration Verification Standard

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

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

12) Sample Preparation/Analysis

12.1 Sample Preparation

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

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

12.2 Screening

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

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

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

Where:

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

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

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

12.3 Analytical Sequence and Data System Setup

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

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

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

Analytical Sequence Guideline

With Calibration	Tune Check[1] Calibration Standards (5 Standards Minimum) ICV Standard[2] (Acts as the ICV and LCS) QC Canister Checks[6] MB[7] Sample(s) – 1-19 Laboratory Duplicate[4]
With Continuing	Tune Check[1] CCV Standard[5] QC Canister Checks[6] MB[7] LCS[3] MRL Check Standard[8] Sample(s) – 1-19 Laboratory Duplicate[4]

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

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

12.4 Conditions

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

Adsorbent Trap

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

Refocusing Trap

Temperature:	-180˚C
Injection Temp.:	160˚C
Injection Time:	1.0 min

Adsorbent Trap Reconditioning Conditions

Temperature:	265°C
Initial Bakeout:	2 hours or until clean blank is obtained

After each run: 5-8 minutes

Sample Run Time

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

12.4.2 GC/MS System

Optimize GC conditions for compound separation and sensitivity.

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

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

12.5 Instrument Performance Check

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

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

- Inject 50ng or less (on column)
- Three scans (peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.
- Background subtraction is conducted using a single scan prior to the elution of BFB.
- All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.
- The ion abundance criteria must not be changed from the requirement stated in this document (TO-15 or TO-14A, as requested).

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

12.6 Continuing Calibration Verification Standard

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

12.7 Canister Quality Control Check and Method Blank

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

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

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

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

12.8 Laboratory Control Sample

The laboratory control sample is a sample matrix, which is free from the analytes of interest and spiked with a standard containing known amounts of analytes. The laboratory control sample is an injection of the initial calibration verification standard. Inject the LCS (ICV) at concentrations below the midpoint of the calibration curve.

Make sure that all of the pertinent information is included on the quantitation report including the sample identification (LCS), concentration, standard used, and analyst.

12.9 Sample Analysis

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

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

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

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

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

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

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

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

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

12.9.1 Sample Dilution If any target analyte results are above the highest level of the initial calibration, a smaller sample aliquot should be analyzed. The dynamic range of volume aliquots for the automatic cryogenic concentrator is 20cc to 1L. If a volume smaller than 20cc is to be analyzed, a dilution should be made in a Tedlar bag, or the sample directly injected using a gastight syringe. Guidance in performing dilutions and exceptions to this requirement are given below.

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

Tedlar bag dilution:

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

Direct injection:

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

12.9.2 Tentatively Identified Compounds When requested, a mass spectral library search may be made for the purpose of tentatively identifying sample components not associated with the calibration standards. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system mass spectral library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

Certain programs may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the

analyst assign a tentative identification. The following guidelines are used for making tentative identifications.

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

12.10 Duplicate

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

12.11 Internal Standard (IS)

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

12.12 Surrogates

Internal standards/surrogates must be added at the same volume for every standard, sample and QC sample. Surrogate compound recoveries are requested by a number of clients, but are more appropriately used as system monitoring compounds. This is due to the fact that the compounds are introduced directly into the analytical system and not into the canisters or bags. It is for this reason that they are not considered to be true surrogates and a fixed window is applied. Additionally, surrogates are not included in the ICAL because they are not required by the method and are only system monitoring compounds.

12.13 Manual Integration and Q Deletion

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

12.13.1 Manual Integration The integration for each peak must be legally defensible and shall be checked to ensure that it has been integrated properly and consistently between samples, standards and QC samples. All peak reviews and manual integrations must follow the requirements specified in the *SOP for Manual Integration Policy* and the *SOP for Laboratory Ethics and Data*

Integrity. The requirements in the above stated procedure include when manual integrations are performed, raw data records shall include a complete audit trail for those manipulations (i.e., chromatograms showing both the integration prior to any manual integrations and those depicting the corresponding manually integrated peaks), and notation of rationale, date, and initials of person performing the manual integration operation. In addition, manual integrations must be reviewed and approved by a second reviewer and the manual integrations maintained in the appropriate job file.

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

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

12.14 Detection Limits and Limits of Detection

The MDL study shall be performed annually for all target analytes on each instrument (with identical configurations) for which this method is performed. The MDL shall be performed in accordance with the procedure outlined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.* The detection limit shall be used to determine the LOD for each analyte.

Once determined on each instrument, the highest LOD (for each analyte from all instrument determinations) shall be used as the uniform LOD. However, if a lower detection limit is reported, then the samples must have been run on that specific instrument on which the lower LOD was determined.

12.14.1 Performance and Acceptance Criteria

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

12.15 Method Reporting Limit Check Standard

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

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

12.16 Method Modifications

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

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

13) Troubleshooting

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

14) Data Acquisition

14.1 Storing Electronic Data

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

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

Note: All data records must explicitly connect data to the initial instrument calibration. This includes all samples, continuing calibrations and QC samples.

14.3 The essential information to be associated with analysis, such as computer data files, run logs, etc. shall include: Sample ID code, date and time (if the holding time is 72 hours) of analysis, instrument operating conditions/parameters (or reference to such data), analysis type, all manual calculations including dilutions and manual integrations, analyst's initials, sample preparation (pressure readings and balance gas

if pressurized with helium), standard and reagent origin, receipt, preparation, and use, as well as calibration criteria, frequency and acceptance criteria, data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions.

15) Calculation and Data Reduction Requirements

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

15.2 Initial Calibration

Tabulate each of the following:

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

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

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

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

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

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

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

N is the number of calibration concentration levels.

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

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

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

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

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

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

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

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

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

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

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

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

$\overline{RRT}$ Mean relative retention time (seconds) for the target compound for all initial calibration levels.
RRT_i Relative retention time for the target compound in level i.
n Number of calibration levels

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

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

Y_i Area response for the primary quantitation ion for the internal standard for each initial calibration standard.
n number of calibration concentration levels

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

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

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

RT_i Retention time for the internal standard for each initial calibration standard, seconds.
n number of initial calibration levels

15.3 Continuing Calibration Verification

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

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

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

RRF_x is the RRF from the CCV being evaluated.
$\overline{RRF}$ is the mean RRF from the current calibration curve.

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

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

%R = X/TV x 100

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

15.5 Duplicate Analysis

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

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

x_1 First measurement value
x_2 Second measurement value
$\overline{x}$ Average of the two values

15.6 Internal Standards (IS)

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

15.7 Pressure Dilution Factor (PDF)

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

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

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

15.8 Results

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

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

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

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

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

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

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

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

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

MW is the molecular weight (Table 2) of the analyte, in g/mole.
24.46 is the molar volume of an ideal gas at 298 K (25 °C) and 760 mmHg (1 atm), in liters per mole (l/mol).
C_x the final analyte concentration in micrograms per cubic meter.

15.8.4 Equation Number 16 – Helium Pressurization (Injection Amount)

Applicable to canisters pressurized with helium and injected utilizing the mass flow controller of the AutoCAN. For full instructions and calculations, refer to

the 1st tab of the template located at: J:\A-GCMS\Helium Pressurization\MFC_GCF _backfill.

15.9 Data Review

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

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

15.10 Reporting

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

If the project requires that results be reported below the MRL (LOQ), but above the LOD all of the requirements specified for normal reporting apply (3:1 S/N ratio and ion abundance). This is regardless of the fact that the results will be qualified as estimated.

15.10.1 Analysis Observations / Case Narrative Summary Form

This form, which is included in the *SOP for Laboratory Storage, Analysis and Tracking*, must be generated when there are specific sample composition information or analysis issues and/or observations. In addition, during the analysis, specific identification information or problems, interferences, calibration issues, flags, and additional/expanded explanation of flags should be added to the form. This form may be modified as long as the sections and basic concepts are reserved. All data qualifiers and flags should follow those listed in the most recent Quality Assurance Manual or as defined in any client requirements.

This form is necessary as a means for documentation. This form, among other information, will be reviewed when compiling the final report and case narrative. All information regarding the job shall remain in the file, in order that sufficient documentation is available to recreate the job from sample receipt through analysis, data reduction, and reporting.

15.10.2 NELAC\TNI Requirements

The following items do not comply with NELAC\TNI standard requirements and must be reported accordingly. A statement, however worded, must be included in the final report indicating that data reported does not fall under the laboratory's NELAC certificate of approval.

- Reporting any compound which is not included in the second source standard (ICV or LCS) does not meet NELAC requirements.
- In addition, a report that contains a compound not included on the NELAC certificate of approval must also include the statement listed above.

15.10.2.1 Modifications

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

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

15.10.3 Surrogates

Only report surrogates at the request of the client. If any surrogate is out of control, all samples results (with surrogates requested) associated with the surrogate must be reported with the appropriate data qualifier.

15.10.4 DoD Requirements

Report results with the appropriate data qualifiers, if samples cannot be reanalyzed for any reason. In addition and at a minimum, the following situations are to be noted in the case narrative: manual integrations, CCV out of control, and results exceeding the calibration range.

16) Quality Control, Acceptance Criteria, and Corrective Action

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

16.2 Corrective actions shall follow the procedures outlined in the *SOP for Nonconformance and Corrective Action*, where appropriate. Any maintenance which may alter instrument sensitivity or linearity must result in the re-analysis of the entire sequence including the tune compound, ICAL or CCV or any batch QC.

16.3 Instrument Performance Check

16.3.1 Acceptance Criteria

Refer to Tables 1 and 1A for the required ion abundance criteria.

16.3.2 Corrective Action Perform auto tune or manual tune and then re-analyze BFB. If the BFB acceptance criteria are still not met, the MS must be retuned according to the procedure outlined in the instrument user's manual. Perform necessary maintenance and make notations in the instrument maintenance logbook. It may be necessary to clean the ion source, or quadrupole, or take other necessary actions to achieve the acceptance criteria. An acceptable tune is required for sample results to be calculated and reported.

16.4 Initial Calibration

16.4.1 Acceptance Criteria Refer to the following acceptance criteria for the initial calibration.

- The RRT for each target compound at each calibration level must be within 0.06RRT units of the mean RRT for the compound.

- The calculated %RSD for the RRF for each compound in the calibration standard must be less than 30% with at most two exceptions up to a limit of 40% (this may not be true for all projects).
- For each Internal Standard the area response (Y) at each calibration level must be within 40% of the mean area response $\overline{Y}$ over the initial calibration range.
- The retention time shift for each of the internal standards at each calibration level must be within 20s of the mean retention time over the initial calibration range for each internal standard.
- All of the following information must be retained to permit reconstruction of the initial instrument calibration: calibration date, test method, instrument, analysis date, analyte identification, analyst's initials, concentration and responses, and response factors.
- All initial instrument calibrations must be verified with an acceptable ICV.

16.4.2 Corrective Action Follow the initial calibration requirements detailed in Section 11.1 for information on re-analyzing or dropping points and the restriction of maintenance performed during the analysis of the initial calibration standards.

If the initial calibration results are outside the established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed, if reanalysis of the samples is not possible, data associated with an unacceptable initial calibration shall be reported as estimated with the appropriate data qualifiers.

16.5 Initial Calibration Verification Standard (ICV)

16.5.1 Acceptance Criteria The percent recovery for each compound in the ICV must be between 70%-130% for all analytes except vinyl acetate, which must be within 50-150%. Exceptions to this allowance for the vinyl acetate recovery are project specific requirements and any DoD type project, which shall adhere to the 70-130% requirement for all target compounds.

16.5.2 Corrective Action If the initial calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column. Perform a new initial calibration if any performed maintenance has altered instrument linearity and/or sensitivity. Perform another initial calibration or if reanalysis is not possible, data associated with an unacceptable ICAL/ICV shall be reported as estimated with the appropriate data qualifiers.

16.6 Continuing Calibration Verification (CCV)

16.6.1 Acceptance Criteria All compounds must be evaluated prior to rounding. The percent difference for each target analyte must be within plus or minus 30% of the initial calibration average RRFs.

16.6.2 Corrective Action If the continuing calibration verification technical acceptance criteria are not met, reanalyze and if it fails again, prepare a new canister and analyze. If the criteria are still not met inspect the system for possible sources of the problem and perform any necessary maintenance and make a notation in the maintenance logbook of any steps taken. It may be necessary to clean the ion source or change the column.

If any corrective action and/or reanalysis fails to produce continuing calibration verification within acceptance criteria (analyzed immediately following the initial failure), then either two consecutive successful verifications must be performed following corrective action or a new initial calibration must be performed; however, refer to 16.5.1 below.

16.6.2.1 Method Reporting Limit Check Standard

If the MRL check standard is unacceptable for any compound (sensitivity; ratio or %D), reanalyze at the same or higher level within the same batch and report data with the CCV flag and case narrative notes accordingly.

16.6.3 *DOD REQUIREMENT:* If a CCV fails, the laboratory must immediately analyze two additional consecutive CCVs (immediately is defined as within one hour).

- Both of these CCVs must meet acceptance criteria in order for samples to be reported without reanalysis
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
- Flagging data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

16.7 Canister Quality Control Check

The actual cleaning procedure, number of cans to select for analysis (to release a cleaning batch) and corrective actions are covered in the *SOP for Cleaning and Certification of Summa Canister and Other Specially Prepared Canisters* and are not covered in this section. However, the procedure for analyzing and certifying a cleaning batch is included. If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, NELAC\TNI, and Department of Defense Quality Systems Manual – DoD QSM, etc.).

16.7.1 Scan Analyses A canister is considered "clean" for normal SCAN analyses if the analysis shows <0.2ppbv of any target analyte (analyte exceptions listed in table below). If a canister passes as a QC canister it meets all of the requirements for a method blank (Method, NELAC\TNI, and Department of Defense Quality Systems Manual - DoD QSM, etc.).

Low Level SCAN Analyses For those analytes with a MRL of 0.1ug/m3, the QC criteria of <MRL is acceptable; otherwise, <0.2ppbV is required (analyte exceptions listed in table below).

SIM Analyses Results <MRL will be acceptable as this complies with the <0.2ppbV method requirement.

ANALYTE EXCEPTION LIST					
Compounds	ppbV	On Column (ng)	Compounds	ppbV	On Column (ng)
Target Analytes	0.2	0.50	Acrylonitrile	0.2	0.43
Chloromethane	0.2	0.41	Acetone	1.5	3.5
1,3-Butadiene	0.2	0.44	Ethanol	1.9	3.5
Acetonitrile	0.2	0.33	Vinyl acetate	0.99	3.5
Acrolein	0.65	1.5	1-Butanol	0.23	0.70
Isopropanol	0.28	0.70	Carbon Disulfide	1.1	3.5
2-Butanone	1.2	3.5			

Document the status of the check in LIMS and return the canister to the canister conditioning room. Additionally, if the check was found to be acceptable, the quantitation report must be kept on file for future reference

16.7.2 Tentatively Identified Compounds (TIC) If the batch of canisters are to be used for tentatively identified compounds (TIC) analysis, any non-target peaks present in the QC check canister analysis must be evaluated and determined to be less than the TIC reporting limit (10% of the internal standard). The concentration is estimated by assuming a RRF of 1.0 and comparing the response of the TIC to the response of the nearest internal standard.

16.8 Method Blank

16.8.1 Acceptance Criteria

- The concentration of a targeted analyte in the blank cannot be at or above the MRL, AND be greater than 1/10 of the amount measured in any associated sample. For any project that requires reported results less than the MRL, all associated measurements found in the MB should result in a qualifier; however, project requirements may differ and must be followed. Refer to DoD requirements listed below.
- The method blank should not contain additional compounds with elution characteristics and mass spectral features that would interfere with identification and measurement of a method analyte.
- For DoD samples, the method blank will be considered to be contaminated if:

  1. The concentration of any target analyte in the blank exceeds 1/2 the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater);
  2. The concentration of any common laboratory contaminant (acetone, ethanol, carbon disulfide, and methylene chloride) in the blank exceeds the reporting limit and is greater than 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater); or
  3. The blank result otherwise affects the samples results as per the test method requirements or the project-specific objectives.

The laboratory shall evaluate whether reprocessing of the samples is necessary based on the above criteria.

16.8.2 Corrective Action If the analyte concentration results in the blank do not meet the acceptance criteria repeat analysis with remaining QC canisters until results are acceptable or prepare a canister per Section 12.7. If the analyte results in the blank still do not meet the acceptance criteria the source of the problem

must be investigated and measures taken to eliminate the source. Each method blank must be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch. Determine whether the contamination is from the instrument or due to contamination in the blank container (if results from the new can are not acceptable then the system is probably contaminated). In all cases, the corrective action (reprocessing or data qualifying codes) must be documented. However, the specific corrective action depends on the type of project the blank is utilized for; therefore, refer (below) to the reporting/reprocessing requirements.

DEPARTMENT OF DEFENSE (DoD) QSM PROJECT: Any sample associated with a blank that fails the criteria shall be reprocessed in the same or subsequent analytical batch, except when the sample analysis resulted in a non-detect. If reanalysis is not performed, the results shall be reported with appropriate data qualifier.

OTHER PROJECT TYPE: Appropriate corrective measures must be taken and documented before sample analysis proceeds. However, if this is not a possibility and the results must be reported follow the reporting requirements stated in Section 18.4.

16.9 Laboratory Control Sample (LCS)

16.9.1 Acceptance Criteria Round all results to the nearest whole number prior to determining if the acceptance criteria have been met. The percent recoveries must be within the laboratory-generated limits and are referenced in the electronic TO-15 Method Manual. However, Arizona requires the percent recovery for each compound in the LCS to be 70%-130% (to match the ICV requirement). Therefore, the ICV exception for vinyl acetate stated in Section 16.5 requires the percent recovery for AZ samples to be 50-150%.

Note: Client project requirements, AFCEE and DoD requirements shall take precedence over the AZ requirement for AZ samples. Meaning if a sample is collected for a DoD project in AZ, DoD requirements specified in this document and the project specific QAPP (if supplied) are to be followed.

DoD Requirement: In the absence of client specified LCS reporting criteria, the LCS control limits outlined in the DoD QSM 5.0 Appendix C tables shall be used when reporting data for DoD projects.

16.9.2 Corrective Action If the LCS criteria are not met, determine whether the cause is instrumentation or the result of a poor injection. If the problem is instrumentation, perform maintenance and if the problem is with the injection re-analyze the LCS. DoD considers the same analyte exceeding the LCS control limits two out of three consecutive LCS to be indicative of non-random behavior; therefore, this trend should be monitored and the appropriate corrective action taken when it occurs.

16.10 Sample Results

16.10.1 Acceptance Criteria

- Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.
- The field sample must be analyzed on a GC/MS system meeting the BFB tuning, initial calibration, initial calibration verification technical acceptance criteria described in this document.

- All target analyte peaks must be within the initial calibration range, diluted or reported with the appropriate data qualifier.

16.10.2 Corrective Action

- If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis as needed.
- If the area for any internal standard changes by more than ±40 percent between the sample and the most recent calibration, check for possible matrix interferences and re-analyze at a greater dilution. If the requirement is still not met and matrix interference is not detected the GC/MS system must be inspected for malfunction and maintenance made where necessary.
- When corrective actions are made, samples analyzed while the instrument was not functioning properly must be re-analyzed or the appropriate data qualifiers must be attached to the results.

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

16.11 Laboratory Duplicate

16.11.1 Acceptance Criteria The relative percent difference must fall within ±25%. This RPD criterion also applies to duplicate laboratory control samples (DLCS).

16.11.2 Corrective Action If the duplicate results do not meet the technical acceptance criteria, perform another duplicate analysis. If the results are still unacceptable and the associated samples are not reanalyzed then all of the sample results in the associated batch must be flagged accordingly.

16.12 Internal Standards

16.12.1 Acceptance Criteria The following acceptance criteria must be applied to each run (except the ICAL – see Section 16.4).

- The area response for each internal standard in the blank must be within ±40 percent of the area response for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).
- The retention time for each internal standard must be within ±0.33 minutes of the retention time for each internal standard in the most recent valid calibration. (CCV or mid-point from the initial calibration, whichever is most current).

16.12.2 Corrective Action

- Internal Standard Responses If the problem is with the instrument, perform maintenance. If the problem is with a sample, check for interferences. If the response is high, it is likely that interference is present. In this case, lower the volume or aliquot of the sample and re-analyze. If the problem persists, report the results with the best quality and qualify the results. If the problem is corrected with the lower volume analysis, report those results.

- Internal Standard Retention Times If the retention time for any internal standard within the sample changes by more than 20 sec from the latest daily calibration or initial calibration mid-point standard, the GC/MS system must be inspected for malfunctions, and maintenance performed as required. Repeat sample analysis where required.

16.13 Surrogates

16.13.1 Acceptance Criteria Since the matrix precludes the use of true surrogates and there is no established method criterion, acceptable surrogate recoveries are based on a fixed window of 70 - 130%. This is the typical requirement from clients. Additionally, these limits are referenced in SW-846 for use as guidance in evaluating recoveries. These limits are sufficient for evaluating the effect indicated for the individual sample results.

16.13.2 Corrective Action Poor surrogate recovery should be followed by re-analyzing a smaller aliquot to mitigate any matrix interferences. Evaluate the out of control surrogate for the effect on individual sample results.

16.14 Method Reporting Limit Check Standard

16.14.1 Acceptance Criteria Per client requirements or if the CCV is biased low for any compound, then evaluate the MRL check standard. Analyte must be detected reliably and identified by the method-specific criteria (i.e, ion confirmation) and produce a signal that is at least 3 times the instrument's noise level (3:1 signal to noise ratio). Also, a percent difference +/-50% is recommended.

16.15 Sample Holding Time Expired

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

17) Data Records Management

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

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

18) Contingencies for Handling Out of Control Data

18.1 The following is specific information on how to report unacceptable data. If the data requires a data qualifier flag, as specified in this SOP, refer to Appendix D of the most recent version of the Quality Assurance Manual for the appropriate data qualifier.

18.2 Initial Calibration and/or Initial Calibration Verification

All results reported with an unacceptable ICAL must be reported as estimated and all data shall be reported using defined qualifiers or flags or explained in the case narrative accordingly.

18.3 Continuing Calibration Verification

All results associated with an unacceptable CCV (other than #1 below) must be reported with the appropriate data qualifier, flag and/or explained in the case narrative.

1. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported without a qualifier.
2. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples with detects, then those detects must be reported with a qualifier, flag and/or explained in the case narrative.
3. If however, the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, and there are associated samples that are non-detects, then those non-detects must be reported with qualifiers, flags and/or explained in the case narrative as having less certainty. However, along with the data qualifiers, the case narrative may include information stating the fact that the results were not significantly affected if:
   a. *An MRL check standard was analyzed and found to be acceptable. The MRL must be the same as that analyzed in the MRL check standard for those analytes that were biased low in the CCV. Adjust MRLs (if required), flag data and state the certainty in the case narrative where the sensitivity of the instrument was demonstrated at the MRL; therefore, results were not significantly affected.*
   b. *With the reporting limit adjusted to the next level in the calibration curve (typically 5 times higher) to prove the nonexistence of a false negative and note procedure in case narrative.*
4. If the acceptance criteria was exceeded (biased high) for the CCV and there were detectable results in a sample, the results may be "qualified" if the results exceeded the regulatory/decision limit (this is to be stated in the case narrative along with the data qualifiers or flags).

18.4 Method Blank

- If an analyte in the blank is found to be out of control and the analyte is also found in associated samples, those sample results shall be "flagged" in the report and the method blank results reported.
- If the analyte is found in the blank but not in the sample then the results for the sample may be reported without a qualifier.

18.5 Laboratory Control Sample

All results associated with an out of control laboratory control sample must be reported with the appropriate data qualifier. An indication of whether the LCS was out high or low should also be included.

18.6 Surrogate

Report sample results with the appropriate data qualifier.

18.7 Laboratory Duplicate

All batch sample results associated with an out of control laboratory duplicate must be flagged with the appropriate data qualifier.

18.8 Internal Standard

All target analytes associated with an out of control internal standard must be flagged with the appropriate data qualifier.

18.9 Estimated Sample Results

18.9.1 Sample Hold Time All occurrences of missed holding times must be included on the final report including those samples received and/or analyzed outside of the specified hold times detailed in this SOP.

18.9.2 Matrix Interference Sample data associated with matrix interference must be flagged with the appropriate data qualifier.

18.9.3 Results Outside Initial Calibration Range All sample results not bracketed by initial calibration standards (within calibration range) must be reported as having less certainty by reporting with the appropriate data qualifier.

19) Method Performance

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

19.2 Method Detection Limit (MDL)

The procedure used to determine the method detection limits are as stated in the *Code of Federal Regulations* (40 CFR 136 Appendix B) as defined in the *SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations are listed in Tables 2 and 2A for both SCAN and SIM modes and were obtained using spiked canisters prepared with humidified zero air, making at least seven replicate measurements of the compounds of interest, computing the standard deviation, and multiplying this value by the appropriate Student's t value for 99 percent confidence. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. All MDLs, regardless of the mode of operation, meet the method performance criteria of <0.5ppbV.

19.3 Accuracy and Precision

Refer to Section 11.4 in the referenced method for information on replicate precision criteria for method performance. Single laboratory accuracy is presented as the second source initial calibration verification standard, which meets the method performance criteria of 30%. Additionally, laboratory generated control limit data for LCSs are presented for the analytes of interest and may be referenced in the electronic TO-15 Method Manual. Refer to Section 11.1.4.2 for the accuracy and precision requirements for concentrations at the LOQ/MRL.

19.4 Selectivity

Mass spectrometry is considered a more definitive identification technique than single specific detectors such as flame ionization detector (FID), electron capture detector (ECD), photoionization detector (PID), or a multidetector arrangement of these (see discussion in Compendium Method TO-14A). The use of both gas chromatographic retention time and the generally unique mass fragmentation patterns reduce the chances for misidentification.

It is necessary to establish that a given GC/MS meets tuning and standard mass spectral abundance criteria prior to initiating any data collection. Upon sample

injection onto the column, the GC/MS system is operated so that the MS scans the atomic mass range from 35 to 300 amu. At least ten scans per eluting chromatographic peak must be acquired. Scanning also allows identification of unknown compounds in the sample by searching through library spectra.

The sample analysis using the GC/MS is based in part on a combination of retention times and relative abundances of selected ions. The retention time of each chromatographic peak should be ±0.10 minutes of the library/reference retention time of the compound. The acceptance level for relative abundance should be set at ±20% of the expected abundance. The data should be manually examined by the analyst to determine the reason for the # flag [(#) = qualifier out of range], if present and whether the compound should be reported as found or if there is matrix interference. A background subtraction may aid in this determination. Manual inspection of the qualitative results should also be performed to verify concentrations outside the expected range.

Specific selectivity information is provided in this section and document (such as relative retention time) as well as in the referenced method. Refer to the method for additional information on selectivity.

- Use NIST Library 98 or newer version
- The *reference spectra updates* must be performed with every new ICAL utilizing the mid-level standard (minimum). If needed, the reference spectra may be updated sooner with the continuing calibration standard.
- *Retention time updates* must be performed using EasyID and not by updating to the method (InitCal \ Update Calibration). Refer to the Help selection of the software.

19.5 Demonstration of Capability

This laboratory has continuously performed this method since before July 1999. Therefore, ongoing demonstration of capable shall be performed and documented; however, the initial demonstration of method capability is not required.

19.6 Proficiency Testing (PT) Program

The laboratory shall participate in an air and emissions PT study for TO-15. The testing shall be performed in accordance with this document and meet the frequency and proficiency requirements detailed in the DoD QSM Version 5.0.

20) Summary of Changes

Table 20.1

Revision Number	Effective Date	Document Editor	Description of Changes
21.0	02/15/14	C. Humphrey W. Ang C. Parnell	Major document format revision. SOP updated using current ALS SOP template. New cover page and footer. Sections reorganized. See list of changes below.

Section 1.1	Added glass bottles to first paragraph
Section 4	Renamed “Health and Safety Warnings”; Previously Sections 6.0 and 15.0
Section 4.1	Updated MSDS to SDS
Section 4.2	Updated Material Safety Data Sheets (MSDS) to Safety Data Sheets (SDS)
Section 5	Renamed “Cautions”; Previously Section 10.0
Section 6	Previously Section 5.0

Section 7	Renamed "Personnel Qualifications and Responsibilities"; Previously Sections 4.0 and 18.0
Section 7.4	Updated QSM version and removed reference to requirement box
Section 8	Renamed "Sample Collection, Handling, and Preservation"; Previously Section 7.0
Section 8.1	Revised to include Bottle Vacs
Section 8.2	Revised to include Bottle Vacs
Section 9	Renamed "Equipment and Supplies"; Previously Section 8.0
Section 10	Renamed "Standards and Reagents"; Previously Section 9.0
Section 10.2.1.3	Updated Manufacturer name
Section 10.2.2	Updated Manufacturer name and revised canister concentrations
Section 10.2.2.1	Updated standard canister concentrations throughout section Updated Note 1
Section 11	Renamed "Method Calibration"; Information previously in Section 11.0
Section 11.1	#13 – updated ICAL point in last bullet
Section 11.1.1	Updated ICAL points and standard concentrations throughout section
Section 12	Renamed "Sample Preparation/Analysis"; Information previously in Section 11.0
Section 12.4.1	Updated Desorb Temperature
Section 12.4.2	Updated Flow Rate
Section 12.9	Updated 1st and 2nd bullet
Section 12.14.1	#2 – Updated LOD spiking criteria to 2-4x the MDL
Section 12.16	Information previously in Section 19.0
Section 13	Renamed "Troubleshooting"; New Section
Section 14	Renamed "Data Acquisition"; Information previously in Sections 11.0 and 13.0
Section 15	Renamed "Calculation and Data Reduction Requirements"; Information previously in Section 13.0
Section 15.9	Revised
Section 16	Renamed "Quality Control, Acceptance Criteria and Corrective Action"; Information previously in Sections 12.0 and 16.0
Section 16.6.3	New Section
Section 16.7.1	Updated last paragraph
Section 16.9.1	Added DoD Requirement section
Section 17	Renamed "Data Records Management"; New Section
Section 18	Renamed "Contingencies for Handling Out of Control Data"; Previously Section 17.0
Section 19	Renamed "Method Performance"; Previously Section 14.0
Section 19.6	Updated DoD QSM version and removed reference to requirement box
Section 20	Renamed "Summary of Changes"
Section 21	Renamed "Reference and Related Documents"
Section 21.7	Updated reference
Section 22	Renamed "Appendix"
Tables 2, 2A	Updated
Tables 3, 3A	Updated
Tables 4, 4A	Updated
Attachment 3	Revised

21) References and Related Documents

21.1 EPA Method TO-14A, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.2 EPA Method TO-15, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA/625/R-96/010b, U.S. Environmental Protection Agency, Research Triangle Park, NC, January 1997.

21.3 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, January 1999.

21.4 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Addendum, January 17, 2002.

21.5 National Environmental Laboratory Accreditation Conference, *2003 NELAC Standard*, June 5, 2003, EPA 600/R-04/003 and 2009 TNI Standards.

21.6 *Preparation of Gas Phase Standards for Ambient Air Analysis,* Tekmar-DOHRMANN Application Note, Spring 96, Vol. 6.5.

21.7 *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 5.0, July 2013.

21.8 Arizona Administrative Code, Title 9. Health Services, Chapter 14. Department of Health Services Laboratories, December 31, 2006.

21.9 Florida Department of Environmental Protection, Chapter 62-160.

21.10 Minnesota Department of Health, 4740.2065, *Standard Operating Procedures*, Statutory Authority: MS s 144.97; 144.98; History: 31 SR 446, Posted: October 09, 2006, Revised April 16, 2010.

22) Appendix

22.1 Tables

Table 1: Instrument Tune Check Ion Abundance Criteria (TO-15)

Table 1A: Instrument Tune Check Ion Abundance Criteria (TO-14A)

Table 2: Volatile Organic Compounds, EPA Compendium Method TO-15 (SCAN)

Table 2A: Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)

Table 3: Standard Concentrations (SCAN) (Primary Sources)

Table 3A: Standard Concentrations (SIM) (Primary Sources)

Table 4: Standard Concentrations (SCAN) (Secondary Sources)

Table 4A: Standard Concentrations (SIM) (Secondary Sources)

22.2 Attachments

Attachment 1 – Training Plan

Attachment 2 – Initial Calibration Checklist

Attachment 3 – Daily QC and Sample Review Checklists

Attachment 4 – State and Project Specific Requirements

TABLE 1

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-15

Mass	Ion Abundance Criteria[1]
50	8.0 to 40.0 percent of m/e 95
75	30.0 to 66.0 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5.0 to 9.0 Percent of m/e 95
173	Less than 2.0 Percent of m/e 174
174	50.0 to 120.0 Percent of m/e 95
175	4.0 to 9.0 Percent of m/e 174
176	93.0 to 101.0 Percent of m/e 174
177	5.0 to 9.0 Percent of m/e 176

[1] All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120 percent that of m/z 95.

TABLE 1A

Required BFB Key Ions and
Ion Abundance Criteria for Method TO-14A

Mass	Ion Abundance Criteria
50	15 to 40 percent of m/e 95
75	30 to 60 percent of m/e 95
95	Base Peak, 100 Percent Relative Abundance
96	5 to 9 Percent of m/e 95
173	Less than 2 Percent of m/e 174
174	>50 Percent of m/e 95
175	5 to 9 Percent of m/e 174
176	>95 and <101 Percent of m/e 174
177	5 to 9 Percent of m/e 176

Note: The criteria listed in Tables 1 and 1A shall be met or exceeded in order for EPA Compendium Methods TO-15 or TO-14A to be referenced.

TABLE 2 - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Bromochloromethane (IS1)	74-97-5	-	-	130	128, 132	-	-	-
Propene	115-07-1	42.08	NA	42	39,41	0.50	0.14	IS1
Dichlorodifluoromethane (CFC 12)	75-71-8	120.9	1.329	85	87, 101, 103	0.50	0.17	IS1
Chloromethane	74-87-3	50.49	0.911	50	52	0.50	0.15	IS1
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	76-14-2	170.9	1.455	135	137	0.50	0.19	IS1
Vinyl Chloride	75-01-4	62.50	0.9106	62	64	0.50	0.17	IS1
1,3-Butadiene	106-99-0	54.09	0.6149	54	39, 53	0.50	0.22	IS1
Bromomethane	74-83-9	94.94	1.6755	94	96	0.50	0.19	IS1
Chloroethane	75-00-3	64.52	0.8902	64	66	0.50	0.17	IS1
Ethanol	64-17-5	46.07	0.7893	45	46	5.0	0.80	IS1
Acetonitrile	75-05-8	41.05	0.7857	41	40	0.50	0.18	IS1
Acrolein	107-02-8	56.06	0.840	56	55	2.0	0.17	IS1
Acetone	67-64-1	58.08	0.7845	58	43	5.0	0.77	IS1
Trichlorofluoromethane	75-69-4	137.4	NA	101	103	0.50	0.17	IS1
Isopropyl Alcohol	67-63-0	60.10	0.7809	45	43	5.0	0.42	IS1
Acrylonitrile	107-13-1	53.06	0.8060	53	52	0.50	0.17	IS1
1,1-Dichloroethene	75-35-4	96.94	1.213	96	61	0.50	0.17	IS1
tert-Butanol	75-65-0	74.12	0.7887	59	57,41,43	1.0	0.33	IS1
Methylene Chloride	75-09-2	84.94	1.3266	84	49	0.50	0.17	IS1
Allyl Chloride	107-05-1	76.53	0.9376	41	76	0.50	0.16	IS1
Trichlorotrifluoroethane	76-13-1	187.38	1.5635	151	101	0.50	0.17	IS1

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Carbon Disulfide	75-15-0	76.14	1.2632	76	78	5.0	0.15	IS1
trans-1,2-Dichloroethene	156-60-5	96.94	1.2565	61	96	0.50	0.19	IS1
1,1-Dichloroethane	75-34-3	98.96	1.1757	63	65	0.50	0.16	IS1
Methyl tert-Butyl Ether	1634-04-4	88.15	0.7402	73	57	0.50	0.17	IS1
Vinyl Acetate	108-05-4	86.09	0.9317	86	43	5.0	0.65	IS1
2-Butanone (MEK)	78-93-3	72.11	0.7999	72	43	5.0	0.21	IS1
cis-1,2-Dichloroethene	156-59-2	96.94	1.2837	61	96	0.50	0.16	IS1
Diisopropyl Ether	108-20-3	102.18	0.7241	87	45,59,43	0.50	0.19	IS1
Ethyl Acetate	141-78-6	88.106	0.9003	61	70	1.0	0.35	IS1
n-Hexane	110-54-3	86.18	0.6548	57	86	0.50	0.15	IS1
Chloroform	67-66-3	119.4	1.4832	83	85	0.50	0.17	IS1
1,2-Dichloroethane-d4(S)	17060-07-0	-	-	65	67	-	-	IS1
Tetrahydrofuran	109-99-9	72.11	0.8892	72	71,42	0.50	0.20	IS1
Ethyl tert-Butyl Ether	637-92-3	102.176	0.7519	87	59,57	0.50	0.18	IS1
1,2-Dichloroethane	107-06-2	98.96	1.2351	62	64	0.50	0.16	IS1
1,4-Difluorobenzene(IS2)	540-36-3	-	-	114	88	-	-	-
1,1,1-Trichloroethane	71-55-6	133.4	1.3390	97	99, 61	0.50	0.17	IS2
Isopropyl acetate	108-21-4	102.13	0.8718	61	87,43	1.0	0.32	IS2
1-Butanol	71-36-3	74.1224	0.8098	56	41	1.0	0.48	IS2
Benzene	71-43-2	78.11	0.8765	78	77	0.50	0.16	IS2
Carbon Tetrachloride	56-23-5	153.8	1.5940	117	119	0.50	0.15	IS2

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Cyclohexane	110-82-7	84.16	0.7739	84	69,56	1.0	0.29	IS2
tert-Amyl Methyl Ether	994-05-8	102.176	0.7703	73	87,55,43	0.50	0.15	IS2
1,2-Dichloropropane	78-87-5	113	1.1560	63	62	0.50	0.16	IS2
Bromodichloromethane	75-27-4	163.8	1.980	83	85	0.50	0.15	IS2
Trichloroethene	79-01-6	131.4	1.4642	130	132	0.50	0.14	IS2
1,4-Dioxane	123-91-1	88.11	1.0337	88	58	0.50	0.16	IS2
Isooctane	540-84-1	114.23	0.6877	57	41	0.50	0.15	IS2
Methyl Methacrylate	80-62-6	100.12	0.944	100	69	1.0	0.31	IS2
n-Heptane	142-82-5	100.2	0.6837	71	57,100	0.50	0.17	IS2
cis-1,3-Dichloropropene	10061-01-5	111	1.224	75	77	0.50	0.14	IS2
4-Methyl-2-Pentanone	108-10-1	100.2	0.7965	58	85	0.50	0.16	IS2
trans-1,3-Dichloropropene	10061-02-6	111	1.217	75	77	0.50	0.16	IS2
1,1,2-Trichloroethane	79-00-5	133.4	1.4397	97	83	0.50	0.16	IS2
Chlorobenzene-d5(IS3)	3114-55-4	-	-	82	117	-	-	-
Toluene-d8(S)	2037-26-5	-	-	98	100	-	-	IS3
Toluene	108-88-3	92.14	0.8669	91	92	0.50	0.17	IS3
2-Hexanone	591-78-6	100.16	0.8113	43	58	0.50	0.16	IS3
Dibromochloromethane	124-48-1	208.3	2.451	129	127	0.50	0.16	IS3
1,2-Dibromoethane	106-93-4	187.9	2.1791	107	109	0.50	0.16	IS3
n-Butyl Acetate	123-86-4	116.16	0.8825	43	56, 73	0.50	0.16	IS3
n-Octane	111-65-9	114.23	0.6986	57	114	0.50	0.18	IS3

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	**CAS Number**	**Molecular Weight**	**Density**	**Primary Ion[2]**	**Secondary Ion(s)[2]**	**MRL[3] (µg/m³)**	**MDL[3] (µg/m³)**	**IS[4]**
Tetrachloroethene	127-18-4	165.8	1.6227	166	164	0.50	0.14	IS3
Chlorobenzene	108-90-7	112.6	1.1058	112	114	0.50	0.16	IS3
Ethylbenzene	100-41-4	106.2	0.8670	91	106	0.50	0.16	IS3
m-, p-Xylenes	179601-23-1	106.2	0.8642, 0.8611	91	106	1.0	0.30	IS3
Bromoform	75-25-2	252.8	2.899	173	175	0.50	0.15	IS3
Styrene	100-42-5	104.1	0.9060	104	78, 103	0.50	0.15	IS3
o-Xylene	95-47-6	106.2	0.8802	91	106	0.50	0.15	IS3
n-Nonane	111-84-2	128.26	0.7176	43	57, 85	0.50	0.15	IS3
1,1,2,2-Tetrachloroethane	79-34-5	167.9	1.5953	83	85	0.50	0.15	IS3
4-Bromofluorobenzene(S)	460-00-4	-	-	174	176	-	-	IS3
Cumene	98-82-8	120.2	0.8618	105	120	0.50	0.15	IS3
alpha-Pinene	80-56-8	136.24	0.8582	93	77	0.50	0.14	IS3
n-Propylbenzene	103-65-1	120.1938	0.8670	91	120,65	0.50	0.16	IS3
3-Ethyltoluene	620-14-4	120.2	0.8645	105	120	0.50	0.15	IS3
4-Ethyltoluene	622-96-8	120.2	0.8614	105	120	0.50	0.16	IS3
1,3,5-Trimethylbenzene	108-67-8	120.2	0.8652	105	120	0.50	0.16	IS3
alpha-Methylstyrene	98-83-9	118.19	0.9106	118	103,117	0.50	0.15	IS3
2-Ethyltoluene	611-14-3	120.2	0.8807	105	120	0.50	0.15	IS3
1,2,4-Trimethylbenzene	95-63-6	120.2	0.8758	105	120	0.50	0.15	IS3
n-Decane	124-18-5	142.28	0.7300	57	71,85	0.50	0.16	IS3
Benzyl Chloride	100-44-7	126.59	1.1004	91	126	0.50	0.11	IS3

TABLE 2 (Continued) - VOLATILE ORGANIC COMPOUNDS, EPA COMPENDIUM METHOD TO-15 (SCAN)								
Compound[1]	CAS Number	Molecular Weight	Density	Primary Ion[2]	Secondary Ion(s)[2]	MRL[3] (µg/m³)	MDL[3] (µg/m³)	IS[4]
1,3-Dichlorobenzene	541-73-1	147	1.2884	146	148	0.50	0.15	IS3
1,4-Dichlorobenzene	106-46-7	147	1.2475	146	148	0.50	0.14	IS3
sec-Butylbenzene	135-98-8	134.2206	0.8601	105	134,91	0.50	0.16	IS3
p-Isopropyltoluene	99-87-6	134.2206	0.8573	119	134,91	0.50	0.15	IS3
1,2,3-Trimethylbenzene	526-73-8	120.1938	0.8944	105	120	0.50	0.15	IS3
1,2-Dichlorobenzene	95-50-1	147	1.3059	146	148	0.50	0.15	IS3
d-Limonene	5989-27-5	136.24	0.8402	68	93	0.50	0.14	IS3
1,2,Dibromo-3-Chloropropane	96-12-8	236.33	2.093	157	75, 39	0.50	0.099	IS3
n-Undecane	1120-21-4	156.31	0.7402	57	71, 85	0.50	0.15	IS3
1,2,4-Trichlorobenzene	120-82-1	181.5	1.459	180	182, 184	0.50	0.16	IS3
Naphthalene	91-20-3	128.17	1.0253	128	129	0.50	0.18	IS3
n-Dodecane	112-40-3	170.34	0.7487	57	71,85	0.50	0.13	IS3
Hexachlorobutadiene	87-68-3	260.8	1.556	225	227	0.50	0.14	IS3
Cyclohexanone	108-94-1	98.14	0.9478	55	42, 98	0.50	0.12	IS3
tert-Butylbenzene	98-06-6	134.22	0.867	119	134	0.50	0.15	IS3
n-Butylbenzene	104-51-8	134.22	0.867	91	134	0.50	0.17	IS3

(S) = Surrogate (IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3
NA = Not Available

Note 1: Additional compounds may be reported as long as the minimum requirements of this document are met. The compounds listed in this table are reported using TO-15 SCAN. The Selected Ion Monitoring (SIM) compounds are a subset of this list and are included in Table 2A.

Note 2: These are suggested primary and secondary ions. However, any ions in the analyte spectra that are sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 3: The laboratory performs three concentration level analyses (SIM, SCAN and Low Level SCAN). The method reporting limit listed is the standard SCAN limit (at or above lowest concentration in the initial calibration curve), but may change with each new initial calibration performed. Therefore, current reporting limits for the three analysis levels, MRLs in ppbv, and those from the Low Level SCAN should be reviewed in the electronic TO-15 Method Manual.

Note 4: The listing of the internal standard by which the compounds are quantitated is for TO-15 SCAN only. SIM compounds (SCAN subset) and their corresponding ions and internal standards are listed in Table 2A.

Note 5: m/e 101 is ~10% or less of m/e 85 (the base peak) and may not be present for low level results. Retention times must be carefully verified.

Table 2A – Volatile Organic Compounds, EPA Compendium Method TO-15 (SIM)					
Compound	**Primary Ion[1]**	**Secondary Ion[1]**	**MRL[2] (ug/m3)**	**MDL[2] (ug/m3)**	**IS**
Dichlorodifluoromethane	85	87	0.025	0.0070	IS1
Chloromethane	52	50	0.025	0.0092	IS1
Vinyl Chloride	62	64	0.025	0.0068	IS1
1,3-Butadiene	54	39	0.025	0.0060	IS1
Bromomethane	94	96	0.025	0.0067	IS1
Chloroethane	64	66	0.025	0.0065	IS1
Acetone*	58	43	0.50	NA	IS1
Freon 11	101	103	0.025	0.0062	IS1
1,1-Dichloroethene	96	98,61	0.025	0.0066	IS1
Methylene Chloride	84	49	0.10	0.0078	IS1
Trichlorotrifluoroethane	151	153	0.025	0.0067	IS1
trans-1,2-Dichloroethene	96	98,61	0.025	0.0064	IS1
1,1-Dichloroethane	63	65	0.025	0.0068	IS1
Methyl tert-Butyl Ether*	73	57	0.025	0.0048	IS1
cis-1,2-Dichloroethene	96	98,61	0.025	0.0061	IS1
Chloroform	83	85	0.10	0.0064	IS1
1,2-Dichloroethane	62	64	0.025	0.0069	IS1
1,1,1-Trichloroethane	97	99	0.025	0.0057	IS1
Benzene	78	77	0.075	0.0074	IS1
Carbon Tetrachloride	117	119	0.025	0.0059	IS1
1,2-Dichloropropane	63	62,76	0.025	0.0066	IS2
Bromodichloromethane	83	85	0.025	0.0081	IS2
Trichloroethene	130	132	0.025	0.0072	IS2
1,4-Dioxane*	88	58	0.10	0.0058	IS2
cis-1,3-Dichloropropene	75	77,39	0.025	0.0066	IS2
trans-1,3-Dichloropropene	75	77,39	0.025	0.0072	IS2
1,1,2-Trichloroethane	83	97,61	0.10	0.0073	IS2
Toluene	91	92	0.10	0.0072	IS2
1,2-Dibromoethane	107	109	0.025	0.0095	IS2
Tetrachloroethene	166	164	0.025	0.0074	IS2
Chlorobenzene	112	114	0.10	0.0082	IS3
Ethylbenzene	91	106	0.10	0.0085	IS3
m-&-p-Xylene	91	106	0.10	0.014	IS3
o-Xylene	91	106	0.10	0.0092	IS3
1,1,2,2-Tetrachloroethane	83	85	0.025	0.0071	IS3
1,3-Dichlorobenzene	146	148	0.025	0.0090	IS3
1,4-Dichlorobenzene	146	148	0.025	0.0083	IS3
1,2-Dichlorobenzene	146	148	0.025	0.0079	IS3
1,2-Dibromo-3-chloropropane	157	75	0.10	0.0075	IS3
1,2,4-Trichlorobenzene	182	184	0.025	0.0096	IS3
Naphthalene	128	129	0.10	0.0085	IS3
Hexachlorobutadiene*	225	227	0.025	0.0064	IS3

* Reported upon request NA = Not Available
(IS1) = Internal Standard 1 (IS2) = Internal Standard 2 (IS3) = Internal Standard 3

Note 1: These are suggested primary and secondary ions. However, any ions in the analyte spectra that is sufficient enough in response to reach the desired reporting limit and having a limited amount of interference, is acceptable for both the primary and secondary ion selection. Analyst experience should be utilized in determining appropriate ions.

Note 2: The method reporting limit listed is the standard SIM limit (lowest concentration in the initial calibration curve; must be higher than MDL), but may change with each new initial calibration performed. Therefore, current reporting limits should be reviewed. MDLs in ppbV may be reviewed in the electronic TO-15 Method Manual.

Table 3
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
Bromochloromethane (IS1)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Propene	0.0784	0.196	0.392	0.98	4.90	24.50	49.0	98
Dichlorodifluoromethane (CFC 12)	0.0800	0.200	0.400	1.00	5.00	25.00	50.0	100
Chloromethane	0.0768	0.192	0.384	0.96	4.80	24.00	48.0	96
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	0.0800	0.200	0.400	1.00	5.00	25.00	50.0	100
Vinyl Chloride	0.0776	0.194	0.388	0.97	4.85	24.25	48.5	97
1,3-Butadiene	0.0944	0.236	0.472	1.18	5.90	29.50	59.0	118
Bromomethane	0.0776	0.194	0.388	0.97	4.85	24.25	48.5	97
Chloroethane	0.0776	0.194	0.388	0.97	4.85	24.25	48.5	97
Ethanol	0.4040	1.010	2.020	5.05	25.25	126.25	252.5	505
Acetonitrile	0.0792	0.198	0.396	0.99	4.95	24.75	49.5	99
Acrolein	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
Acetone	0.4232	1.058	2.116	5.29	26.45	132.25	264.5	529
Trichlorofluoromethane	0.0792	0.198	0.396	0.99	4.95	24.75	49.5	99
Isopropyl Alcohol	0.1672	0.418	0.836	2.09	10.45	52.25	104.5	209
Acrylonitrile	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
1,1-Dichloroethene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
tert-Butanol	0.1664	0.416	0.832	2.08	10.40	52.00	104.0	208
Methylene Chloride	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
Allyl Chloride	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
Trichlorotrifluoroethane	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
Carbon Disulfide	0.0784	0.196	0.392	0.98	4.90	24.50	49.0	98
trans-1,2-Dichloroethene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
1,1-Dichloroethane	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
Methyl tert-Butyl Ether	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
Vinyl Acetate	0.4080	1.020	2.040	5.10	25.50	127.50	255.0	510
2-Butanone (MEK)	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
cis-1,2-Dichloroethene	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
Diisopropyl Ether	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Ethyl Acetate	0.1672	0.418	0.836	2.09	10.45	52.25	104.5	209
n-Hexane	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
Chloroform	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
1,2-Dichloroethane-d4 (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Tetrahydrofuran	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Ethyl tert-Butyl Ether	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
1,2-Dichloroethane	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
1,4-Difluorobenzene(IS2)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
1,1,1-Trichloroethane	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
Isopropyl acetate	0.1744	0.436	0.872	2.18	10.90	54.50	109.0	218
1-Butanol	0.1752	0.438	0.876	2.19	10.95	54.75	109.5	219

Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
Benzene	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
Carbon Tetrachloride	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
Cyclohexane	0.1648	0.412	0.824	2.06	10.30	51.50	103.0	206
tert-Amyl Methyl Ether	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
1,2-Dichloropropane	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
Bromodichloromethane	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
Trichloroethene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
1,4-Dioxane	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Isooctane	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
Methyl Methacrylate	0.1664	0.416	0.832	2.08	10.40	52.00	104.0	208
n-Heptane	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
cis-1,3-Dichloropropene	0.0816	0.204	0.408	1.02	5.10	25.50	51.0	102
4-Methyl-2-Pentanone	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
trans-1,3-Dichloropropene	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
1,1,2-Trichloroethane	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
Chlorobenzene-d5 (IS3)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Toluene-d8 (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Toluene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
2-Hexanone	0.0888	0.222	0.444	1.11	5.55	27.75	55.5	111
Dibromochloromethane	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
1,2-Dibromoethane	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
n-Butyl Acetate	0.0896	0.224	0.448	1.12	5.60	28.00	56.0	112
n-Octane	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
Tetrachloroethene	0.0784	0.196	0.392	0.98	4.90	24.50	49.0	98
Chlorobenzene	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
Ethylbenzene	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
m- & p-Xylene	0.1680	0.420	0.840	2.10	10.50	52.50	105.0	210
Bromoform	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
Styrene	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
o-Xylene	0.0824	0.206	0.412	1.03	5.15	25.75	51.5	103
n-Nonane	0.0816	0.204	0.408	1.02	5.10	25.50	51.0	102
1,1,2,2-Tetrachloroethane	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
4-Bromofluorobenzene (S)	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**	**25.0**
Cumene	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
alpha-Pinene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
n-Propylbenzene	0.0800	0.200	0.400	1.00	5.00	25.00	50.0	100
3-Ethyltoluene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
4-Ethyltoluene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
1,3,5-Trimethylbenzene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
alpha-Methylstyrene	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
2-Ethyltoluene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
1,2,4-Trimethylbenzene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105

Table 3 - Continued
Standard Concentrations (SCAN) (Primary Sources)[1]

Compound Name	0.08ng	0.2ng	0.4ng	1.0ng	5.0ng	25ng	50ng	100ng
n-Decane	0.0816	0.204	0.408	1.02	5.10	25.50	51.0	102
Benzyl Chloride	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
1,3-Dichlorobenzene	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110
1,4-Dichlorobenzene	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
sec-Butylbenzene	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
p-Isopropyltoluene	0.0816	0.204	0.408	1.02	5.10	25.50	51.0	102
1,2,3-Trimethylbenzene	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
1,2-Dichlorobenzene	0.0864	0.216	0.432	1.08	5.40	27.00	54.0	108
d-Limonene	0.0840	0.210	0.420	1.05	5.25	26.25	52.5	105
1,2-Dibromo-3-Chloropropane	0.0848	0.212	0.424	1.06	5.30	26.50	53.0	106
n-Undecane	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
1,2,4-Trichlorobenzene	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Naphthalene	0.0816	0.204	0.408	1.02	5.10	25.50	51.0	102
n-Dodecane	0.0832	0.208	0.416	1.04	5.20	26.00	52.0	104
Hexachlorobutadiene	0.0872	0.218	0.436	1.09	5.45	27.25	54.5	109
Methacrylonitrile	0.0808	0.202	0.404	1.01	5.05	25.25	50.5	101
Cyclohexanone	0.0896	0.224	0.448	1.12	5.60	28.00	56.0	112
tert-Butylbenzene	0.0856	0.214	0.428	1.07	5.35	26.75	53.5	107
n-Butylbenzene	0.0880	0.220	0.440	1.10	5.50	27.50	55.0	110

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 3A - Standard Concentrations (SIM) (Primary Sources)[1]

Compound Name	10pg	20pg	50pg	100pg	500pg	1000pg	2500pg	10,000pg	20,000pg	50,000pg
Freon-12	10.00	20.00	50.00	100.0	500	1000	2500	10000	20000	50000
Chloromethane	9.60	19.20	48.00	96.0	480	960	2400	9600	19200	48000
Vinyl Chloride	9.70	19.40	48.50	97.0	485	970	2425	9700	19400	48500
1,3-Butadiene	11.80	23.60	59.00	118.0	590	1180	2950	11800	23600	59000
Bromomethane	9.70	19.40	48.50	97.0	485	970	2425	9700	19400	48500
Chloroethane	9.70	19.40	48.50	97.0	485	970	2425	9700	19400	48500
Acetone*	52.90	105.80	264.50	529.0	2645	5290	13225	52900	105800	264500
Freon-11	9.90	19.80	49.50	99.0	495	990	2475	9900	19800	49500
1,1-Dichloroethene	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
Methylene Chloride	10.10	20.20	50.50	101.0	505	1010	2525	10100	20200	50500
Freon-113	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
trans-1,2-Dichloroethene	10.40	20.80	52.00	104.0	520	1040	2600	10400	20800	52000
1,1-Dichloroethane	10.40	20.80	52.00	104.0	520	1040	2600	10400	20800	52000
Methyl tert-Butyl Ether*	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
cis-1,2-Dichloroethene	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
Chloroform	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
1,2-Dichloroethane	10.50	21.00	52.50	105.0	525	1050	2625	10500	21000	52500
1,1,1-Trichloroethane	10.30	20.60	51.50	103.0	515	1030	2575	10300	20600	51500
Benzene	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
Carbon Tetrachloride	10.40	20.80	52.00	104.0	520	1040	2600	10400	20800	52000
1,2-Dichloropropane	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
Trichloroethene	10.40	20.80	52.00	104.0	520	1040	2600	10400	20800	52000
Bromodichloromethane	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
1,4-Dioxane*	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
cis-1,3-Dichloropropene	10.20	20.40	51.00	102.0	510	1020	2550	10200	20400	51000
trans-1,3-Dichloropropene	10.30	20.60	51.50	103.0	515	1030	2575	10300	20600	51500
1,1,2-Trichloroethane	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
Toluene	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
1,2-Dibromoethane	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
Tetrachloroethene	9.80	19.60	49.00	98.0	490	980	2450	9800	19600	49000
Chlorobenzene	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
Ethylbenzene	10.70	21.40	53.50	107.0	535	1070	2675	10700	21400	53500
m,p-Xylenes	21.00	42.00	105.00	210.0	1050	2100	5250	21000	42000	105000
o-Xylene	10.30	20.60	51.50	103.0	515	1030	2575	10300	20600	51500
1,1,2,2-Tetrachloroethane	10.10	20.20	50.50	101.0	505	1010	2525	10100	20200	50500
1,3-Dichlorobenzene	11.00	22.00	55.00	110.0	550	1100	2750	11000	22000	55000
1,4-Dichlorobenzene	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
1,2-Dichlorobenzene	10.80	21.60	54.00	108.0	540	1080	2700	10800	21600	54000
1,2-Dibromo-3-chloropropane	10.60	21.20	53.00	106.0	530	1060	2650	10600	21200	53000
1,2,4-Trichlorobenzene	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500
Naphthalene	10.20	20.40	51.00	102.0	510	1020	2550	10200	20400	51000
Hexachloro-1,3-butadiene*	10.90	21.80	54.50	109.0	545	1090	2725	10900	21800	54500

*Reported upon request.

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 4 - Standard Concentrations (SCAN) (Secondary Sources)[1]

Compound Name	25ng	Compound Name	25ng	Compound Name	25ng
Bromochloromethane (IS1)	**25.0**	1,1,1-Trichloroethane	25.75	alpha-Pinene	26.00
Propene	25.00	Isopropyl acetate	54.50	n-Propylbenzene	25.25
Dichlorodifluoromethane (CFC 12)	25.50	1-Butanol	54.75	3-Ethyltoluene	26.50
Chloromethane	24.75	Benzene	27.50	4-Ethyltoluene	26.50
1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)	25.50	Carbon Tetrachloride	26.25	1,3,5-Trimethylbenzene	26.50
Vinyl Chloride	25.00	Cyclohexane	52.50	alpha-Methylstyrene	26.00
1,3-Butadiene	26.50	tert-Amyl Methyl Ether	26.25	2-Ethyltoluene	26.25
Bromomethane	25.00	1,2-Dichloropropane	26.50	1,2,4-Trimethylbenzene	26.50
Chloroethane	25.25	Bromodichloromethane	26.75	n-Decane	25.75
Ethanol	127.75	Trichloroethene	26.00	Benzyl Chloride	27.75
Acetonitrile	25.25	1,4-Dioxane	27.25	1,3-Dichlorobenzene	27.75
Acrolein	26.75	Isooctane	26.00	1,4-Dichlorobenzene	26.75
Acetone	134.50	Methyl Methacrylate	52.50	sec-Butylbenzene	27.00
Trichlorofluoromethane	24.75	n-Heptane	26.75	p-Isopropyltoluene	25.25
Isopropyl Alcohol	52.50	cis-1,3-Dichloropropene	28.25	1,2,3-Trimethylbenzene	26.50
Acrylonitrile	26.00	4-Methyl-2-Pentanone	27.25	1,2-Dichlorobenzene	27.25
1,1-Dichloroethene	26.75	trans-1,3-Dichloropropene	27.00	d-Limonene	26.25
tert-Butanol	52.25	1,1,2-Trichloroethane	26.25	1,2-Dibromo-3-Chloropropane	26.50
Methylene Chloride	27.00	**Chlorobenzene-d5 (IS3)**	**25.0**	n-Undecane	25.75
Allyl Chloride	27.00	**Toluene-d8 (S)**	**25.0**	1,2,4-Trichlorobenzene	27.50
Trichlorotrifluoroethane	26.75	Toluene	26.25	Naphthalene	25.50
Carbon Disulfide	24.50	2-Hexanone	27.75	n-Dodecane	26.00
trans-1,2-Dichloroethene	26.50	Dibromochloromethane	27.50	Hexachlorobutadiene	27.25
1,1-Dichloroethane	26.00	1,2-Dibromoethane	27.00	Methacrylonitrile	26.00
Methyl tert-Butyl Ether	26.50	Butyl Acetate	28.00	Cyclohexanone	27.50
Vinyl Acetate	128.00	n-Octane	26.00	tert-Butylbenzene	26.75
2-Butanone (MEK)	27.25	Tetrachloroethene	24.50	n-Butylbenzene	27.50
cis-1,2-Dichloroethene	26.75	Chlorobenzene	27.00		
Diisopropyl Ether	27.25	Ethylbenzene	26.50		
Ethyl Acetate	53.25	m- & p-Xylene	52.50		
n-Hexane	26.25	Bromoform	27.00		
Chloroform	26.75	Styrene	27.25		
1,2-Dichloroethane-d4 (S)	**25.0**	o-Xylene	25.75		
Tetrahydrofuran	25.75	n-Nonane	25.75		
Ethyl tert-Butyl Ether	26.50	1,1,2,2-Tetrachloroethane	25.25		
1,2-Dichloroethane	26.25	**4-Bromofluorobenzene (S)**	**25.0**		
1,4-Difluorobenzene(IS2)	**25.0**	Cumene	25.50		

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Table 4A – ICV/LCS Standard Concentrations (SIM) (Secondary Sources)[1]

Compound Name	500pg
Freon-12	510
Chloromethane	495
Vinyl Chloride	500
1,3-Butadiene	530
Bromomethane	500
Chloroethane	505
Acetone*	2690
Freon-11	495
1,1-Dichloroethene	535
Methylene Chloride	540
Freon-113	535
trans-1,2-Dichloroethene	530
1,1-Dichloroethane	520
Methyl tert-Butyl Ether*	530
cis-1,2-Dichloroethene	535
Chloroform	535
1,2-Dichloroethane	525
1,1,1-Trichloroethane	515
Benzene	550
Carbon Tetrachloride	525
1,2-Dichloropropane	530
Trichloroethene	520
Bromodichloromethane	535
1,4-Dioxane*	545
cis-1,3-Dichloropropene	565
trans-1,3-Dichloropropene	540
1,1,2-Trichloroethane	525
Toluene	525
1,2-Dibromoethane	540
Tetrachloroethene	490
Chlorobenzene	540
Ethylbenzene	530
m,p-Xylenes	1050
o-Xylene	515
1,1,2,2-Tetrachloroethane	505
1,3-Dichlorobenzene	555
1,4-Dichlorobenzene	535
1,2-Dichlorobenzene	545
1,2-Dibromo-3-chloropropane	530
1,2,4-Trichlorobenzene	550
Naphthalene	510
Hexachloro-1,3-butadiene*	545

*Report upon request

Note 1: The concentrations detailed in this table may change with each standard purchased or internally prepared. Refer to the appropriate initial calibration file, where necessary for the corresponding concentrations.

Attachment 1

Training Plan

Training Plan for Analysis of VOCs by GC/MS

Trainee _____ Trainer _____ Instrument _____ Training Completion Date _____

1. Read SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____

2. Read Methods TO-14A & TO-15A *Training Duration* _____ Trainer ____ Trainee ____ Date _____

3. Demonstrated understanding of the scientific basis of the analysis Trainer ____ Trainee ____ Date _____
   Whole air sample preconcentration techniques
   Gas chromatography *Training Duration* _____
   Mass spectrometry

4. Demonstrated familiarity with related SOPs Trainer ____ Trainee ____ Date _____
   SOP for Batches and Sequences; Rev. ___
   SOP for Making Entries onto Analytical Records; Rev. ___ *Training Duration* _____
   SOP for Manual Integration Policy; Rev. ____
   SOP for Significant Figures; Rev. ____
   SOP for Nonconformance and Corrective Action; Rev. ____
   SOP for Performing MDL Studies and Establishing Limits of Detection and Quantitation; Rev. ____
   SOP for Cleaning and Certification of Summa Canisters; Rev. ____

5. Observe performance of SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant introduction (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

6. Perform SOP with supervision *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant use (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)

7. Independent performance of the SOP *Training Duration* _____ Trainer ____ Trainee ____ Date _____
   ___sample preparation/dilution and sample loading and analysis
   ___analytical sequence setup
   ___standard preparation
   ___BFB tuning evaluation
   ___initial calibration (model, calculations, manual integrations)/initial calibration verification
   ___manual integrations
   ___continuing calibration verification
   ___EnviroQuant proficiency (recognizing saturation and sensitivity issues)
   ___data reduction and reporting including reporting req. for various agencies, autotexts, documentation
   ___canister and bag handling (including leakers)
   ___initial demonstration of competency (4 Laboratory Control Samples)

8. Instrument operation and maintenance Trainer ____ Trainee ____ Date _____
   ___autosampler *Training Duration* _____
   ___GC and capillary column installation *Training Duration* _____
   ___mass spectrometer *Training Duration* _____
   ___data system *Training Duration* _____

Attachment 2

Initial Calibration Checklist

Initial Calibration Review Checklist - EPA Compendium Method TO-15

ICAL Date:_____ ICAL ID:_____ LIMS ICAL ID:_____

Instrument: ☐ MS3 ☐ MS7 ☐ MS8 ☐ MS9 ☐ MS11 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan Scan Low Level (0.1ng): ☐ Yes ☐ No

Analyst		Reviewer
☐	1. Is the required documentation in the ICAL file?	☐
	☐ BFB Tune analysis Report	☐
	☐ Calibration Status Report (aka Calibration History)	☐
	☐ Response Factor Report/Percent RSD	☐
	☐ Quantitation Report for each calibration standard (including manual integration documentation)	☐
	☐ ICV Quantitation Report	☐
	☐ TO-15 Standard Calculation Spreadsheet	☐
☐	2. Was the ICAL performed continuously (not interrupted for maintenance or sample analysis)?	☐
☐	3. Have all the calibration standards been analyzed within 24 hours of each other?	☐
☐	4. Does the BFB tune check standard analysis at the start meet the tune criteria?	☐
☐	5. Are all the analytes in the blank analysis <MRL?	☐
☐	6. Does each analyte's ICAL include a minimum of 5 concentrations at 5 consecutive levels?	☐
☐	7. Were the standards analyzed from low concentration to high concentration?	☐
☐	8. For each analyte, are there no levels skipped?	☐
☐	9. For each analyte, is there only one value used for each calibration level?	☐
☐	10. For each analyte, is the lowest standard's concentration at or below the analyte's MRL?	☐
☐	11. For each analyte, is the corresponding signal to noise ratio at least 3:1 at the lowest point on the curve?	☐
☐	12. For each analyte, are the corresponding upper levels free from saturation?	☐
☐	13. If a calibration level is dropped, are all the responses for each target analyte dropped and is the information noted in the ICAL explaining the reason?	☐
☐	14. Is the average RSD ≤30% for all analytes, with no more than two exceptions ≤40%?	☐
☐	15. Is the response Y at each calibration level within 40% of the mean area response over the initial calibration range for each internal standard?	☐
☐	16. Percent recovery for each analyte in the ICV 70%-130% (50-150% for VA, unless AFCEE or DoD)?	☐
☐	17. Was the RRT for each target compound at each calibration level within 0.06RRT units of the mean RRT for the compound?	☐
☐	18. Is the retention time shift for each of the internal standards at each calibration level within 20s of the mean retention time over the initial calibration range for each standard?	☐
☐	19. If there are any manual integrations, are they performed correctly according to the corresponding SOP? If so, initial and date the appropriate pages.	☐
☐	20. Is the ICAL good at 0.5ng (or 0.1ng)–100ng (Scan) or 10-20000pg (SIM) for all compounds? ☐ Yes ☐ No If not, note exceptions and the corresponding MRLs below – *Specify applicable range...*	☐
☐	21. Are ALL of the peak selections for each analyte correct according to retention time (all RTs must be checked by both the initial and peer reviewer)?	☐

COMMENTS:

Analyst:_____ Date: _____

Secondary Reviewer: _____ Date: _____

Attachment 3

Daily QC and Sample Review Checklists

EPA Compendium Method TO-15 - Daily QC Review Checklist

(Note exceptions in Comments Section and include Analysis Observations / Case Narrative Summary Form as appropriate)

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____

Instrument: ☐ MS3 ☐ MS7 ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst		Reviewer
☐	1. Is the required documentation present? ☐ CORRECT BFB Tune analysis Report ☐ CCV analysis Quantitation Report & %D Report ☐ LCS analysis Quantitation Report ☐ MB analysis Quantitation Report	☐
☐	2. BFB **tune** check standard analysis meet the tune criteria for the method indicated above?	☐
☐	3. Analyses within the tune's **24-hr window or** ☐ **Client's 12hr window requirement**?	☐
☐	4. Does the **CCV** have a difference ≤30% for all analytes? [Note <u>all</u> outliers biased high and/or low]	☐
☐	5. All **IS** retention times within 20 seconds of the CCV RT or the RT from the midpoint (ICAL)?	☐
☐	6. All **IS** responses within ±40% of CCV or the midpoint in the ICAL?	☐
☐	7. All **surrogate** recoveries (in CCVs, MB, LCSs, etc.) within acceptance limits (70%-130%)	☐
☐	8. All analytes in the **MB** <MRL? (DoD <1/2MRL, except Acetone, MeCl2, EtOH, Carbon Disulfide)?	☐
☐	9. **LCS** %R within the lab control limits for all analytes except AZ samples (70%-130%, VA 50%-150%)?	☐
☐	10. All analytes in the **Lab Duplicate / DLCS** within ±25% or the client specified limits?	☐

COMMENTS:

☐ LIMS Run Approval ☐ LIMS Supervisor Approval

Analyst: _____ Secondary Reviewer: _____

Date: _____ Date: _____

EPA Compendium Method TO-15 - Sample Review Checklist

(Note exceptions in Comments Section and include Analysis Observations / Case Narrative Summary Form as appropriate)

Method: ☐ EPA TO-15 ☐ EPA TO-14A **Analysis Date:** _____ **Project #:** _____

Instrument: ☐ MS3 ☐ MS7 ☐ MS8 ☐ MS9 ☐ MS13 ☐ MS16 ☐ MS19 ☐ MS21

Mode: ☐ SIM ☐ Scan **Scan Low Level (0.1ng):** ☐ Yes ☐ No **DOD:** ☐ Yes ☐ No

Analyst		Reviewer
☐	1. All analyte hits in the samples within the **calibration range** and/or noted?	☐
☐	2. All **peak integrations** acceptable?	☐
☐	3. All **manual integrations** flagged and documented?	☐
☐	4. Have **Q values** been verified for each peak?	☐
☐	6. All **calculations** correct?	☐
☐	7. Has the analyst initialed and dated each **quantitation report**?	☐
☐	8. For **TICs** are the relative intensity and other requirements met?	☐
☐	9. **Auto report** correct?	☐
☐	10. **MRL** = _____ ☐ ng ☐ pg (ethanol, acetone, vinyl acetate = 5.0ng)	☐
☐	11. Pressurized with **Helium**? Is the worksheet completed for all samples?	☐
☐	12. Report to **MDL**? ☐ Yes ☐ No	☐
☐	13. **Global Minimum Detection Limit** = _____ ☐ ng ☐ pg	☐
☐	14. **DOD:** Are **manual integrations** notated in the **case narrative?**	☐

COMMENTS:

☐ LIMS Run Approval

Analyst: _____

Date: _____

☐ LIMS Supervisor Approval

Secondary Reviewer: _____

Date: _____

Attachment 4

State and Project Specific Requirements

Minnesota Requirements	
Item	**Criteria**
Holding Time (HT)	14 days
Tedlar bags	Not allowed for sampling or sample dilution
Canisters and flow controllers	Individually certified Individually leak checked before shipment
	Samples with concentrations outside of the calibration curve will have a zero canister analysis performed to check for carryover. If carryover is detected, system bake out shall be performed and documented. Additionally, in instances where the laboratory has evidence on file that a particular compound when present at a high concentration does not exhibit carry-over, the samples will not be reanalyzed. When samples are analyzed that have a higher concentration than the evidence on file, the above requirements must be followed. Also, samples that have hits below the MRL will not be reanalyzed when analyzed after a sample with concentrations over the calibration range.
Method Reporting Verification Check	Analyze a Method Reporting Verification at the beginning of the sequence prior to analyzing samples. Acceptance criteria ±40%.
Duplicates	10 percent laboratory duplicates
Record retention	MN/NELAC 5 years MPCA (Minnesota Pollution Control Agency) compliant samples 10 years
Tier level	TIII

Arizona Requirements	
Item	**Criteria**
LCS	70-130% (vinyl acetate 50-150%)

Department of Toxic Substances Control (DTSC) Requirements	
Item	**Criteria**
Holding Time (HT)	72 hour hold time for canisters

EPA Region 9 Requirements	
Item	**Criteria**
Holding Time (HT)	14 days

STANDARD OPERATING PROCEDURE
MICROWAVE DIGESTION - AQUEOUS
SW846-3015A

Issue/Implementation Date: 15 December 2013

Last Review Date: 15 December 2013

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

Approved By:

Robert E. Kyer, Prep Supervisor — Date: 12/9/13

Wade T. DeLong, Quality Assurance Officer — Date: 12-10-13

Leslie S. Bucina, Laboratory Manager — Date: 12/10/13

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This procedure utilizes SW-846 Method 3015A and is an acid digestion procedure used to prepare surface water, groundwater, TCLP and mobility procedure extracts, and waste samples that contain suspended solids for analysis by Inductively Coupled Argon Plasma Spectroscopy (ICP) or by ICP-MS. Samples prepared by this method may be analyzed by ICP or ICP-MS for the metals in Table 1.

1.2 For the analysis of dissolved metals, the sample is filtered at the time of collection, prior to acidification with nitric acid.

1.3 Forty milliliters or less of a well shaken sample is transferred to a digestion vessel. Nitric acid is added and in the case of TCLP (SW846 1311) and SW846 6010/EPA 200.7 batches hydrochloric acid is also added. The closed vessels are microwave digested. The cooled digestates are transferred to graduated digestion tubes and brought to final volume. Samples are centrifuged prior to transfer and voluming if necessary.

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.

DI water	Deionized water
HCl	hydrochloric acid
HNO_3	nitric acid
ICP	Inductively Coupled Plasma
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NCR	Nonconformance Report
OES	Optical Emission Spectroscopy
QA/QC	Quality Assurance/Quality Control
RGT	Reagent
SOP	Standard Operating Procedure
STD	Standard
TCLP	Toxicity Characteristic Leachate Procedure

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

2.0 SAFETY PRECAUTIONS

2.1 Safety glasses, gloves and lab coats must always be worn when doing this procedure.

2.2 Always use a fume hood when adding concentrated Nitric acid (HNO_3) or hydrochloric acid (HCl) to the vessels.

2.3 Venting of the vessels must only be done when contents are at room temperature inside a hood with shield lowered to avoid the potential for chemical burns.

3.0 SAMPLE PRESERVATION AND STORAGE

Measurement	Digestion Volume Requirement (mL)	Collection Volume (mL)	Preservative/ Holding Time*
Total	40	600	HNO_3 to pH <2 / 6 months
Dissolved	40	600	Filter on site; HNO_3 to pH <2 / 6 months
Suspended	40	600	Filter on site / 6 months

* Holding time is the storage time allowed between sample collection and analysis when properly preserved and stored.

3.1 All samples must be collected by the use of techniques that prevent contamination and cross-contamination between samples.

3.2 All sample containers must be pre-cleaned. Glass or plastic are both acceptable.

3.3 Total recoverable metals: All samples must be acidified at the time of collection with concentrated HNO_3 (5 mL/L).

3.4 Dissolved metals: All samples must be filtered through a 0.45 micrometer filter and then acidified at the time of collection with concentrated HNO_3 (5 mL/L).

3.5 Aqueous samples must be preserved to pH less than 2 with HNO_3. The pH is checked by the sample receiving department prior to login. For determinations of

dissolved and suspended metals, the sample must be filtered before preservation on site. Samples that are received unpreserved are preserved by the digestion laboratory personnel on site and must sit for 24 hours prior to digestion. The pH determined upon receipt, after adjustment and after 24 hours is recorded in the Laboratory Preservation Log book. If the pH after resting 24 hours is found to be noncompliant, additional acid is added and the sample must sit for another 24 hours prior to a further pH check

4.0 METHOD PERFORMANCE

4.1 For estimated quantitation limit and working linear range, refer to Section 4.0 of Microbac SOPs ME600 and ME700. Method performance data are acquired as per Microbac SOP 45.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Very reactive or volatile materials that create high pressure when heated may cause venting of the vessels with potential loss of sample and analytes. Samples that contain carbonates or other carbon dioxide generating compounds may cause enough pressure to vent the vessel. If this situation is anticipated the analyst may wish to use a smaller amount of sample.

6.0 EQUIPMENT AND SUPPLIES

6.1 Major Instrumentation

- Mars Xpress unit. Microwave unit must provide programmable power with a minimum of 574W and can be programmed to within ± 10W of required power.
- 75 mL Vessels for the Mars Express

6.2 Apparatus or Equipment

- Beckman GS-6 centrifuge or equivalent
- Analytical balance (600g capacity) or equivalent

6.3 Other Supplies

- Graduated digest tubes (50 mL or 100 mL Capacity)

- Quantitative filter paper, Whatman 41 or equivalent
- Volumetric pipettes
- VWR 50 mL disposable centrifuge tubes or equivalent

7.0 STANDARDS AND REAGENTS

Acids used in the preparation of samples must be reagent grade or better. Redistilled acids may be used.

All purchased stock standards and reagents are logged into the LIMS system and assigned certificate of analysis (COA) numbers. All intermediate and working solutions are similarly logged into the LIMS and assigned STD or RGT numbers. Detailed information regarding solution concentrations, aliquot volumes and final volumes and concentrations are included under the STD or RGT number.

7.1 QC-MS-1 Spike – 10 mg/L, As, Al, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Sb,Se, Ag, Tl, U, V and Zn in 2% HNO3 and tr HF – CPI International or equivalent.

7.2 Custom multi-element solution MIC-SPK-1A from Inorganic Ventures or equivalent containing:

250 mg/L:	K, Na
50 mg/L:	Al, Ca, Mg
25 mg/L:	Si
20 mg/L:	Fe
6 mg/L:	Sb
5 mg/L:	Ba, Li, Mo, Ti, V, Zn, Sr
2.5 mg/L:	Cr, Cu, Mn, Ni, Pb, Tl,
2 mg/L:	Ag, As, Se
1 mg/L:	Co
0.25 mg/L:	Be, Cd
10 mg/L:	B

7.3 5 ug/mL: Sn, Zr

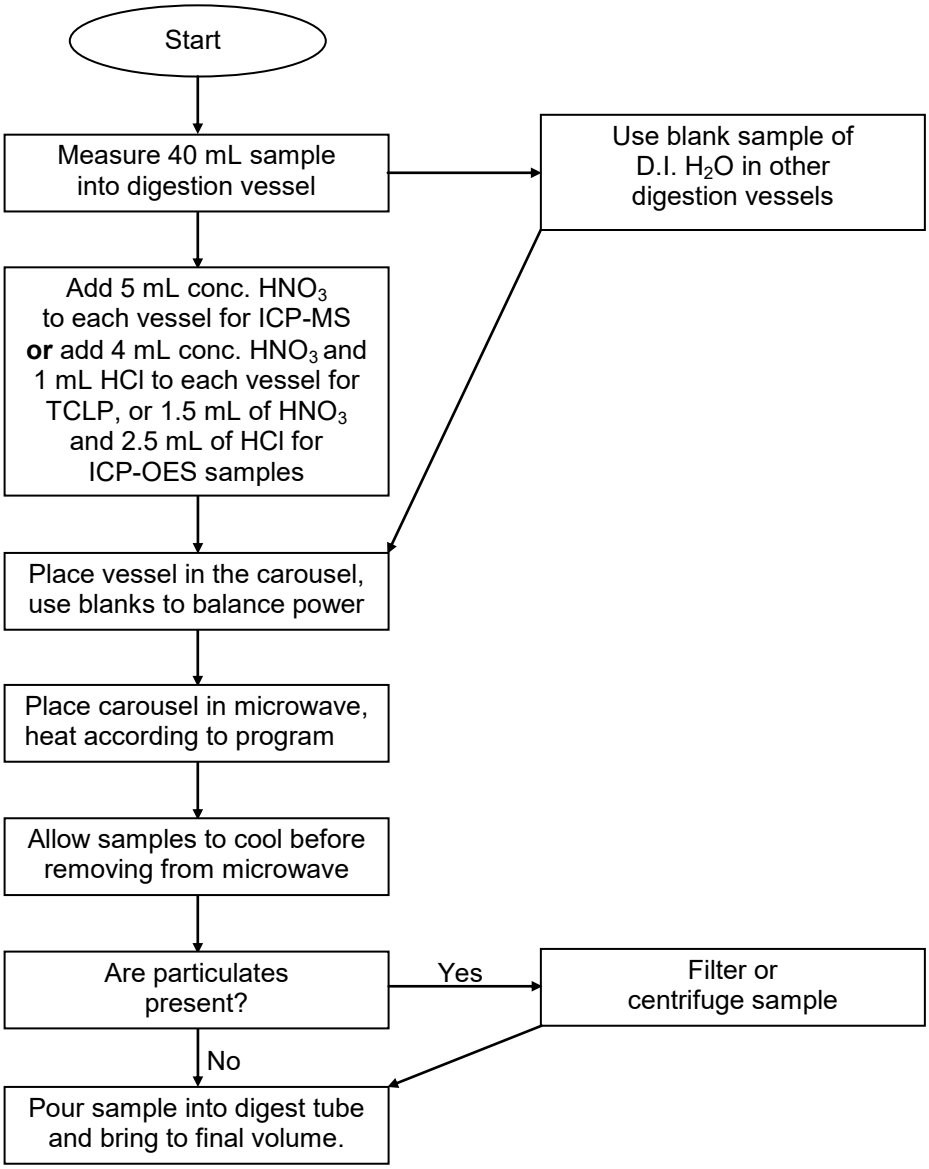
7.4 50 ug/mL: P

7.5 Concentrated HNO_3 (Baker Instra analyzed or equivalent).

7.6 ASTM Type II Water (ASTM D1192): Water must be monitored for impurities.

7.7 Concentrated HCl (Baker Instra analyzed or equivalent)

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES



9.0 SAMPLE PREPARATION

9.1 Samples are shaken to homogenize prior to digestion.

10.0 CALIBRATION PROCEDURES

10.1 The Mars Express is calibrated annually by the Manufacturer.

11.0 ANALYTICAL PROCEDURES

11.1 Choose 20 samples of similar matrix for the preparation batch.

11.2 Measure 40 mL of a well shaken sample into a digest tube and transfer the aliquot into a digestion vessel. Add 5 mL concentrated HNO_3 to each vessel for ICP-MS except TCLP batches and DIG-OES samples. **NOTE**: If a high organic content is suspected, such as in TCLP extracts, 5 mL or less of sample may be used (the difference is made up with DI water). To TCLP extracts add 4 mL concentrated HNO_3 and 1 mL concentrated HCl, and for DIG-OES samples use 1.5 mL of HNO_3, and 2.5 mL of HCl. The blank and LCS are made by using 40 mL of DI water. The LCS and MS/MSD's are spiked with the appropriate spike.

11.2.1 Water batches for ICP-MS analysis are spiked with 0.25 mL of QC-MS-1 spike (7.1) CPI International or equivalent.

11.2.2 TCLP batches are spiked with 5 mL of custom multielement solution MIC-SPK-1A (7.2) Inorganic Ventures or equivalent.

11.2.3 200.7 and 6010 Batches are spiked with 5 mL of custom multielement solution MIC-SPK-1A.

11.2.4 As per methods 200.7 and 200.8, there must be a sample duplicate per every batch of twenty (20) samples or less and a sample MS per every ten (10) samples or less.

11.3 Mars Xpress – Follow these guidlines

11.4 Seal all samples with rubber stoppers and also the vessel cap. Hand tighten vessel cap only.

Weigh vessels and record weight in microwave electronic digestion log template.

Place vessels in carousel making sure they are pushed down completely. Then place the carousel in the microwave oven.

Click the "Run Date/Time:" box in the electronic digestion log template to insert the current date and time.

Select "Load Method" from main menu of microwave.

Select "User Directory".

Select appropriate 3015 method to be used (EPA 3015-8V-Xpress, - 16 press, -24 Xpress).

Push start button.

11.4.1 After the program has finished, allow sample to cool down.

11.4.2 Remove carousel from unit and reweigh vessels and record weight in electronic digestion log template. If the weight has decreased more than 1% from the original weight, discard sample and start the sample over again.

11.4.3 Transfer the solution to a graduated digest tube and bring up to a 50 mL volume with DI H_2O.

Digestion tubes are calibrated volumetrically by lot number when received as per Microbac SOP K0002 "Calibration Procedures".

11.4.4 Centrifugation: Transfer sample in 50 graduated centrifuge tube and place in centrifuge for at least 10 minutes at 2,000 – 3,000 RPM. Slowly decant sample into a clean digest tube and bring up to a 50 mL volume with DI Water. The sample is now ready for analysis. If filtration is needed after centrifugation, this is done by filtering sample through a .45 uL filter into a clean digest tube and bringing up to a 50 mL volume with DI water. The sample is now ready for analysis.

11.5 See Figure 11.1 and 11.2 for examples of the Metals Digest Logs.

12.0 DETAILS OF CALCULATIONS

12.1 Refer to individual methodology.

13.0 QUALITY CONTROL REQUIREMENTS

13.1 Each batch of up to 20 samples requires the following:

13.1.1 Method blank (MB) – a 40 mL aliquot of DI water that is digested with the sample batch.

13.1.2 LCS – a 40 mL aliquot of DI water that is spiked with spiking solution and digested with the sample batch.

13.1.3 Sample duplicate – Batches that include samples for Methods 200.7 and 200.8 will include a sample prepared in duplicate, both carried through the batch digestion.

13.1.4 MS and MSD – two additional aliquots of a sample that are spiked with spiking solution and digested with the sample batch. Sn spike is added to each MS/MSD when Sn is required. Batches that include samples for method 200.8 will include a spiked sample for every ten (10) 200.8 samples.

13.2 Results of the analysis of the QA/QC samples are kept for easy reference.

13.3 All batch QC samples are subjected to exactly the same digestion as those used on actual samples in the digestion batch.

13.4 Control of Nonconforming Data

The laboratory implements general procedures to be followed when departures from documented policies, procedures and quality control have occurred. The policies and procedures are found in Section 13.0 of Microbac SOP LQAP (Laboratory Quality Assurance Program), Microbac SOP GP-CAPA (Corrective Action/Preventive Action: Initiating, Tracking and Monitoring) and Microbac SOP GP-RCA (Root Cause Analysis).

13.4.1 Nonconformances Requiring Corrections

A nonconformance occurs when any aspect of the method QC in an analysis, as outlined in Tables 13.1 and 13.2 of Microbac SOPs ME600E and ME600G and Tables 13.1 and 13.4 of Microbac SOP ME700, does not meet acceptance criteria. When nonconforming data occurs the employee initiates an NCR and proceeds with indicated corrections as per Tables 13.1 and 13.2 of Microbac SOPs ME600E and ME600G and Tables 13.1 and 13.4 of Microbac SOP ME700.

All data shall be scrutinized by the analysts for method and project specific compliance. Checklists are utilized and accompany each data batch Figure 14.1 of Microbac SOPs ME600E, ME600G and ME700. A nonconformance shall be documented in the NCR followed by one or more of the following actions.

- Reanalysis of the sample(s) in question
- Discussion and qualification of data (report and narrative)
- Client notification with approval
- Data qualification (Q-flagging)
- Re-sampling and reanalysis (client decision)

13.4.2 Nonconformances Requiring Corrective Action

Corrective action is required when a nonconformance is recurring, if the correction is ineffective or if the departure is so significant that it negatively effects data quality, sample integrity or customer satisfaction. When an event requiring corrective action is identified, the employee shall initiate a Corrective Action/ Preventive Action form as per Microbac SOP GP-CAPA. The corrective action process includes a root cause analysis as per Microbac SOP GP-RCA, corrections, corrective action(s) and evidence of effectiveness.

13.4.3 Nonconformances Not Requiring Corrections

There are some standard contingencies to the traditional corrections that maybe invoked, provided they comply with the project QAPP requirements. In many situations it may not be necessary to perform sample reanalysis or re-extraction for the following quality control departures, provided they are not a chronic problem or indicative of a trend, and the laboratory provides documentation in the report narrative and project files. In addition, the employee is required to initiate a NCR to record the event.

- An LCS or surrogate recovery exceeds the upper control limit, but the corresponding sample results are non-detect.
- A method blank or calibration blank exceeds the upper limit, but the corresponding sample results are non-detect.
- A method blank or calibration blank exceeds the upper limit, but the corresponding sample results are greater than ten (10) times the level in the blank.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 The appropriate log books must be checked and signed by the department supervisor for completeness.

15.0 PREVENTIVE MAINTENANCE

15.1 Monthly examine the door, door seals and door interlocks to verify they are clean and working properly. Ensure that the door closes securely.

15.2 Clean the inside of the microwave cavity, including the exhaust screen at the back of the cavity, with warm soapy water applied with a soft cloth. Rinse and thoroughly dry all cleaned areas.

15.3 Clean the exhaust outlet by removing the exhaust hose and wiping the space inside the exhaust outlet with a disposable cloth. To clean the exhaust hose, disconnect if from the blower exhaust duct, flush it with water and allow it to dry before reconnecting it to the blower duct.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

16.2 The following are waste streams in the sample preparation area.

16.2.1 Non – Halogenated solvents: Acetone

16.2.2 Solid Waste: Filters, tongue depressors, gloves, any solid material that is a waste after being processed in the lab.

16.2.3 Acid: Dilute acid waste from soak tanks.

16.3 Laboratory policies and procedures for management of hazardous waste are found in Microbac SOP 33, Laboratory Waste Management and the waste management section of the analytical SOPs contain procedures specific to each method. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee received training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

17.0 REFERENCES

17.1 Microwave Assisted Acid Digestion of Aqueous Samples and Extracts, US EPA SW-846 Method 3015, Revision 0, September 1994, EPA Publication SW-846.

17.2 Microwave Assisted Acid Digestion of Aqueous Samples and Extracts, US EPA SW-846, Method 3015A, Revision 1, February 2007, EPA Publication SW-846.

17.3 Microbac SOP LQAP "Laboratory Quality Assurance Plan"

17.4 Microbac SOP 33 "Laboratory Waste Management"

17.5 Microbac SOP 45 "Method Validation Procedures"

17.6 Microbac SOP GP-CAPA "Corrective Action and Preventive Action; Initiating, Tracking, and Monitoring"

17.7 Microbac SOP GP-RCA "Root Cause Analysis"

17.8 Microbac SOP ME600E "Perkin Elmer OPTIMA 4300 Inductively Coupled Plasma Atomic Emission Spectroscopy"

17.9 Microbac SOP ME600G "Thermo iCAP 6000 Series Inductively Coupled Plasma Atomic Emission Spectroscopy"

17.10 Microbac SOP ME700 "Perkin Elmer Elan 6100 Inductively Coupled Plasma/Mass Spectrometer"

17.11 Microbac SOP K0002 "Calibration Procedures"

17.12 40CFR Part136.3 Table II – Required Containers, Preservation Techniques, and Holding Times, footnote 19

An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (See footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

Footnote 2 defines immediately as within 15 minutes of collection.

17.13 Microbac SOP TCLP01 "Toxicity Characteristic Leachate Procedure"

Table 1
Method Analytes

Name	Symbol	Cas Number
Aluminum	Al	7429-90-5
Antimony	Sb	7440-36-0
Arsenic	As	7440-38-2
Barium	Ba	7440-39-3
Beryllium	Be	7440-41-7
Boron	B	7440-42-8
Cadmium	Cd	7440-43-9
Calcium	Ca	7440-70-2
Chromium	Cr	7440-47-3
Cobalt	Co	7440-48-4
Copper	Cu	7440-50-8
Iron	Fe	7439-89-6
Lead	Pb	7439-92-1
Lithium	Li	7439-93-2
Magnesium	Mg	7439-95-4
Manganese	Mn	7439-96-5
Molybdenum	Mo	7439-98-7
Nickel	Ni	7440-02-0
Potassium	K	7440-09-7
Selenium	Se	7782-49-2
Silicon	Si	7440-21-3
Silver	Ag	7440-22-4
Sodium	Na	7440-23-5
Strontium	Sr	7440-23-5
Thallium	Tl	7440-28-0
Tin	Sn	7440-31-5
Titanium	Ti	7440-32-6
Vanadium	V	7440-62-2
Zinc	Zn	7440-66-6
Calculated		
Hardness, Calculated (as $CaCO_3$)	$CaCO_3$	72608-12-9
Silica (as SiO_2)	SiO_2	99439-28-8

Figure 11.1

Microbac Laboratories Inc.
Microwave Digestion Log

Workgroup: WG346402
Analyst: VC
Spike Analyst: VC
Run Date: 10/22/2010 06:16
Method: 3015

SOP: ME407 Revison 11
Spike Solution: STD39374
Spike Witness: ERP
Digestion Tubes Lot #: COA14948
HNO3 Lot #: COA14980

	SAMPLE #	Type	Matrix	Initial Amount	Final Volume	Initial Vessel Wt	Final Vessel Wt	Spike Amount	Due Date
1	WG346402-02	BLANK	1	40 mL	100 mL	206.128 g	206.125 g		
2	WG346402-03	LCS	1	40 mL	100 mL	208.537 g	208.534 g	.25 mL	
3	L10100496-09	SAMP	1	40 mL	100 mL	207.184 g	207.171 g		10/25/10
4	L10100496-10	SAMP	1	40 mL	100 mL	206.81 g	206.806 g		10/25/10
5	L10100543-01	SAMP	1	40 mL	100 mL	207.826 g	207.807 g		10/29/10
6	L10100561-01	SAMP	1	40 mL	100 mL	205.445 g	205.434 g		11/03/10
7	L10100561-02	SAMP	1	40 mL	100 mL	207.579 g	207.564 g		11/03/10
8	L10100561-03	SAMP	1	40 mL	100 mL	205.861 g	205.852 g		11/03/10
9	L10100561-04	SAMP	1	40 mL	100 mL	209.453 g	209.442 g		11/03/10
10	L10100561-05	SAMP	1	40 mL	100 mL	207.956 g	207.944 g		11/03/10
11	L10100561-06	SAMP	1	40 mL	100 mL	206.353 g	206.344 g		11/03/10
12	L10100561-07	SAMP	1	40 mL	100 mL	206.21 g	206.198 g		11/03/10
13	L10100561-08	SAMP	1	40 mL	100 mL	207.139 g	207.134 g		11/03/10
14	L10100561-09	SAMP	1	40 mL	100 mL	208.202 g	208.197 g		11/03/10
15	L10100561-10	SAMP	1	40 mL	100 mL	207.766 g	207.763 g		11/03/10
16	L10100567-01	SAMP	1	40 mL	100 mL	205.947 g	205.948 g		11/01/10
17	L10100567-02	SAMP	1	40 mL	100 mL	206.693 g	206.687 g		11/01/10
18	L10100584-01	SAMP	1	40 mL	100 mL	209.577 g	209.568 g		11/04/10
19	L10100584-02	SAMP	1	40 mL	100 mL	206.725 g	206.714 g		11/04/10
20	WG346402-01	REF	1	40 mL	100 mL	206.759 g	206.756 g		
21	L10100584-03	SAMP	1	40 mL	100 mL	206.759 g	206.756 g		11/04/10
22	WG346402-04	MS	1	40 mL	100 mL	206.072 g	206.072 g	.25 mL	
23	WG346402-05	MSD	1	40 mL	100 mL	206.393 g	206.383 g	.25 mL	

Analyst: Vicki Collins

Reviewer: Erin Potter

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Figure 11.2

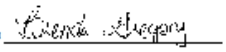
Microbac Laboratories Inc.
Microwave Digestion Log

Workgroup: WG419896
Analyst: REK
Spike Analyst: REK
Run Date: 02/05/2013 05:40
Method: 3015
Balance: BAL016
Instrument: MW-2
Instrument Start: 02/05/2013 05:48

SOP: ME407 Revison 13
Spike Solution: STD56032
Spike Witness: VC
Digestion Tubes Lot #: COA16400
HCL Lot #: COA16547
HNO3 Lot #: COA16631
6010 h2o mdl verfs LOQ L STD55795

	SAMPLE #	Type	Matrix	Initial Amount	Final Volume	Initial Vessel Wt	Final Vessel Wt	Spike Amount	Due Date
1	WG419896-01	BLANK	1	40 mL	50 mL	202.949 g	202.943 g		
2	WG419896-02	LCS	1	40 mL	50 mL	207.665 g	207.643 g	5 mL	
3	L13010746-01	ML01	1	40 mL	50 mL	203.512 g	203.485 g	40 mL	02/08/13
4	L13010746-02	ML02	1	40 mL	50 mL	204.145 g	204.125 g	40 mL	02/08/13
5	L13010746-03	ML03	1	40 mL	50 mL	203.801 g	203.789 g	40 mL	02/08/13
6	L13010746-04	ML04	1	40 mL	50 mL	204.308 g	204.293 g	40 mL	02/08/13
7	L13010746-05	ML05	1	40 mL	50 mL	204.184 g	204.168 g	40 mL	02/08/13
8	L13010746-06	ML06	1	40 mL	50 mL	203.302 g	203.289 g	40 mL	02/08/13
9	L13010746-07	ML07	1	40 mL	50 mL	205.097 g	205.087 g	40 mL	02/08/13

Analyst: 

Reviewer: 

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STANDARD OPERATING PROCEDURE
SAMPLE RECEIVING AND LOGIN

Issue/Implementation Date: 12 November 2013

Last Review Date: 12 November 2013

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

Approved By:

Wade T. DeLong, Quality Assurance Officer — Date: 11-12-13

Leslie S. Bucina, Laboratory Manager — Date: 11/13/13

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This document outlines procedures for sample handling, custody, container preparation, preservation, receipt, inspection/pH, login, Internal Chain of Custody, and storage. The document also addresses Temperature Monitoring and IR gun calibration.

1.2 Except as noted in Section 7.0, the support services supervisor shall have primary responsibility for implementation of these policies and procedures. Section 6.0 describes the duties of the sample custodians. Section 7.0 describes the specific duties of the customer service staff with respect to the procedures for logging samples in KOBRA.

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
IR gun	Infrared Temperature Gun
LIMS	Laboratory Information Management System
LQAP	Laboratory Quality Assurance Plan
SOP	Standard Operating Procedure
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compounds

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

2.0 SAFETY PRECAUTIONS

2.1 Safety glasses with side shields, gloves, and lab coats are worn when samples are being handled. (Safety glasses are worn at all times in the laboratory.) Additional personal safety equipment (respirators and dust masks) are available in the login area.

2.2 Occasionally samples are received broken. When this occurs, the cooler is placed immediately under the hood and samples are removed. When possible, the broken sample is placed in another container. The client is then notified by his/her Service Representative to confirm what appropriate action to take.

2.3 Broken glass is disposed of in the proper containers provided in the laboratory.

2.4 The cooler is cleaned by waste disposal personnel. All contaminated material is disposed of properly using proper waste disposal procedures. See Microbac SOP 33.

2.5 Unknown waste samples that are received broken are placed in a container and taken to sample archive for proper disposal.

2.6 Radiological screening of samples may be performed on coolers received for special projects if required by the project QAPP. The screening is performed by the login personnel and noted in receipt information.

2.7 When highly contaminated samples are received, special precautions are taken. These samples receive special handling and storage and are tagged with a "Special Instructions" sticker. Any comments available are entered into the LIMS for the laboratory.

3.0 EQUIPMENT AND SUPPLIES

3.1 Thermometers

3.2 pH strips: Low range 0.0 - 6.0; High range 7.5 - 14.0

3.3 Hood

3.4 IR Temperature Guns

3.5 Pipets - disposable

3.6 Geiger Counter

3.7 Gloves - disposable

3.8 PDA, Laptop or notebook computer (equipped for bar coding)

4.0 REAGENTS

4.1 20% Nitric Acid = HNO_3 Baker Instra-analyzed, or equivalent; prepared when needed in the metals laboratory. A 1000 mL glass dispenser is used to store and dispense the preservative. When HNO_3 is used as a preservative, a red label with

HNO_3 is placed on the lid of the container. Label container with expiration date of 6 months after date prepared.

4.2 1:1 Hydrochloric Acid = HCL Baker Instra-analyzed, or equivalent; prepared in the metals laboratory. 500 mL of concentrated HCL is added to 400 mL of DI water and diluted to liter. A 1000 mL glass dispenser is used to store and dispense the preservative. When HCL is used as a preservative, a blue label with HCL is placed on the lid of the container. Label container with expiration date of 6 months after date prepared.

4.3 1:1 Sulfuric Acid = H_2SO_4 Baker Instra-analyzed or equivalent; prepared when needed in the conventional lab. 500 mL of concentrated H_2SO_4 is added to 400 mL of DI water and diluted to 1 L. A 4 L glass container is used to refill to 1000 mL glass dispenser. When H_2SO_4 is used as a preservative, a yellow label with H_2SO_4 is placed on the lid of the container. Label the container with expiration date of 6 months after date prepared.

4.4 50% Sodium Hydroxide = NaOH comes in a ready to use solution, or equivalent. No mixing is required. A 250 mL glass container is used to store and dispense this preservative. When NaOH is used as a preservative, a blue label with NaOH is placed on the lid of the container. A sterile pipet is used to add preservative to the container. Use expiration date from the manufacturer.

4.5 Zinc Acetate/Sodium Hydroxide = ZnAc/NaOH Zinc Acetate; prepared when needed in the conventional lab. 88 g of Zinc Acetate dihydrate crystal is dissolved in 200 mL of DI water and diluted to 250 mL. A 250 mL amber glass container is used to store and dispense the preservative. To make ZnAc/NaOH, 2 mL of ZnAc is added to the container using a sterile pipet. Add 2 mL of NaOH with another sterile pipet. Then a gold dot with ZnAc/NaOH is placed on the lid of the container. This preservative is only used for sulfide. Label container with expiration date for 6 months after date prepared.

4.6 Ascorbic Acid = C6H806; comes in a ready to use Ascorbic Acid, Fine Powder. No mixing is required. A 125 mL pre-cleaned wide mouth glass container is used to store and dispense the preservative. A spatula is used to add 25 mg to a 40 mL, pre-cleaned vial with septa lid. Use expiration date from the manufacturer.

4.7 Hexane = 95% ULTRA resi-Analyzed; used in PCB – wipes.

4.8 Methylene Chloride = CH2CL2; ULTRA – Resi – Analyzed used in PAH – wipes.

4.9 Sodium Thiosulfate = Na2S2O3 (tablet) comes ready to use in the 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 a sales/service representative generates a work order (B Number) and the associated kit request/packing list.

5.1.2 The support service staff member uses the packing list to assemble the specified containers for the work order.

5.1.3 Staff member then prints out labels from the LIMS database table that states client, test, preservative and the site where the samples will be taken.

5.1.4 Containers are then set up on the work station table to be labeled and preserved.

5.1.5 Sample containers are then packed in coolers with packing material, packing lists, custody seals and chain of custody forms.

5.1.6 Cooler is then sealed with the custody seal and shipped or delivered to client.

5.1.7 Container Preparation:

All sample containers are received pre-cleaned. Pre-cleaned containers are received in cases that are labeled. Lot numbers and analyte certification records are tracked and filed with start and end dates of the Certificate of Analysis. Sample containers are never reused.

5.2 Sample Containers, Volume and Preservative

Tables 1-6 list the container type, minimum volume, and type of preservative for each analysis parameter or method. Additional instructions are provided below for conventional and wet chemistry parameters.

5.2.1 Acidity (acid) – by itself in 250 mL plastic. No preservative.

5.2.2 Alkalinity (Alk) – by itself in 250 mL plastic. No preservative.

5.2.3 Biochemical Oxygen Demand (BOD) – by itself in 500 mL plastic. No preservative.

5.2.4 Bromide (Br) – by itself in 1000 mL plastic. No preservative.

5.2.5 Cyanide (CN) – by itself in 250 mL plastic with 2 mL of 50% NAOH = pH > 12.

5.2.6 Cyanide, Amenable (CN-A) – by itself in 250 mL plastic or with CN with 2 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 1000 mL plastic. No preservative.

5.2.11 Hardness (Hard) – by itself in 250 mL plastic with 3 mL of 20% HNO_3 pH < 2.

5.2.12 Iodide (I) – by itself in 1000 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) – by itself in 1000 mL plastic with 3 mL plastic of 1:1 H_2SO_4 pH<2.

5.2.15 Oil and Grease (OG) – by itself in 1000 mL glass with 5 mL of 1:1 HCL pH<2.

5.2.16 Phenolics, Total (T-Phen) – by itself in 250 mL amber glass with 1 mL of 1:1 H_2SO_4= pH<2.

5.2.17 Sulfite (SO_3) – by itself in 500 mL plastic. No preservative. Must notify lab.

5.2.18 Settleable Solids (Set –S) – by itself in 1000 mL plastic. No preservative.

5.2.19 Silica Dissolved (Silica) – by itself in 500 mL plastic. No preservative.

5.2.20 Sulfide (S) – by itself in 500 mL plastic with 4 mL of ZnAce/NaOH – pH > 9.

5.2.21 Total Dissolved Solids (TDS) – by itself in a 250 mL plastic or together with TSS in a 500 mL plastic. No preservative.

5.2.22 Total Organic Carbon (TOC) – can be put with other parameters with the same preservative. If by itself it goes into 250 mL plastic with 1 mL 1:1 H_2SO_4. Other requirements for TOC are as follows: TOC-4 used 4x250 mL plastic with 1:1 H_2SO_4, TOC -14 uses 1x250 mL glass with 1:1 H_2SO_4, TOC-44 uses 4x250 mL glass with 1:1 H_2SO_4.

5.2.23 Total Organic Halides (TOX) – by itself in 250 mL amber glass, septa lid with 1 mL of 1:1 H_2SO_4. No headspace in sample. Other requirements for TOX are TOX-4 uses 4x250 mL amber glass, septa lid with 1:1 H_2SO_4. No headspace in sample.

5.2.24 Total Suspended Solids (TSS) – by itself in a 250 mL plastic or together with TDS in a 500 mL plastic. No preservative.

5.3 Volume of Preservative use for Container Sizes:

H_2SO_4:	40 mL	½ mL = pH < 2
	250 mL	1 mL = pH < 2
	500 mL	2 mL = pH < 2
	1000 mL	4 mL = pH < 2
HNO_3:	250 mL	3 mL = pH < 2
	500 mL	5 mL = pH < 2
	1000 mL	10 mL = pH < 2
HCL:	40 mL	3 drops = pH < 2
	1000 mL	5 mL = pH < 2
NAOH:	250 mL	2 mL = pH < 2
MeOH:	40 mL	10 mL used in 5035 Field Prep/tare weight

5.4 Special Procedures for Volatile Organics Analysis (VOA)

5.4.1 General

Samples for VOA analysis should always be collected in separate containers. If this is not possible, the VOA analyses should be performed first or split into a separate VOA container to avoid contamination of the sample with common lab solvents.

5.4.2 Water

All water VOA containers must be filled completely with no headspace/bubbles >6mm. Preservatives, if required, consists 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.

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 QA Manager before proceeding.

5.5.1 Print labels with client's name, parameter and preservative (MeOH).

5.5.2 Go to volatiles lab and get 40 mL VOA vial for prep of 5035 method, place label on bottles.

5.5.3 Using a calibrated volumetric dispenser, add 10 mL of MeOH to each vial. Replace cap and septum.

5.5.4 Determine the tare weight to 0.01 g and record on the vial label (beside the parameter) using a permanent marker.

5.5.5 The kit request will specify the number of containers to be provided. Place the containers in bubble bags and pack them in shipping cooler. Label the outside of the cooler with a label that states "This package conforms to 49CFR – 173.4".

6.0 SAMPLE HANDLING AND CUSTODY PROCEDURES

6.1 This section describes the procedures for receipt, inspection/pH, labeling, storage internal custody and handling of laboratory samples. These are the primary duties and responsibilities of the sample custodian(s) under the direction of the support services supervisor. Many of the processes in this section require knowledge of the Microbac-OVD LIMS (KOBRA and the ROR module) 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 service representative 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. The custodian must open all coolers from AFCEE projects under the fume hood. This practice must be followed for all coolers containing potentially hazardous samples, unknowns, or any broken or leaking containers.

6.3.4 A temperature blank or sample is immediately removed from the cooler and the temperature is taken using the IR Temperature Gun. The temperature is taken by shooting the bottom of a sample. If a cooler 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 Service Representative is notified. The Service Representative 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. 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 (DRO/TPH) and plastic pipets are used to check inorganic parameters. The assistant will verify that the pH is acceptable. The LIMS will create and update the pH record for each sample container checked in the sample delivery group (L#). If there are pH exceptions to the default acceptance criteria, the assistant/analyst will edit the pH data in the LIMS Container Records, and forward the exceptions/discrepancies to the client representative. If additional preservative is added to the container, the amount, concentration, and adjusted pH shall be recorded in the comments field in Container Records table. If the pH is out of range, the sample ID and product of the sample are recorded in the ROR System sample discrepancy section of the Cooler Inspection Form as a discrepancy by login personnel. Prior to the adjusting and storing of the samples, approval must be determined from the client through the Service Representative. 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 and in the LIMS system along with the client instructions. Minimum volume, container type, preservation and hold time are listed in Table 1-6.

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/VOC vials for the acceptable levels of headspace. If any containers contain bubbles larger than

6mm, this information must be noted on the Cooler Inspections Form as a discrepancy.

6.3.10 When all information is correct, the samples are logged into the LIMS system. The LIMS automatically assigns a unique login number. See Sections 7.0 and 8.0 for logging procedures.

6.4 Electronic Checklist – Procedures for Using the PDA / Laptop / Notebook

This section describes the use of a PDA / Laptop or equivalent, (Section 3.8) to automate many of the cooler and sample checking procedures presented in Section 6.3. The system incorporates a barcode reader and a wireless connection to the LIMS, allowing the checklist to be completed in paperless mode. The screen displays an electronic facsimile of Figure 1, and the user is prompted to enter the information in a logical, step-wise manner. Staff must not attempt these procedures until they have been trained in the use of the PDA / Laptop or equivalent.

6.4.1 Turn the unit on and open a new receipt. The system will automatically assign a unique number for later tracing of the cooler and samples to a specific delivery group (B, P and final L number(s)).

6.4.2 Select the mode of delivery by pressing the appropriate radio button.

6.4.3 Go to “Add cooler information”

6.4.4 Scan the barcode that identifies the carrier’s airbill number.

6.4.5 Scan the barcode that identifies the cooler’s tracking number.

6.4.6 Remove the COC(s) and scan or record the COC number(s). (This can also be done later in the sequence if there are multiple COCs.). This scan of the COC will be included in the final report to our customer(s). It is important the scan be an accurate reflection of the COC as received. Therefore, maintenance of the scanning device is critical. No markings must be allowed to be introduced by the scanning process. For example, lines introduced by dust or debris on the scanner’s internal parts are not permitted. Careful and timely maintenance of the scanner is important. See Figure 2 for scanner maintenance and cleaning instructions.

6.4.7 Determine the cooler temperature with the IR Gun and enter temperature.

6.4.8 Complete the remaining checklist items using the keypad.

6.4.9 Note each exception or problem in the discrepancy files.

6.4.10 Some samples will require priority due to short hold time or turn around time. Use the "Priority" drop-down menu to select appropriate priority need. The short hold time may not be readily determined by information on the chain of custody. Tables 1 through 6 list hold times for each analysis. An abbreviated list is presented in Figure 3; this list is posted at the cooler inspection areas.

6.4.11 Proceed as in Section 6.3.5.

6.4.12 When you have finished this process, you may edit any checklist entry, add comments, or address discrepancy details at the login computer keyboard.

6.5 Sample Labeling

Labels for containers and mailing reports are then printed, label bottles, and put mailing report labels along with chain of custody form in folder.

6.6 Sample Storage

Samples are stored according to analyses. In the walk-in cooler (W-1) shelves are designated by departments. These identify the sample storage location for easy retrieval by the laboratory. Volatile samples (V-1) are stored in a separate refrigerator with the exception of waste samples. These are kept in the walk-in cooler or on a holding shelf in archive until analyses can be performed.

6.7 Internal Chain of Custody Procedures

If a client requests samples to be tracked throughout the lab, an ICOC form is generated through a computer program. "ICOC" prints out on the label. This label also contains a unique bar code. The analyst is responsible for scanning out the samples using his/her bar coded badges. The samples are then relinquished by the appropriate personnel in the login area. Other information as to the location of the sample, which department the sample is going to can be scanned from a bar coded template. When the analyst is ready to return the samples, it is scanned back into the computer by the relinquishing employee using his/her bar coded badges, one login personnel's badge and the location to which the sample is being returned (walk-in/archive/disposal). It is not always possible to have a custodian present when samples are removed from storage. The analyst will scan a special barcode designated for all "after hours" removal of samples from the login area storage unit.

6.8 Sample Disposal

After all analyses have been completed, the sample custodians will move the sample residuals to the archive storage units, where they will remain for the time specified in the client agreement. At regular intervals the sample custodian or other trained staff member will dispose of aqueous, solid, and organic-matrix samples in accordance with Microbac SOP 33 (Laboratory Waste Management). Alternatively, the laboratory may return selected sample residuals to the client.

7.0 OVERVIEW OF LOGGING PROCESS

This section describes the duties of the sales/service team, specifically the customer service representatives and their assistants, as they pertain to logging of project and sample information into the LIMS (KOBRA). These duties are summarized below:

7.1 The sales/service department must enter account and project information into the KOBRA LIMS as a prerequisite to preparing quotations and work-orders or to the actual logging of samples. The LIMS assigns unique serial numbers for each account and for the associate project.

7.2 Quotations (Q number)

Quotations are used to enter special pricing in the LIMS and are often included as part of the sales proposal. The LIMS assigns a unique serial number for each quotation. (Q-number)

7.3 Work-orders (B number)

The service team creates a work order upon new project award, or when sample containers are requested for an existing project. The work order includes sample, matrix and product information in sufficient detail to generate a packing list for sample kits and for the efficient logging of samples once received at the laboratory and to create templates for repetitive sampling events. The LIMS assigns a unique serial number (B number) for each work order.

7.4 Sample Prelogging (P number)

Prelogging 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 prelog 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 prelog 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 Service representative 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 prelog step of the process. The service team is also responsible for communication and resolution of any discrepancies identified at the time of sample receipt and inspection. Other duties of the service teams are presented in Microbac SOP 44 and Microbac SOP MISDATA01.

8.0 DETAILED LOGGING PROCEDURE

8.1 Log into KOBRA.

8.2 Go to Sample Management; Login; Login ID – enter the correct workorder (B number) or template (T number); Check Pre-Login and click OK.

8.3 Check all products in the matrix for each client ID. **DO NOT CHANGE ANY INFORMATION IN THE B# SCREEN.** Check all projects/products, etc. in this screen to make sure they match the chain of custody. **Read Login information in the top right corner for specific instructions.**

8.4 Go to Copy; Copy Template; Source Template B number; Pre-Login; Collect Date (Change date if needed); then click OK.

8.5 The prelog (P number) Screen will come up at this time. This is the screen you edit if necessary.

8.6 Place your cursor on the Client ID. Check the matrix below to see if it is correct against the chain of custody and the bottles lined up. If the matrix matches the chain of custody and the bottles, go back to the client ID.

- You may have to change the client ID to match the bottle or chain of custody ID.
- Tab over to the received date; enter 0 and tab for the current date and add the time received from the printed barcode label that the ROR system generates.
- Tab over to the collect date; the date should be filled in but you will need to add the time. If no time is given, default to 00:01.
- Tab over to the TAT; do not change this number; if the TAT is different from the chain of custody, put this information in the "Prelim" button, not the TAT.
- Tab to the end of the line checking for any errors and also to check comments and QC tags.

8.7 Move your cursor down to the next fraction (line) and start over with these instructions from Section 8.6. When tabbing to the receive and collect dates/times use the F3 key and the dates/times will copy from the above fraction ***or*** you can also "Copy Duplicate Fractions" or "Copy Products" by going to the Copy tab on the Toolbar, and clicking on one or the other. To delete fractions or products, shift F6.

8.8 If nothing else needs added or deleted go to Copy; order fractions; click on the Auto Calc due dates button and the F10 to save. This will automatically fill in the due dates and client dates. Click on the add prods button at the bottom of the page. Be sure to add prods on LAST fraction.

8.9 At this point, your folder which is now a P number needs to go through Peer Review or given to the client representative for review. When the folder is returned to you after review, you may make corrections if needed and then take it to an L number. Be sure to take care of discrepancies if needed.

8.10 In order to take a P number to an L number go to copy; source template P# _____; Login.

8.11 The L number screen will come up at this time. If a green button comes up at the top of the screen, click on it at this time.

8.12 Click on Reports; The L number will come up automatically at this time in the Login Reports screen; go to Login Labels; Printer; Go and your labels will print with

address labels attached. Watch for the ICOC on label. Use pen to color labels for help with archiving.

8.13 Label the bottles and put them away in their appropriate walk-in, refrigerator or freezer.

8.14 Place the client Chain of Custody form, and the extra client address labels in a manila master folder.

8.15 Place the client address label on the folder tab and in the bottom right hand side, on the front of the folder, facing out (sideways).

8.16 If the TAT is less than 7 days or these are short hold, make a note on the outside of the master folder to help in prioritizing review.

8.17 Folders are then taken to the Service team.

9.0 CALIBRATION AND QUALITY CONTROL

9.1 IR Gun Calibration

9.1.1 The IR Temperature Guns are calibrated once a year by Cole-Parmer and a certificate is supplied and are maintained in the QA office. The guns are calibrated quarterly (every 3 months) by Wet Lab personnel.

9.1.2 The calibration of the IR temperature guns are checked once daily, in the morning before used against the ROR system Quality Control probe thermometer in the 1005/WI walk-in cooler by Support Service personnel and readings are recorded in the temperature log books. The IR gun must read within .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.

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 Service Representative is notified so that the client may be advised that their samples were received with the temperature out of

range. All sample containers in a cooler with a temperature exceeding the 6ºC when required or received frozen must be listed in the Discrepancy section of the inspection report. 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 Service Representative is notified. A temperature is taken for each cooler.

9.3 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 and charting. 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.

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
Boron	20	P	Cool, ≤ 6° C	6 Months
Bromide	1000	P, G	Cool, ≤ 6° C	28 Days
BTU	10	P, G	Cool, ≤ 6° C	---
Formaldehyde	20	G	Cool, ≤ 6° C	7 Days
Chloride	25	P, G	Cool, ≤ 6° C	28 Days
Chloride, Total Residual	100	P, G	Cool, ≤ 6° C	6 Hours
Cyanide (midi)	50	P, G	Cool, ≤ 6° C, NaOH, pH>12	14 Days
Cyanide, Amenable to Chlorination (midi)	100	P,G	Cool, ≤ 6° C, NaOH, pH>12	14 Days
Chemical Oxygen Demand	25	G	Cool, ≤ 6° C, H_2SO_4, pH<2	28 Days
Color, Platinum-Cobalt	50	P, G	Cool, ≤ 6° C	48 Hours
Coliform, Fecal	120	P, G	Cool, < 10° C, $Na_2S_2O_3$	6 Hours
Fecal Streptococcus	100	P, G	Cool, < 10° C, $Na_2S_2O_3$	6 Hours
Coliform, Total	100	P, G	Cool, < 10° C, $Na_2S_2O_3$	6 Hours
Specific Conductance	100	P, G	Cool, ≤ 6° C	28 Days
Corrosivity	500	P, G	Cool, ≤ 6° C	---
Corrosivity (pH)	40	P, G	Cool, ≤ 6° C	---
Chromium, Trivalent	200	P, G	Cool, ≤ 6° C	---
Chromium, Hexavalent	150	P, G	Cool, ≤ 6° C	24 Hours
Dissolved Oxygen	300	G	Cool, ≤ 6° C	6 Hours
Fluoride	25	P, G	Cool, ≤ 6° C	28 Days
Ignitability	75	P, G	Cool, ≤ 6° C	---
Fluoride, Total (Distilled/Non-Distilled)	200	P, G	Cool, ≤ 6° C	28 Days
Hardness	100	P, G	Cool, ≤ 6° C, HNO3, pH<2	6 Months
Iodide	400	P, G	Cool, ≤ 6° C	24 Hours
Surfactants (MBAS)	100	P, G	Cool, ≤ 6° C	48 Hours
Coliform Fecal (MPN)	100	P, G	Cool, < 10° C, $Na_2S_2O_3$	6 Hours
Nitrogen, Ammonia (Distilled/Non-Distilled)	100	P, G	Cool, ≤ 6° C, H_2SO_4, pH<2	28 Days
Nitrogen, Nitrite	50	P, G	Cool, ≤ 6° C	48 Hours
Nitrogen, Nitrate	75	P, G	Cool, ≤ 6° C	48 Hours
Nitrogen, Nitrate-Nitrite	25	P, G	Cool, ≤ 6° C, H_2SO_4, pH<2	28 Days
Nitrogen, Organic	100	P, G	Cool, ≤ 6° C, H_2SO_4, pH<2	28 Days
Threshold Odor	1000	G	Cool, ≤ 6° C	48 Hours
Oil and Grease	1000	G	Cool, ≤ 6° C, HCl, pH<2	28 Days

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

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

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Phenolics, Total	100	Amber, G	Cool, ≤ 6° C, H_2SO_4, pH< 2	28 Days
Phosphorus, Total	50	P, G	Cool, ≤ 6° C, H_2SO_4, pH< 2	28 Days
pH Lab	40	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	24 Hours
Settleable Solids	1000	P, G	Cool, ≤ 6° C	48 Hours
Silica, Dissolved	100	P	Cool, ≤ 6° C	28 Days
Sulfate	100	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	200	P, G	Cool, ≤ 6° C, Zinc Acetate, NaOH, pH> 9	7 Days
Total Dissolved Solids	50	P, G	Cool, ≤ 6° C	7 Days
Total Suspended Solids	200	P, G	Cool, ≤ 6° C	7 Days
Turbidity	50	P, G	Cool, ≤ 6° C	48 Hours
Volatile Dissolved Solids	50	P, G	Cool, ≤ 6° C	7 Days
Total Volatile Solids	50	P, G	Cool, ≤ 6° C	7 Days
Volatile Suspended Solids	200	P, G	Cool, ≤ 6° C	7 Days

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

Table 2
Sample Containers, Preservation and Hold Times
VOLATILE ORGANICS (VOA) - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Gasoline Range Organics	40 mL	G, Septa Caps	Cool, ≤ 6° C, HCl, pH< 2	14 Days
Volatile Aromatics	40 mL	G, Septa Caps	Cool, ≤ 6° C, HCl, pH< 2	14 Days
Volatile Organics (VOA)	40 mL	G, Septa Caps	Cool, ≤ 6° C, HCl, pH< 2	14 Days
VOA – Method 624	40 mL	G, Septa Caps	Cool, ≤ 6° C	7 Days
VOA – Method 624 (chlorinated) *	40 mL	G, Septa Caps	Cool, ≤ 6° C, $Na_2S_2O_3$	7 Days

*Provided upon client request when samples contain chlorine.

Table 3
Sample Containers, Preservation and Hold Times
SEMIVOLATILE ORGANICS - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Diesel Range Organics	1000 mL	G	Cool, ≤ 6° C	7 Days
Pesticides/PCBs	1000 mL	G	Cool, ≤ 6° C	7 Days
Polyaromatic Hydrocarbons	1000 mL	G	Cool, ≤ 6° C	7 Days
Herbicides	1000 mL	G	Cool, ≤ 6° C	7 Days
Semivolatile Organics	1000 mL	G	Cool, ≤ 6° C	7 Days

1. P = Polyethylene (preferred when acceptable)
2. G = Borosilicate glass with Teflon lined cap
3. For more current list of method/preservatives, see KOBRA tables, containers and product

Table 4
Sample Containers, Preservation and Hold Times
METALS - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
All Metals (26)	500 mL	P, G	HNO_3, pH< 2	6 Months*
Mercury	50 mL	P, G	HNO_3, pH< 2, ≤ 6° C	28 Days
Furnace Metals	100 mL	P, G	HNO_3, pH< 2	6 Months

Table 5
Sample Containers, Preservation and Hold Times
TCLP - WATER

PARAMETER	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
TCLP Volatiles	100 mL	G	Cool, ≤ 6° C	14 Days
TCLP Semi-Volatiles	100 mL	G	Cool, ≤ 6° C	14 Days
TCLP Pesticides	100 mL	G	Cool, ≤ 6° C	14 Days
TCLP Herbicides	100 mL	G	Cool, ≤ 6° C	14 Days
TCLP Metals	100 mL	P, G	Cool, ≤ 6° C	6 Months*

* For (1) TCLP parameter 100 mL required; for full TCLP (2) 1000g
* Mercury is 28 days

NOTE:
1. P = Polyethylene (preferred when acceptable)
2. G = Borosilicate glass with Teflon lined cap
3. Triple the volumes above for MS/MSD samples

Table 6
Sample Containers, Preservation and Hold Times
SOIL

METHOD	MINIMUM VOLUME	CONTAINER TYPE	PRESERVATIVE	HOLD TIME
Coliform Fecal	1g	G	Cool, < 10° C	6 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
TPH	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	---

Figure 1

Microbac

Lab Report #: L13080127
Lab Project #: ZZB4.033
Project Name:
Lab Contact: Stephanie Mossburg

Record of Sample Receipt and Inspection

Comments/Discrepancies

This is the record of the shipment conditions and the inspection records for the samples received and reported as a sample delivery group (SDG). All of the samples were inspected and observed to conform to our receipt policies, except as noted below.

There were no discrepancies.

Discrepancy	Resolution

Coolers

Cooler #	Temperature Gun	Temperature	COC #	Airbill #	Temp Required?
00110685	H	5.0			X

Inspection Checklist

#	Question	Result
1	Were shipping coolers sealed?	NA
2	Were custody seals intact?	NA
3	Were cooler temperatures in range of 0-6?	Yes
4	Was ice present?	Yes
5	Were COC's received/information complete/signed and dated?	Yes
6	Were sample containers intact and match COC?	Yes
7	Were sample labels intact and match COC?	Yes
8	Were the correct containers and volumes received?	Yes
9	Were samples received within EPA hold times?	Yes
10	Were correct preservatives used? (water only)	Yes
11	Were pH ranges acceptable? (voa's excluded)	NA
12	Were VOA samples free of headspace (less than 6mm)?	NA

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Figure 2

MICROBAC®

Ohio Valley Division

Cleaning the Scanner

Cleaning procedure

To clean all the parts listed below use glass cleaning wipes that are individually packaged. Using the wipe remove the debris. It may be necessary to remove large chunks of toner material and dust from the rollers. If so gently scrape the material off being careful not to damage the rollers.

Parts that require cleaning

1) Rollers

- Open the top compartment of the paper feed for the scanner.
- Locate the white colored rollers on the top of the open compartment door.
- These should be completely white without any black streaks or spots on them.
- Toner can buildup here and cause lines to form on the scans.

2) Glass

- Open the door to the scanning bed.
- On the left side of the scanning bed there is a narrow strip of glass.
- This glass must be completely free of debris / dust / detritus.
- Imperfections on the glass will cause lines to appear on the scan.

3) Additional Preventative Maintenance

If you look on the top of the lid above the glass you cleaned in step 2 you will see a thin white strip of material. This material must remain free of dust and imperfections. The smallest bit of dust can be picked up by the scanning bed and turned into lines on the scan.

A can of compressed air should be used periodically to clean out paper dust and debris. IT or office staff maintain a supply for use by the lab.

If you require assistance please contact the IT support staff.

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Figure 3

Ohio Valley Division MICROBAC®

Water
Short Hold Times

6 Hour	24 Hour	48 Hour
Fecal / MPN	CR-6	BOD
Cl-TRC	Iodide	Color
DO-L	-	NO3
pH-L	-	NO2
SO3	-	PO4 (orthophosphate)
-	-	Set-5
-	-	ODOR
-	-	Turbidity
-	-	MBAS
-	-	9056 (NO3, NO2 or PO4)
-	-	300

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	-

Last Revised: 08/23/2013 Document Control Form #1329

STANDARD OPERATING PROCEDURE FOR SW-846 METHODS 5030 and 5035 PURGE AND TRAP FOR VOLATILE ORGANICS

Issue/Implementation Date: 15 May 2010

Last Review Date: 15 June 2011

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

Approved by:

Mike Albertson, Volatiles Supervisor — Date 6/20/11

Leslie S. Bucina, Quality Assurance Officer — Date 6/20/11

David E. Vandenberg, Managing Director — Date 6/27/11

Document Control # 286

Issued to: Document Master File

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This SOP describes purge-and-trap procedures for the analysis of volatile organic compounds by USEPA Methods 5030B, 5030C, and 5035/5035A.

1.2 5030B and 5030C describe sample preparation and extraction for the analysis of VOCs in aqueous, water miscible, low level soil, high concentration soil, and waste samples. This method is applicable to gas chromatographic methods 8260B, 624, 602, and 8015B/C/D.

1.3 5035/5035A describes a "closed-system" purge-and-trap process for the analysis of VOCs in solid matrices and mid-level preparation and analysis. This method is designed for collection and analysis of samples containing low levels of VOCs. This method is applicable to gas chromatographic methods 8260B, 8015B, and 8015D.

Sample collection and preparation for low level 5035/5035A analysis can be divided into two classes: field and laboratory. The field procedure utilizes a hermetically sealed sample vial, the seal of which is never broken from time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling and analysis are negligible. This procedure, however, is not performed within the laboratory therefore the procedure will only be briefly outlined in the SOP. The lab procedure involves the preparation and "in-laboratory" preservation of samples transported to the laboratory via specialized sampling tools. Once a sample arrives at the laboratory, the samples are transferred to sample containers and preserved to allow adequate time for sample analysis.

The closed-system purge-and-trap equipment used for low concentration samples is not appropriate for soil samples preserved in methanol. Such samples must be analyzed using methods 5030B and 5030C.

1.4 Methods 5030B, 5030C, and 5035/5035A can be used for most VOCs that have boiling points below 200° C and are insoluble or slightly soluble in water. Volatile compounds that are water-soluble can also be analyzed however quantitation limits (by GC or GC/MS) are elevated due to poor purging efficiency.

1.5 Definitions and Acronyms

The following is a list of terms, definitions, and acronyms referenced in this SOP that are unique to the method.

DI water De-ionized water

GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometer
HCl	Hydrochloric acid
MS	Mass Spectrometer
MSDS	Material Safety Data Sheet
PPE	Personal protective equipment
QC	Quality control
SOP	Standard Operating Procedure
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound

For a more comprehensive list of common terms and definitions, consult Appendix A in Microbac SOP LQAP.

2.0 SAFETY 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 include lab coats and safety glasses with sideshields.

2.2 **WARNING:** The following volatile analytes have been tentatively classified as known or suspected human or mammalian carcinogens:

benzene
carbon tetrachloride
chloroform
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 must be reduced to the lowest possible level. Procedures involving sample and primary standard preparation should be performed in a fume hood.

2.3 **WARNING:** Sodium bisulfate ($NaHS0_4$) used in the preparation and preservation of sample fractions is known to be a skin irritant. The preservative, sulfuric acid ($pH \leq 2$) is the result of adding water to sodium bisulfate. Sulfuric acid is known to be corrosive.

2.4 MSDSs for each analyte and reagent used within the laboratory are available to all employees. MSDSs should be consulted prior to handling chemicals.

3.0 SAMPLE PRESERVATION AND STORAGE

3.1 Water samples preserved with HCl (pH ≤ 2) must be analyzed within 14 days of collection. Unpreserved water samples (pH > 2) must be analyzed within 7 days of collection. Water samples for acrolein and acrylonitrile should not be preserved with HCl but may be preserved with sodium thiosulfate (pH between 4 and 6). Waste, soil, oil, and sludge samples do not require the addition of preservative for Methods 5030B and 5030C. Waste, soil, oil, and sludge samples have a holding time of 14 days from the date of collection. Samples received and identified as liquid wastes have a holding time of 14 days regardless of solubility. 5030B and 5030C samples are stored at ≤ 6° C. 5035A Samples must be stored at -10° C to -20° C or ≤ 6° C depending on the preservative employed. Temperature logs are kept for each of the storage refrigerators and recorded daily.

3.2 Preservation procedures for 5035A depends on the expected concentration range of the sample. Separate techniques are employed for low or high concentration soil and waste samples. Providing sufficient sample volume is available, three aliquots ("A", "B", and "C") for each sample are preserved within 48 hours of collection and stored in a dedicated sample refrigerator (≤ 6° C) or freezer (-10° C to -20° C) thus achieving a holding time of 14 days. Unpreserved samples have a holding time of 48 hours from collection. The client must be notified if samples are preserved past 48 hours from collection.

3.3 5035A Sample preservation must be performed per the following:

Fractions "A" and "B" must be preserved with water and frozen. These fractions are used for low level analysis. For high concentration analysis, fraction "C" is preserved using methanol. Fraction identification and extraction solvent type are clearly labeled on each vial.

40 mL VOA vials containing the sample aliquots are placed in a sample storage freezer. Frozen samples must be analyzed within 14 days of collection.

3.4 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 5030B, 5030C, and 5035A. Soil samples not utilizing Method 5035A should be collected in 125 mL pre-cleaned glass screw cap jars with teflon-lined lids. High concentration soil samples utilizing method 5035A may also be collected in 60 mL short septa jars with teflon faced silicone septa.

3.5 Upon completion of analysis and 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.

3.6 Sample hold-time is defined as time elapsed from sample collection date and times to sample analysis date and time.

3.7 Refer to Section 9.0 for sample preservation procedures.

4.0 METHOD PERFORMANCE

4.1 Refer to method specific SOP.

5.0 INTERFERENCES AND CORRECTIVE ACTION

5.1 Samples for VOC analysis are susceptible to laboratory contaminants (i.e.: methylene chloride, acetone, n-hexane). To eliminate the potential for interferences from other areas of the laboratory, the Volatiles Laboratory has an independent air intake system and positive air pressure is maintained in the laboratory.

5.2 Samples preserved with HCl or sodium bisulfate may result in the loss of 2-chloroethylvinylether as a target or spiked analyte due to the reactivity of this analyte with the preservatives.

For any fraction "A" that effervesces upon preservation with sodium bisulfate fraction "B" is preserved without sodium bisulfate. Refer to Section 9.0 for more information.

5.3 Soil analyses may result in low internal and/or surrogate standard recovery due to the poor purging efficiencies of some soil matrices. In the event that this occurs, reanalysis of the sample is performed to confirm matrix effects.

5.4 Carry-over contamination may occur when a sample containing low levels of VOCs is analyzed immediately following a sample containing high levels of VOCs. After analysis of a sample containing high concentrations of VOCs, a system check blank may be analyzed to monitor for system contamination. If this occurs during a non-monitored analysis, the sample containing the low concentration VOCs may require reanalysis to confirm results.

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 sample storage refrigerators and freezers to monitor for cross-contamination. Water storage blanks consist of (2) 40 mL VOA vials filled with laboratory grade deionized (DI) water. Soil storage blanks consists of (2) 40 mL VOA vials with 5.00 g of sand and 5 mL of laboratory grade DI water. Storage blanks are logged into the LIMS laboratory account and analyzed after remaining in the storage units for 14 days. Refer to Microbac SOP MSV01 for more details.

5.7 As with any method for VOC analysis, samples may be screened to avoid contamination of the purge-and-trap system. Since the 5035A sample container cannot be opened to remove a sample aliquot without compromising the integrity of the sample, multiple sample aliquots must be collected for analysis.

6.0 EQUIPMENT AND SUPPLIES

6.1 Syringe (Hamilton or equivalent): 5 mL, 25 mL gas-tight with Luer lock tip

6.2 Balances (Ohaus Navigator, Mettler PE600 or equivalent): top loading

6.3 Class “A” volumetric flasks: 1 mL, 2 mL, 5 mL, 10 mL, 20 mL, 25 mL, 50 mL, 100 mL, and 200 mL

6.4 Stainless steel and wooden spatula

6.5 Disposable pipets (Kimble brand or equivalent)

6.6 40 mL glass VOA vials (Eagle Pitcher, ESS, or equivalent)

6.7 Encore, or equivalent, closed-system sample collection devices and “T”-handle

7.0 STANDARDS AND REAGENTS

7.1 Reagent water: (ASTM Type II DI water, UV-treated)

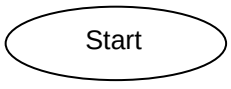
7.2 Purge-and-trap grade methanol: (J.T. Baker or equivalent)

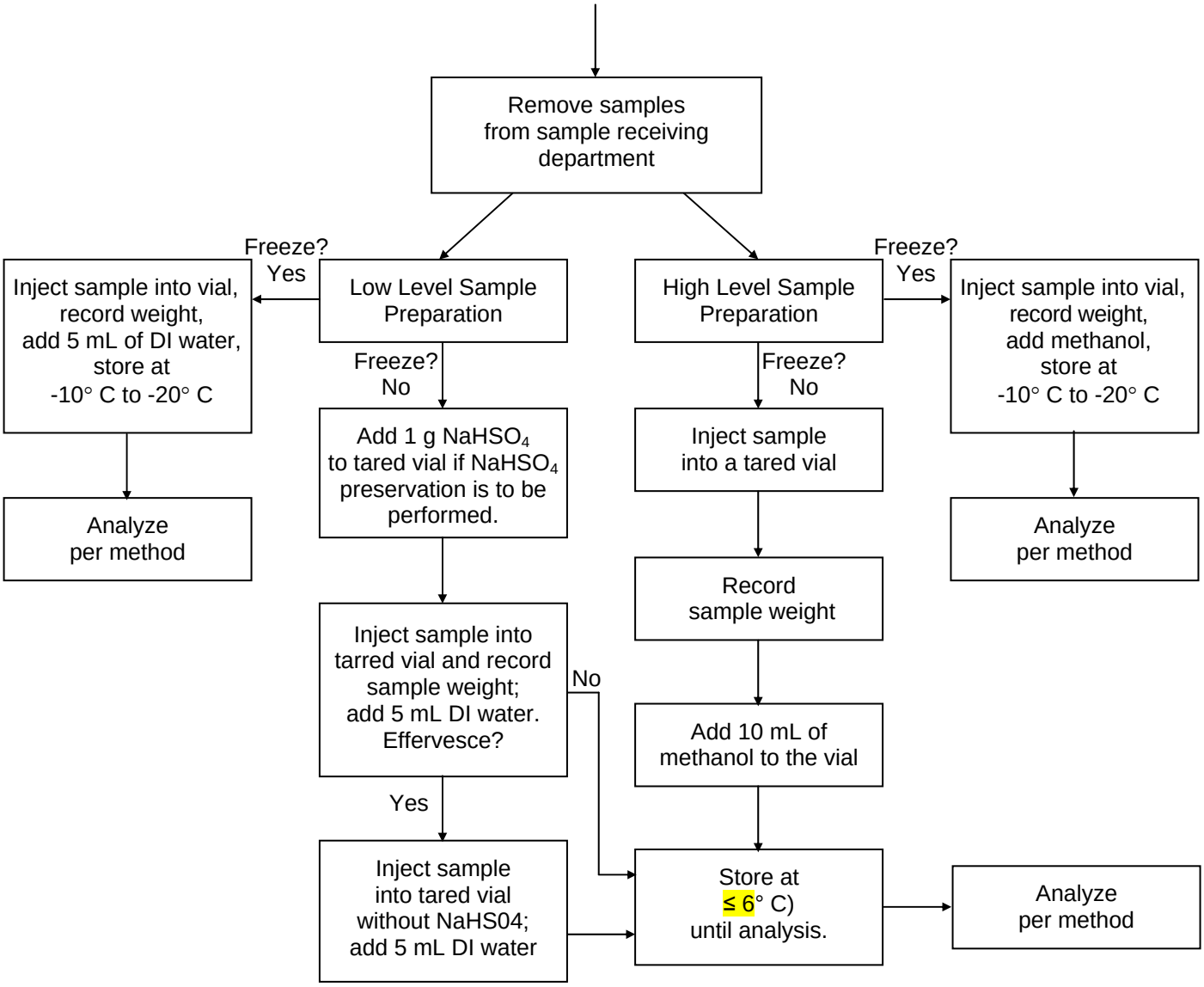
7.3 Sodium bisulfate, granular or monohydrate, reagent grade (J.T. Baker or equivalent)

7.4 Purified sand (J.T. Baker or equivalent)

8.0 DIAGRAM OR TABLE TO OUTLINE PROCEDURES

8.1 5035A





9.0 SAMPLE PREPARATION

9.1 **NOTE:** Screening of samples may provide guidance on whether sample dilution is necessary and may prevent contamination of the purge-and-trap system.

9.2 Water: Allow samples to warm to ambient. Place sample vial in auto-sampler tray and program auto-sampler. After analysis record sample pH. Dilutions are performed when quantitative results exceed the upper calibration limit. Dilutions are prepared in volumetric flasks (or by programming the auto-sampler) to obtain quantitative results within the calibrated range of the instrument. After analysis the auto-sampler performs self-rinsing procedures.

9.3 Low level soil/sediment collected by methods 5030B and 5030C: Weigh 5.00 g (± 0.5 g) of sample into a tared 40 mL VOA vial. Record the actual weight (± 0.01 g) in the VOA extraction log book. Add 5 mL of UV-treated DI water to vial. Place vial on auto-sampler. If needed dilute the sample accordingly, however, no less than 1.00 g (± 0.1 g) of sample is to be analyzed.

NOTE: The auto-sampler adds internal and surrogate standards.

9.4 High concentration soil/sediment, waste collected by methods 5030B and 5030C:

Soil/sediment:

Weigh 5.00 g (± 0.5 g) of sample into a 40 mL VOA vial or scintillation vial. Record the actual weight to (± 0.01 g). Add 10 mL of methanol. Shake vial and allow contents to settle. Dilute 1 mL of extract with DI water to a final volume of 50 mL using a glass volumetric flask or calibrated vial. Place in auto-sample tray. Dilute extract as necessary and record the initial volume (V_i) and final volume (V_f) on the quantitation report header.

Methanol-insoluble waste (“oil”):

Per soil/sediment above except 1.00 g (± 0.1 g) of sample is used. Dilute to 10 mL with methanol. Record the actual weight to (± 0.01 g). Dilute extract as necessary.

Methanol–soluble waste:

Weigh 1.00 g (± 0.1 g) of sample into a tared 40 mL VOA vial or calibrated scintillation vial. Record the weight to (± 0.01 g). Dilute to 10 mL with methanol. Dilute extract as necessary and record the initial volume (V_i) and final volume (V_f) on the quantitation report header.

9.5 Soil/sediment collected by method 5035A: Samples are submitted to the laboratory in Encore (or equivalent) collection devices or 40 mL VOA vials. Three

aliquots per sample ("A", "B", "C") are preserved unless directed otherwise by the client. Fractions "A" and "B" are preserved for low-level analysis; fraction "C" is preserved for mid-level analysis. Low-level preservation is performed using either sodium bisulfate or freezing; mid-level preservation is performed using methanol extraction or freezing.

9.5.1 Preservation of samples submitted in Encore (or equivalent) collection devices:

A packet containing 3 sample aliquots should be shipped to the laboratory in air-tight, o-ring sealed containers. Obtain samples from the sample custodian.

Record all pertinent sample information in the sample preservation logbook. (see Figure 1) or weighing may be performed and recorded electronically (see Figure 2).

Place pre-printed labels provided by sample receiving department onto appropriate containers. Pre-cleaned 40 mL glass screw-cap VOA vials are used for 5 g samples and pre-cleaned 60 mL wide-mouth VOA jars with septa are used for 25 g samples.

Prior to preservation, each sample container must be inspected to ensure the Encore container is properly sealed. As each Encore sample container is removed from the bag inspect the cap, plunger, and clasps. If a sample is received in the laboratory improperly sealed the aliquot is preserved as Fraction "C", unless otherwise requested by the client. Improperly sealed samples are recorded in the preservation log. Improperly sealed samples include, but are not limited to:

- Any part of the Encore is broken or cracked
- Loose caps
- Anything that compromises sample integrity

The client is notified when samples are received improperly sealed prior to analysis. Also, prior to analysis, the client is notified if an improperly sealed Encore will be used for analysis. When possible the laboratory avoids analyzing aliquots obtained from improperly sealed Encores.

Preservation of the low-level aliquots with sodium bisulfate requires the addition of 1.00 g (± 0.1 g) of sodium bisulfate to the vial prior to the addition of sample. Preservation of mid-level aliquots requires the addition of 10 mL methanol to the sample.

Place one pre-labeled container onto a top-loading balance and tare the balance. Lock an Encore sample container into T-handle. Slide the slots of the Encore container over two metal knobs in the barrel of the sample tool. Push down and twist to lock. A spring loaded lever will automatically engage to keep the encore container from twisting when locked. Pull off the cap, immediately inject the sample into the appropriate container. To inject, place the VOA container completely over the Encore sample container mouth and twist the metal plunger of the Encore sampling tool while pushing down to unlock the Encore sample container plunger. Record the sample weight. Add 5 mL of DI water to aliquot "A" and "B"; add 10 mL of methanol to aliquot "C". Immediately cap vial and shake vigorously to break up the sample.

Protocol to check for effervescence: If during sodium bisulfate preservation effervescence occurs, preservation with sodium bisulfate is halted and fraction "B" is preserved solely with water. If a separate sample aliquot is provided for a non-volatile analysis, a portion of the sample may be removed and added to a vial containing sodium bisulfate. Water is added and the mixture is monitored for effervescence. If effervescence occurs, sodium bisulfate preservation is not performed as previously stated. Effervescence is noted in the comments column of the preservation/preparation log.

9.5.2 Preparation of samples submitted in 40 mL VOA vials:

Sample aliquots are submitted to the laboratory either preserved or un-preserved. Preserved aliquots are placed in sample storage units until analysis. Un-preserved aliquots must be preserved upon receipt or placed directly in the sample storage units.

Prior to analysis, preserved aliquots are removed from the sample storage units and permitted to warm to ambient. If sample weights were not provided by the client the vial is weighed and compared to the tare weight. The difference between the vial weight and tare weight is the sample weight.

Prior to analysis, un-preserved aliquots are removed from the sample storage units and permitted to warm to ambient. If sample weights were not provided by the client the vial is weighed and compared to the tare weight. The difference between the vial weight and tare weight is the sample weight. 5 mL of DI water is injected through the septa of aliquots "A" and "B" and 10 mL of methanol is injected through the septum of aliquot "C". Vials are then vigorously shaken.

10.0 CALIBRATION PROCEDURES

10.1 Refer to method specific SOP.

NOTE: When samples are preserved with sodium bisulfate 1.00 g (± 0.1 g) of sodium bisulfate is also added to method blanks, calibration standards and laboratory control samples.

11.0 ANALYTICAL PROCEDURES

11.1 Refer to method specific SOP.

12.0 DETAILS OF CALCULATIONS

12.1 Refer to method specific SOP.

13.0 QUALITY CONTROL REQUIREMENTS

13.1 Refer to method specific SOP.

NOTE: Sodium bisulfate added to calibration standards, QC samples, and all samples for low-level analysis when required.

14.0 DATA REVIEW AND REPORTING REQUIREMENTS

14.1 Refer to method specific SOP.

15.0 PREVENTIVE MAINTENANCE

15.1 Refer to method specific SOP.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 Microbac is dedicated to eliminating or minimizing any and all laboratory waste which requires disposal or contributes to pollution of any type. To that extent Microbac has implemented new technology and converted to micro techniques when available to facilitate these goals.

Each laboratory generates specific waste streams which are segregated and collected in labeled satellite containers. The analysts in each department are responsible for proper disposal of the spent samples and chemical waste in the specified satellite waste collection vessel. The waste management technician checks the satellite containers either daily, or as needed. They are then combined into waste drums in our explosion-proof waste building located outside of the Microbac laboratory facility. These drums are labeled with start date and a manifest is created for each. They are picked up on a regular basis for disposal at a licensed disposal facility.

16.2. The waste streams are as follows:

- 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. All laboratory waste is accumulated, stored and disposed in accordance with all federal and state laws and regulations. Each employee receives training in the proper handling and disposal of hazardous waste that is specific to their job description. As a hazardous generator, we are subject to inspection from the Ohio EPA.

17.0 REFERENCES

17.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* SW-846, 3rd Edition. Method 5035A, July 2002; Methods 5030B/5035, December 1996; Method 5030C, May 2003; Method 8000B, December 1996. Method 8000C, March 2003.

17.2 Microbac SOP MSV01 “Analysis of Volatile Organic Analytes by Method 8260B”

17.3 Microbac SOP GCV05 “Analysis of GRO by Method 8015B/C/D”

17.4 Microbac SOP GCV09 “Analysis of Purgeable Aromatics by Method 602”

17.5 Microbac SOP MSV10 “Analysis of Volatile Organic Analytes by Method 624”

17.4 Microbac SOP LQAP “Laboratory Quality Assurance Plan”

17.6 Microbac SOP 33 “Laboratory Waste Management”

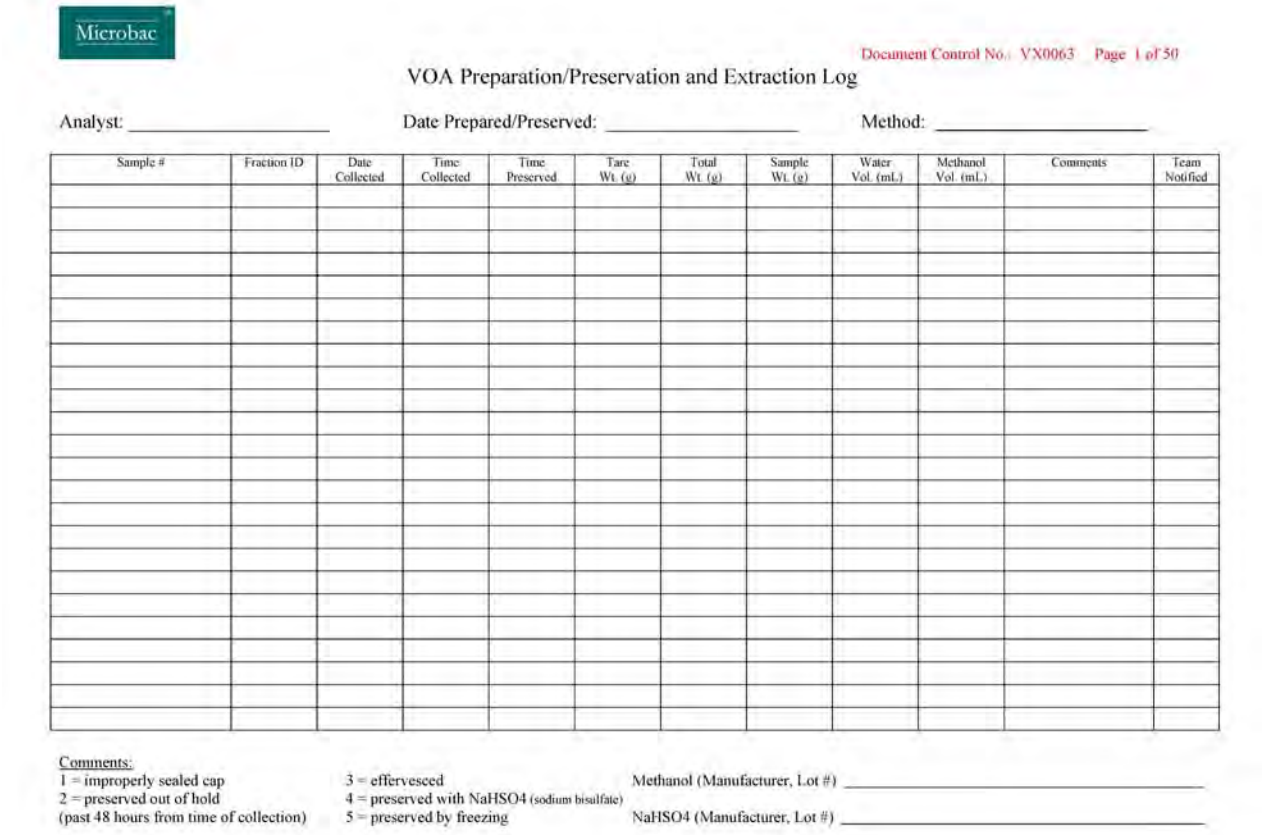
17.5 Microbac SOP 45 “Method Validation Procedures”

17.7 Microbac SOP GP-CAPA "Corrective Action/Preventive Action: Initiating, Tracking and Monitoring"

17.8 Microbac SOP GP-RCA "Root Cause Analysis"

17.10 Microbac SOP VGW01 "Cleaning Glassware in Organic Analysis"

Figure 1
VOA Preparation/Preservation and Extraction Log



Microbac

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VOA Preparation/Preservation and Extraction Log

Analyst: _____ Date Prepared/Preserved: _____ Method: _____

Sample #	Fraction ID	Date Collected	Time Collected	Time Preserved	Tare Wt. (g)	Total Wt. (g)	Sample Wt. (g)	Water Vol. (mL)	Methanol Vol. (mL)	Comments	Team Notified

Comments:
1 = improperly sealed cap
2 = preserved out of hold (past 48 hours from time of collection)
3 = effervesced
4 = preserved with NaHSO4 (sodium bisulfate)
5 = preserved by freezing

Methanol (Manufacturer, Lot #) _____

NaHSO4 (Manufacturer, Lot #) _____

Figure 2
VOA Preparation/Preservation and Extraction Log

Microbac Laboratories Inc.
VOA Preparation/Preservation/Extraction Log

Workgroup (AAB#):WG270339
Method:8260
Reagent ID:RGT10001

Analyst:DGB
Run Date:06/05/2008 09:50

SAMPLE #	Fraction	Collected	Preserved	PCT-S	Tare Wt	Total Wt	Sample Wt	Water	MeOH	Vt	Comments
L08020355-08	A	04/20/08 11:12	06/05/08 09:50	79.06			30.12	5		5	5
L08020355-09	A	04/20/08 11:25	06/05/08 09:50	77.08			30.12	5		5	5
L08020355-09	B	04/20/08 11:25	06/05/08 09:51	77.08	5.12	30.1	24.98	5		5	5
L08020355-09	C	04/20/08 11:25	06/05/08 09:52	77.08			25.08		10	15.748816	5

Comments: 1 = improperly sealed cap
2 = preserved out of hold
3 = effervesced
4 = preserved with NaHSO4
5 = preserved by freezing
6 = preserved in field

Analyst: Douglas Butcher

VOA_EXT - Modified 05/22/2008
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ALS Standard Operating Procedure

Confidential & Non-Controlled

DOCUMENT TITLE:	SAMPLE RECEIVING, ACCEPTANCE, AND LOGIN
REFERENCED METHOD:	N/A
SOP ID:	SMO-SMPL_REC
REV. NUMBER:	14.0
EFFECTIVE DATE:	02/22/2014

ALS Environmental

SAMPLE RECEIVING, ACCEPTANCE, AND LOGIN

SOP ID:	SMO-SMPL_REC	Rev. Number:	14.0	Effective Date:	02/22/2014

Approved By: _____ Date: 2/17/14
Sample Management Custodian - Manny Zamora

Approved By: _____ Date: 2/17/14
QA Manager - Chaney Humphrey

Approved By: _____ Date: 2/18/14
Laboratory Director - Kelly Horiuchi

Archival Date: _____	Doc Control ID#: Non-Controlled	Editor: _____

TABLE OF CONTENTS

DATA AND RECORD ARCHIVING

1) Scope and Applicability

1.1 The purpose of this standard operating procedure (SOP) is to describe the requirements and guidelines necessary for effective sample receiving as well as the documentation associated with this process. Additionally, this document describes the procedures relating to the Sample Management Office for initiating any subcontract documentation.

1.2 This standard operating procedure (SOP) is applicable to all samples delivered to this laboratory and subcontracted out for analysis.

2) Summary of Procedure

2.1 For the purposes of this document sample receiving is considered to be an all-inclusive system, which comprises sample custody transfer, sample acceptance, and sample login.

2.2 This procedure is essential in identifying compromised samples and ensuring the validity of the laboratory's sample data. Improper sample handling affects the credibility and acceptability of analytical results, regardless of their accuracy and precision. Therefore, it is essential that all samples be properly received and handled and that the documentation maintained accurately reflects the integrity and processing of samples.

3) Definitions

3.1 Custody The guardianship or safe keeping of a sample. A sample is considered to be in a person's custody if it is physically in their possession, or it is in their view after being in their possession, or it was in their possession and then locked up or sealed to prevent tampering, or it is in a secure area.

3.2 Chain of Custody (COC) Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. (NELAC)

3.3 Internal Chain-of-Custody Procedures employed to record the possession of samples from the time of sample receipt until disposal/storage and are performed at the special request of the client. These protocols are handled electronically through LIMS.

3.4 Compromised Samples Those samples which are improperly sampled, insufficiently documented, improperly preserved, collected in improper containers, exceeding holding times and/or not received intact when delivered to a laboratory.

3.5 Holding Times (Maximum Allowable Holding Times) The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid or not compromised. (40 CFR Part 136)

3.6 Preservation Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

3.7 Service Request (SR) / Job File A unique, computer generated laboratory number which is assigned to a sample or group of samples submitted (at the same time) by the client representing one job or project. The job or project includes specific sample management information, analysis data, client correspondence, analysis report and

other pertinent information comprising a single sample submission containing one or more samples in a client's project.

3.8 COC Chain-of-Custody

3.9 SACF Sample Acceptance Check Form

3.10 LIMS Laboratory Information Management System

3.11 SMO Sample Management Office

3.12 PM Project Manager (may be referred to in other lab documents as PC/Project Chemist)

3.13 SMC Sample Management Custodian

3.14 SDG Sample Delivery Group

3.15 EDD Electronic Data Deliverable

4) Health and Safety Warnings

4.1 Handle all samples as potentially hazardous. Gloves should be worn when handling all samples, safety glasses, and a lab coat shall be worn when handling liquid or soil (solid) samples. Always work under a hood when chemically preserving samples. Also place broken or leaking samples under the hood. Get assistance when confronted with any situation that appears to be dangerous.

4.2 In the event of broken liquid or soil samples, SMO needs to cleanup using one of the following procedures:

- Liquids: Broken glass is handled carefully using disposable gloves and disposed of in the Glass Disposal Box. Remaining sample and cleanup materials are disposed of in accordance with the *SOP for Waste Disposal*.
- Soils: Broken glass is disposed of in the Glass Disposal Box, and the soil is disposed of into the 55-gallon soil drum. This information is noted on the Service Request Form and the PM is notified. Soil that is still intact in a glass jar may be salvaged with client's approval.

5) Personnel Qualifications and Responsibilities

5.1 All employees involved with sample receiving, acceptance and login must ensure the procedures described in this document are followed. More specifically, SMO personnel, Project Managers and the Sample Management Custodian are responsible for complying with and implementing the procedures listed in this document.

6) Procedure

6.1 Upon sample receipt, the condition, including any abnormalities or departures from normal or specified conditions as described in the test method or method standard operating procedure must be recorded. All of the information including any other observances must be recorded on the Sample Acceptance Check Form (Attachment 2) and other associated documentation as detailed in the following procedures. Refer to Section 6.4 for the necessary procedures and documentation requirements dictated by abnormalities or departures.

6.2 Sample Custody

Upon delivery to the laboratory, the sample(s) must be transferred (as soon as possible) to a Sample Management Custodian (SMC) or a representative of the

laboratory who accepts and assumes custody of the sample(s). Samples are transported to the laboratory by a number of means including courier, common carrier, sampler or client representative. The acceptance of a sample is achieved by presenting a signature, date and time of receipt in accordance with the requirements of the transmitter and client such as an electronic board (i.e. FedEx) and Chain of Custody Form. Sample shipping containers are examined for the presence and condition of custody seals, locks, shipping waybills, etc. After opening shipping containers, remove any other documents in order to evaluate login priority (see note below) and continue processing the samples.

Note: Rush requests and samples with short holding times are given top priority for processing. Sample Custodian alerts Project Manager and analysts by calling them and distributing copies of the COC and any other pertinent documentation. Refer to Appendix F in the Quality Assurance Manual for Sample Preservation and Holding Times which list maximum allowable hold times.

6.2.1 Shipping Receipts and Chain of Custody (COC) Forms

6.2.1.1 Packing Slips

A copy of the packing slip must be kept, whenever possible, as part of the permanent chain of custody process and placed in the job file.

6.2.1.2 Chain of Custody Forms

These forms may be identical to the one issued by the laboratory (see Figures 11-1 and 11-2 in the Quality Assurance Manual) or clients may submit samples using a similar form. The SMC or designee shall sign the COC and add the date and time of receipt. In addition, the service request number must be added to the COC form at the time of sample login.

6.2.2 Legal/Internal Chain of Custody (COC)

When samples are logged in using LIMS, the system automatically generates an internal chain of custody each time a sample is scanned into possession for use within the laboratory. This internal COC may be accessed anytime during the laboratory procedures and is provided to the client upon request.

6.3 Sample Receipt and Login

In order to evaluate the state of a sample upon receipt, the laboratory must evaluate certain parameters including container type, volume and preservation (thermal and chemical). Compare the findings against the specified criteria in Sample Preservation and Holding Times Tables in the most recent Quality Assurance Manual (Appendix F). Refer to Section 6.4 for the discrepancy/exception and the rejection of samples procedures.

Important: For odorous samples, refer to Section 6.8.1 for the handling procedure.

6.3.1 Service Request Form

A Service Request form (Attachment 3) shall be completed in LIMS for all samples received by the laboratory using the information provided on the sample receipt documentation (e.g., COC) and data collected by the SMC. A copy of this completed form shall accompany the sample(s). The following includes a description of the key components.

1. Service Request Number: Client's job file number (automatically assigned)
2. Report Name: Name of Client that shall be on report.

3. Reporting Address: Address of the Client that will be on the report.
4. Project Name: Client's referenced study or project name.
5. Project Number: Client's reference study or project number.
6. ISR Number (if applicable): Internal Service Request (between laboratories in the network using the same LIMS system)
7. Date Received: Date the laboratory actually received samples.
8. Purchase Order: Client's purchase order number or verbal notation (default).
9. Project Manager: The PM responsible for all client activity for job file.
10. TAT: Sample turnaround time (normal TAT, if not specified).
11. Initials: Initials of SMC or alternate logging in the sample(s).
12. Sample Type: Type/container of sample submitted by client.
13. Comments: Any comments concerning the sample or samples being submitted including short hold times.
14. Tier: QC level if one is given on the ISR or COC.
15. EDD: If EDD is required or not.
16. Method: Specified method for the samples to be analyzed.
17. Sample ID: Client's specified sample identification.
18. Test(s) Required: Number of methods for analysis on the samples.
19. Date Collected: Sampling date for each sample.
20. Time Collected: Sampling time for each sample.
21. Sample Type: Sample matrix for each sample.

Note: Some of the information (client's project name or number) may not be provided and will not be included on the form.

6.3.2 LIMS Login

Prior to sample arrival, the Project Manager may create a sample delivery group (SDG) in LIMS based on project information and in accordance with the *SOP for Project Management*. Analysis information associated with each sample is stored in this SDG. When samples arrive, the custodian uses this SDG as a template to create a job folder specific to the samples received. The custodian could either manually search SDG information from LIMS or find it by scanning the barcode of the bottle order form (also known as Bottle Order \ Sample Supplies Summary form).

Once the correct SDG has been selected, a sample template is chosen from the SDG template that best matches the analyses stated on the COC for each sample included on the COC. Once all the samples are chosen the custodian creates a unique job folder. Job folder is then edited as necessary (e.g., project name and number, date and time of sample collection, and client sample IDs).

Each sample container for a sample is given a unique lab code by the LIMS system. This lab code is express in the format of PYYJJJJ-sss.ccc.

Where:

"P" is the current lab ID code for Simi Valley,
"YY" is the two-digit year code (e.g., 14 for Y2014),
"JJJJJ" is the five-digit job number (e.g., 00001 for the first project),
"sss" is a three-digit sample ID number;
"ccc" is the three-digit container ID number.

An example for the second container of the first sample for the first job of year for 2014 would be P1400001-001.002. The alphanumeric code before the dash is the job number, the number after the dash is sample ID and the number after the period is container ID.

6.3.3 Sample Acceptance Check Form The SMC shall complete and generate a Sample Acceptance Check form (Attachment 2) based on the information specified in this section. This form is given to the PM and electronically accessible so that Chemists may input additional preservation check information.

Once the samples have been checked and the SACF produced, the form is to be saved at G:\\STARLIMS\Sample Acceptance Check form (as SR#_Client_Project) so that additional information such as pH may be added.

6.3.3.1 Sample Acceptance Policy Sample containers are removed and organized according to the COC identification and analyses. The sample conditions are checked to ensure sample integrity has not been compromised. These steps are listed to complete the criteria for the acceptance or rejection of samples but they do not necessarily occur in this order. Each point is an evaluation requirement which must be used to complete the Sample Acceptance Check form.

- Sample submission documents are properly used, fully completed (in ink) and shall include the client, sample identification, project name or location, date and time of collection, collector's name, sample type, preservation type (if applicable) and any special remarks concerning the sample.
- Proper sample labeling is considered: unique sample identification (ID), durable labels (labels that are not easily removed) and the use of ink.
- Sample containers checked for integrity (broken, leaking, Tedlar® bags are received flat, under inflated or with the valve open, Summa canisters are received under an unacceptable vacuum or with the valve open, etc.). Reject samples with broken or leaking containers.
- Sample container labels and/or tags agree with the sample documentation (ID, required analyses, etc.).
- Adherence to specified holding times (see Appendix F in the Quality Assurance Manual)
- Appropriate containers (size, type) are received for the requested analyses (see Appendix F in the Quality Assurance Manual).
- Proper temperatures of sample containers, if applicable (see Appendix F in the Quality Assurance Manual).
- Adequate sample volume (see Appendix F in the Quality Assurance Manual)
- Assessment of proper sample preservation, where applicable (see Appendix F in the Quality Assurance Manual). Reject samples preserved with the inappropriate preservatives for which the

requested analysis has been compromised (e.g., cyanide samples preserved with acid).
- Any notation made by other persons accepting the sample and any evaluations made and noted on the associated documentation.

Once the samples have been checked against the Sample Acceptance Policy, the sample custodian must generate a Sample Acceptance Check form, sample identification labels, and Service Request form (optional). The Project Manager is responsible for generating and emailing the Sample Receipt Acknowledgment form (Section 6.5) if requested. The sample login forms and labels must be completed to properly track laboratory samples.

6.3.3.2 Measurement of Temperature The temperature of all coolers containing samples requiring thermal preservation shall be taken using a verified thermometer calibrated against NIST standards and the data recorded (with correction factor applied) on the Sample Acceptance Check form (Attachment 2).

A reading shall be taken by placing the thermometer in the cooler so as to give an accurate reflection of the cooler temperature (i.e. not directly on ice or blue ice and at approximate sample level or in the temperature blank, if supplied). The lid must be closed to allow enough time for the thermometer to reach equilibrium (i.e., a minimum of five minutes) before the temperature reading is taken and recorded. The arrival temperature check is considered acceptable if the following is adhered to:

- Samples have a temperature of +/-2˚C of the required temperature or the method specified range; or
- Samples with a required temperature of 4˚C have a temperature ranging from just above freezing of water to 6˚C; or
- IMPORTANT: The US EPA has published revisions to the Code of Federal Regulations at 40 CFR 136 and 40 CFR 141. These revisions, known as the Method Update Rule (MUR), became effective 4/11/07 and contains a revised approved methods tables and temperature requirements. A number of the methods have been updated and for those methods the temperature requirement has been updated to ≤6˚C. Refer to Appendix F in the most recent Quality Assurance Manual for the specific methods that are affected.

Note: Samples that are hand delivered to the laboratory immediately following collection may not meet these criteria. This is considered acceptable if there is evidence that the chilling process has begun such as arrival on ice. Include a notation on the Sample Acceptance Check Form.

6.3.3.3 Chemical Preservation A pH measurement may be required on certain tests, the pH value shall be documented on the Sample Acceptance Check Form. Perform this check in accordance with the applicable method SOP and the *SOP for Laboratory Storage, Analysis, and Tracking.*

The pH of the sample shall be checked with a narrow ranged pH indicator strip (preferable). Take a small aliquot of the sample with a transfer pipette and place a few drops onto the pH indicator strip. Ensure that a new pipette is used for every sample container to prevent

cross-contamination. Refer to Section 6.7 on specific information for subcontracted jobs.

6.3.3.4 Headspace Check for headspace in VOA vials. Pay close attention to samples that are opaque; bubbles may not be easily observed. Samples with heavy sediments may stick to the vial, making it appears to have no bubble when the vial is inverted. Any bubble in the sample should not exceed 5-6 mm.

6.3.3.5 Summa Canisters The pressure of each Summa canister shall be checked and recorded at the time of receipt to ensure the sample has the appropriate volume. The initial (upon receipt) pressure shall be noted on the Service Request Form under Initial Reading (inHg or psig) and on the back of the sample tag. At the time of sample submission, ambient air sampling canisters will likely have a vacuum (negative pressure). If the canister has a negative pressure, the gauge will read in inches of Mercury (inHg) or pounds per square inch (psig) depending on the gauge used. If the reading is inHg, the value must be converted to psig (A conversion chart may be used and is located in the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters* Attachment B). Vacuum readings entered in inHg to the system will be automatically converted to psig.

Returned canisters that are not samples are logged in and handled following the same procedures. Canisters received at an initial pressure *lower than* -9.8 psig (-20.0 inHg) are shelved on a canister rack outside SMO (P-101) for the canister department to clean. Canisters received that have an initial pressure *higher than* -9.8 psig (-20.0 inHg) are placed on a canister rack in SMO for screening before they are returned to the canister department for cleaning. This procedure must be performed in accordance with the *SOP for Evaluation and Pressurization of Specially Prepared Stainless Steel Canisters.*

6.3.3.6 Sample Login Contingency Plan This section is designed to detail the sample custody and receipt procedure for samples that are delivered to the laboratory late in the day or when the SMC or designee is not present. If sample(s) are delivered under thermal preservation, laboratory personnel shall evaluate the cooler temperature per Section 6.3.3.2. The temperature must be noted on the COC form along with the date and initial of the person making the notation. Refer to the Quality Assurance Manual for information on preservation requirements, which are listed by method and sample type. The person, following acceptance, evaluation and analysis (if performed), should place the samples in the appropriate storage location in accordance with the *SOP for Laboratory Storage, Analysis, and Tracking* and submit the paper work to SMO in order for the login process to be completed.

6.3.3.7 Short Hold Times When samples are delivered to the laboratory with little remaining on the hold time it may be possible for the analysis to proceed prior to the login process. The following are circumstances where this is allowed.

- Tedlar bag samples only
- If there is no time for sample(s) evaluation and login prior to hold time expiration and an analyst is able to analyze the sample(s) immediately.

However, there are requirements that must be followed by the analyst(s) if the samples are to be analyzed prior to sample login.

- At a minimum, the analyst shall review/compare the chain of custody with the samples received to ensure that the sample identifications, etc. are correct.
- It is imperative that the client sample ID be referenced on all laboratory analytical documentation.
- Also, the analyst should check the integrity (i.e. leaking or flat Tedlar bag) of the samples and make any notations on the associated documentation.
- Additionally, once the samples have been analyzed they are to be immediately delivered back to SMO for the sample acceptance and login procedures detailed in this SOP.

6.3.3.8 Sample Identification Labels After samples have been logged into the computer and the lab ID assigned, the SMC shall print labels for each sample container received. Each computer-generated label is affixed to the appropriate sample container, where possible. Certain sample containers, such as solid adsorbent cartridges, are placed in a sealed bag identified with the job number and all the laboratory ID codes associated with each sample in the bag.

6.3.3.9 Sample Login/Labeling Verification After labels have been applied to the corresponding sample containers they should be verified by a second person to ensure proper labeling. Place all associated documentation into the job file and submit to the Project Manager.

- Once the documentation has been generated and the labeling verification has been performed, the custodian must complete the first section of the LIMS Sample Login Verification Form (Attachment 4).
- The Project Manager responsible for the project verifies login information. This process is documented on the LIMS Sample Login Verification form. It is only after this secondary review that the job folder is released out of the login console to the job in progress area, making the analysis information available to the analysts.

6.4 Discrepancy / Sample Rejection Procedures

Any discrepancies or concerns are noted on the Sample Acceptance Check Form (per Sample Acceptance Policy, see Section 6.3.3.1) and immediately communicated to the appropriate Project Manager. If and when there is any doubt as to the suitability of a sample to be tested such as a leaking valve, broken container, etc. the SMC shall inform the PM. Regardless of the discrepancy, the PM shall be responsible for coordinating all correspondences and consulting with the client for further instructions before the laboratory may proceed. However, when there are short holding time constraints, the laboratory may complete the sample analysis, where possible for all samples in the client's job file including the sample in question.

6.4.1 Chemical Preservation for Water and Soil Samples Contact the PM and if the PM approves adding preservative to bring sample within the proper range, be sure to record the specific sample container identifications, preservative added, including type, lot number(s), and final pH on the Sample Acceptance Check form (Attachment 2) (even if subcontracting). Refer to Section 6.7 for information on sub-contracting and splitting samples, where appropriate.

When chemical preservation is performed in the laboratory the Preservative Tracking Log (Attachment 2, *SOP for Media Request Fulfillment*) must be utilized for documentation purposes.

6.4.2 Login Revisions Changes to SR forms may be made by anyone authorized for sample login and Project Management capabilities; however, it is recommended that whenever possible documentation of the reasons for the changes and the person making those corrections is documented and any copies of the original must be retained and marked as obsolete.

6.5 Sample Receipt Acknowledgment

An acknowledgment form (Attachment 5) may be accessed and emailed to the client, along with a PDF of any other requested documentation.

6.6 Job File and LIMS Documentation

The sample documentation shall be maintained in each client's job file in accordance with current procedures and shall at a minimum include:

- Original chain of custody form (if utilized) with the laboratory job number
- Service Request Form
- Preservation Tracking Log, if utilized
- Sample Acceptance Check Form
- Sample Login Verification Checklist
- Any documentation including memos or transmittal forms, which are transmitted to the laboratory by the common carrier, courier, sampler, or client.
- Any internal documentation which is pertinent to the handling and/or analysis of the samples.

Note: The original and all copies and revised versions of documentation must be kept in the associated job file.

Once the samples have been received, accepted (or rejected) and logged into the laboratory system, a job file (referencing the corresponding service request number) must be created and all receipt, acceptance and login documentation included. The COC is to be scanned into a PDF and attached to the LIMS job file. The job file must be submitted to the appropriate Project Manager for approval. The job file will be kept in a designated area for the inclusion of all the remaining documentation for the project including analytical data, invoices, etc.

6.7 Sample Transfer between Laboratories

The following must be adhered to for all samples, extracts, digestates and split samples that are transferred, carried or shipped from one laboratory to another (between In-Network laboratories and to laboratories outside of the Network). Samples are generally prepared for shipping by packing bubble wrapped glass containers in a cooler filled with blue ice (or ice). Custody seals are signed and dated and placed on the front of the cooler. The cooler is then sealed with packaging tape. For specific information on sample transfer to the off site preparation facility, refer to the *SOP for Laboratory Storage, Analysis, and Tracking* (Also, refer to Section 6.7.1.3).

Samples not analyzed at the laboratory are subcontracted to pre-approved laboratories (internal and or external). Samples are logged in for the required tests and assigned a subcontract lab (as assigned by the PM in the SDG, by flagging the team column of the folder with the appropriate sublab). A subcontract COC is printed from LIMS once the login has been completed. The subcontract COC is then placed in the job folder after a copy of document is made.

Note: If LIMS does not have the appropriate test or sub-contract laboratory code, a Request for Test Code or "Sublab" form is filled out and submitted to Kelso IT. In addition, if the sublab is not specified in the SDG, it will automatically be flagged and a subcontract lab must be selected. Contact the PM, if this occurs.

6.7.1 In-Network Sample Transfer This laboratory, when transferring samples to an In-Network laboratory, could either initiate a new chain of custody record or use a photocopy of the original chain of custody record. The SR number from the originating laboratory may remain the same when subcontracting to a laboratory within LIMS; and any documentation generated by the laboratory would be included in that job file.

6.7.1.1 A new chain of custody record may be initiated if the number of samples or analyses is small enough so that it is not too time consuming to write out the new chain of custody record. The sample custodian at this laboratory must accurately transfer the entire client and sample information to the new chain of custody record and sign and date relinquishing it and the samples.

6.7.1.2 A photocopy of the original chain of custody record may be used when the number of samples or analyses is large or the chain of custody record is complicated and it would take a lot of time to rewrite the client and sample information on a new chain of custody record. On the chain-of-custody-record-photocopy, the sample custodian preferably using blue ink must:

- Indicate which samples have been sent by crossing out the samples retained;
- Correct the number of sample containers actually being transferred by crossing out the number and writing the number of bottles sent;
- Indicate which analyses the subcontract network laboratory will be performing by highlighting the analyses to be performed and/or crossing out the analyses not subcontracted;
- Write the service request number of the originating laboratory on both the original chain of custody record and on the chain-of-custody-record-photocopy; and
- Sign the chain-of-custody-record-photocopy relinquishing it and the samples.

A photocopy of this completed document shall be placed in this laboratory's project file. The receiving network laboratory should treat this photocopied chain of custody record as its official chain of custody record for their project file. This chain-of-custody-record-photocopy must be signed, preferably using blue ink, when the samples are received and logged in at the receiving network laboratory. It will be retained by the receiving network laboratory and a photocopy returned to the originating network laboratory with the final analytical report.

6.7.1.3 Off-Site Extraction Facility Samples are received at the main laboratory and transported to the off-site extraction facility located at 2360 Shasta Way, Unit G, Simi Valley, CA utilizing LIMS for custody relinquishment.

- Samples (PUF, PUF/XAD-2 cartridges and filters) must be transported, wrapped in aluminum foil in tightly sealed glass jars and maintained at <4˚C with blue ice.

- VOA vials must be wrapped in bubble wrap and transported in a cooler with blue ice to adhere to the temperature requirement of 4˚C+/-2˚C.

The technician must use LIMS when receiving the samples to relinquish extracts to the analyst for storage and analysis.

6.7.2 Sample Transfer to an Out of Network Laboratory (Interlaboratory Transfer)

The originating laboratory, when transferring samples to a laboratory outside the network, must initiate a new chain of custody record. This will help to protect the identity of our customer from the outside laboratory and maintain client confidentiality. The sample custodian will indicate that this laboratory is the client on this new chain of custody record and must accurately transfer all the sample and analysis information. Also, the purchase order number is to be included on the new chain of custody record. The new chain of custody record must be signed and dated relinquishing it and the samples.

6.7.2.1 pH Adjustment Certain methods require a pH check and adjustment to be recorded on the Sample Acceptance Check form. After performing pH adjustment place a yellow tape with the words “pH Check” and “date and time” of adjustment across the top of the bottle. Measure pH after 16 hours; adjust pH if necessary, and repeat the process until proper pH is obtained. The analyst will perform the pH check at the time of analysis.

If received within two weeks of collection, acid preserve upon receipt in the laboratory to lower pH to <2. Following acidification, the sample should be mixed, held for 16 hours, and then verified to be pH <2 just prior to sending out to sub-contract network or out of network laboratory. If for some reason such as high alkalinity, the sample pH is verified to be >2, more acid must be added, and the sample held for 16 hours until verified to be pH <2.

6.7.3 Splitting Samples Avoid splitting whole volume analysis samples; e.g., BNA, pesticides, PCBs. Make appropriate sample splits by pouring sample into containers with appropriate preservative already added.

6.8 Storage and Documentation Distribution

When all samples have been labeled and verified, they are to be placed in the designated storage areas per the *SOP for Laboratory Storage, Analysis, and Tracking*. Where necessary, there are refrigerators and freezers dedicated for specific storage requirements (e.g., Wet Chem, SVOA, etc.) and specific locations entered in the Sample Location module of LIMS.

All documentation (e.g. COCs, Sample Acceptance Check Form, Sample Login Verification, etc.) are to be placed inside the Job Folder and given to the PM. The PM will then distribute the folder to the appropriate department.

6.8.1 Odorous Sample Storage Odorous samples (ex., Tedlar bags or VOAs for sulfur) are to be placed in the SMO hood for login and labeled with a “HIGH SULFUR CONCENTRATION” caution sticker. The PM is to be contacted so that the best course of action may be taken to prevent any laboratory contamination. Following login, every possible precaution is to be taken when storing the samples; therefore, wherever they are stored must minimize any cross-contamination between stored samples and into the lab air for possible contamination into laboratory systems. Segregation of samples must be

performed as necessary to ensure that no contamination occurs between samples, extracts, and standards. After analysis, the odorous samples are returned to the SMO hood for disposal the next day upon PM approval.

7) Equipment and Supplies

7.1 Documentation and Records

Forms, Checklists and other required documentation to be maintained are listed in Section 6.6.

8) Quality Assurance and Quality Control

8.1 Internal system audits shall be performed by the Quality Assurance Manager to assess adherence to the guidelines described in this SOP.

9) Summary of Changes

Table 9.1

Revision Number	Effective Date	Document Editor	Description of Changes
14.0	02/22/2014	C. Humphrey	SOP updated using current ALS SOP Template. New cover page and footer. See changes below.

Section 4 New section – Information previously in Section 2.3
Section 5 Renamed "Personnel Qualifications and Responsibilities"; Previously Section 4
Section 6 Previously Section 5; Updated QA Manual references throughout
Section 6.3.2 Updated year in example
Section 7 Renamed "Equipment and Supplies"
Section 8 Renamed "Quality Assurance and Quality Control"; Previously Section 6
Section 9 Renamed "Summary of Changes"; Previously Section 8
Section 10 Renamed "References and Related Documents"; Previously Section 9
Updated References
Section 11 Previously Section 10
Attachment 1 Updated QA Manual Reference
Attachment 2 Updated
Attachment 3 Updated
Attachment 5 Updated

10) References and Related Documents

10.1 TNI 2009 Standards.

10.2 US EPA Methods Update Rule (MUR), effective 4/11/07.

10.3 *Department of Defense Quality Systems Manual for Environmental Laboratories*, Version 5.0, July 2013.

10.4 *General Requirements for the Competence of Testing and Calibration Laboratories*, ISO/IEC 17025, second edition, 2005-05-15.

10.5 AIHA-LAP, LLC Policy Document – Module 2A Revision 12: Effective Date: March 9, 2013.

10.6 Minnesota Administrative Rules, *Department of Health*, Chapter 4740, Laboratories; Accreditation Requirements.

11) Attachments

11.1 Attachments

Attachment 1	Training plan for Sample Receiving
Attachment 2	Sample Acceptance Check Form
Attachment 3	Service Request Form
Attachment 4	Sample Login Verification Form (also included in the *SOP for Project Management*)
Attachment 5	Sample Acknowledgement Form

Note: Forms are examples and may be modified as long as the minimum requirements of this document are met.

Attachment 1

Training Plan for Sample Receiving

Training Plan for Sample Receiving, Acceptance, and Login

Trainee _____ Trainer _____ Date _____

1. Read SOP — Trainer ____ Trainee ____ Date _____
2. Read Holding Time, Matrix Table (Appendix F of QA Manual) — Trainer ____ Trainee ____ Date _____
3. Demonstrated understanding of — Trainer ____ Trainee ____ Date _____

   Sample Acceptance Check Form & Chain of Custody Form
4. Demonstrated familiarity with related SOPs — Trainer ____ Trainee ____ Date _____

   SOP for Making Entries onto Analytical Records
   SOP for Laboratory Sample Storage, Analysis, and Tracking
   SOP for Nonconformance and Corrective Action
   SOP for Media Request Fulfillment
5. Sample Receipt — Trainer ____ Trainee ____ Date _____

   ☐Understands & knows Sample Hold Times for different methods, media and matrices (or where to find info.)
   ☐Understands Sample Receipt Procedures during business hours as well as after hours
   ☐Knows acceptable temperature for cooler/samples received and how to evaluate and document information
   ☐Knows how to check liquid samples for air bubbles and how to document information
   ☐Knows how to check samples for integrity & if they are compromised (& what this means), how to document
   ☐Knows appropriate containers for samples received according to requested analyses
   ☐Knows adequate sample volume for the analyses requested
   ☐Knows the proper preservation of samples received according to the requested analyses
   ☐Knows when & why the project manager needs to be notified
   ☐Knows how to check canister pressures
6. Sample Login — Trainer ____ Trainee ____ Date _____

   ☐Understands procedure of login and can successfully accomplish the task
   ☐Understands every field of the SR form
   ☐Able to generate a completed project/job – SR form
   ☐Understands the Sample Acceptance Check Form and how to utilize it for different media
   ☐Understands the Sample Receipt Acknowledgment Form and how to utilize it
   ☐Understands the SR form "Draft" copy and know when to utilize it
   ☐Understands the notes that are required at the top of the SR form (i.e., pressurize with helium) and why
   ☐Understands the documentation that must accompany canisters to pressurization
   ☐Understands when an NCAR must be generated in SMO
   ☐Able to submit hardcopy project requirements and how to document special requirements (& what is important to include) on SR form
   ☐Knows steps in documenting samples received outside of hold time
   ☐Know how to properly label samples during login
7. Container Tracking Program — Trainer ____ Trainee ____ Date _____

   ☐Demonstrates knowledge of receiving sample media back into the laboratory
8. Freezer and Refrigerator Temperature Readings — Trainer ____ Trainee ____ Date _____

   ☐Read *SOP for Calibration and Use of Laboratory Support Equipment*
   ☐Logbooks (Calibration logbook & Freezer / Fridge Temperature logbook)
   ☐Knows required temperatures
   ☐Understands what to do if a temperature exceeds the required temperature (i.e., documentation, notification of QA)
   ☐Ability to calibrate thermometers using appropriate NIST traceable thermometer
   ☐Applies correction factors to applicable laboratory thermometers
   ☐Reset digital thermometers when appropriate

Attachment 2

Sample Acceptance Check Form

ALS Environmental
Sample Acceptance Check Form

Client: _____ Work order: _____
Project: _____
Sample(s) received on: _____ Date opened: _____ by: _____

Note: This form is used for all samples received by ALS. The use of this form for custody seals is strictly meant to indicate presence/absence and not as an indication of compliance or nonconformity. Thermal preservation and pH will only be evaluated either at the request of the client and/or as required by the method/SOP.

		Yes	No	N/A
1	Were **sample containers** properly marked with client sample ID?	☐	☐	☐
2	Container(s) **supplied by ALS**?	☐	☐	☐
3	Did **sample containers** arrive in good condition?	☐	☐	☐
4	Were **chain-of-custody** papers used and filled out?	☐	☐	☐
5	Did **sample container labels** and/or tags agree with custody papers?	☐	☐	☐
6	Was **sample volume** received adequate for analysis?	☐	☐	☐
7	Are samples within specified holding times?	☐	☐	☐
8	Was proper **temperature** (thermal preservation) of cooler at receipt adhered to?	☐	☐	☐
9	Was a **trip blank** received?	☐	☐	☐
10	Were **custody seals** on outside of cooler/Box?	☐	☐	☐
	Location of seal(s)? _____ Sealing Lid?	☐	☐	☐
	Were signature and date included?	☐	☐	☐
	Were seals intact?	☐	☐	☐
	Were custody seals on outside of sample container?	☐	☐	☐
	Location of seal(s)? _____ Sealing Lid?	☐	☐	☐
	Were signature and date included?	☐	☐	☐
	Were seals intact?	☐	☐	☐
11	Do containers have appropriate **preservation**, according to method/SOP or Client specified information?	☐	☐	☐
	Is there a client indication that the submitted samples are **pH** preserved?	☐	☐	☐
	Were **VOA vials** checked for presence/absence of air bubbles?	☐	☐	☐
	Does the client/method/SOP require that the analyst check the sample pH and if necessary alter it?	☐	☐	☐
12	**Tubes:** Are the tubes capped and intact?	☐	☐	☐
	Do they contain moisture?	☐	☐	☐
13	**Badges:** Are the badges properly capped and intact?	☐	☐	☐
	Are dual bed badges separated and individually capped and intact?	☐	☐	☐

Lab Sample ID	Container Description	Required pH *	Received pH	Adjusted pH	VOA Headspace (Presence/Absence)	Receipt / Preservation Comments

Explain any discrepancies: (include lab sample ID numbers): _____

RSK - MEEPP, HCL (pH<2); RSK - CO2, (pH 5-8); Sulfur (pH>4)

Attachment 3

Service Request Form

Service Request Summary

Folder #: P1305682
Client Name: ALS Environmental - Simi Valley
Project Name: IHPAT Round 196
Project Number:

Report To: Chaney Humphrey
ALS Environmental - Simi Valley
2655 Park Center Drive, Suite A
Simi Valley, CA 93065
Phone Number: 805-526-7161
Cell Number:
Fax Number:
E-mail: chaney.humphrey@alsglobal.com

Project Chemist: Chaney Humphrey
Originating Lab: SIMIVALLEY
Logged By: MZAMORA
Date Received: 12/23/13
Internal Due Date: 1/24/14
QAP: LAB QAP
Qualifier Set: CAS Standard
Formset: CAS Standard
Merged?: Y
Report to MDL?: N
P.O. Number:
EDD: No EDD Specified

9 - 1 each-Tube Charcoal (50/100)

Location: P-04

Lab Samp No.	Client Samp No.	Matrix	Collected	SVG: OSHA 07/ OV C Tube CS2
P1305682-001	MOH-196-1	Air	12/19/13 0000	I
P1305682-002	MOH-196-2	Air	12/19/13 0000	I
P1305682-003	MOH-196-3	Air	12/19/13 0000	I
P1305682-004	MOH-196-4	Air	12/19/13 0000	I
P1305682-005	Blank	Air	12/19/13 0000	I
P1305682-006	Blank	Air	12/19/13 0000	I
P1305682-007	Blank	Air	12/19/13 0000	I
P1305682-008	Blank	Air	12/19/13 0000	I
P1305682-009	Blank	Air	12/19/13 0000	I

Folder Comments:

Industrial Hygiene PT Samples (PAT Round)

Test Comments:

Group	Test/Method	Samples	Comments
Semivoa GC	OV C Tube CS2/OSHA 07	1-9	Methanol

Attachment 4

Sample Login Verification Form

Sample Login and Verification Checklist

Service Request Number, Client & Project Name	(place folder label here)	SDG Used	PM

Sample(s) delivered by: (circle) Client / ALS Emp. / DHL / GSO / FedEx / UPS / Other_____

Yes	No	N/A	SMO Verification	
			Project number has been correctly entered.	
			Sample IDs from the COC have been correctly entered.	
			Sample date and time collected for each sample has been entered correctly.	
			Date received is correct.	
			Container tags are reconciled and applied to correct containers.	By:
			Container tags have been verified by a second person.	By:
			The analyst and PM have been alerted of Short HT or Rush samples.	Notified:
			Sample receipt discrepancies have been noted on Sample Acc. Check Form.	
			Login Completed	By: Date:

Yes	No	N/A	Client Services Login Verification	
			Folder due date is correct.	
			Project Number, Dates, Times, and Sample IDs are correct.	
			Pricing and Rush charges are correct.	
			The subcontract containers have been tagged and sub COC has been generated.	Sub Lab:
			Samples requiring an MS/MSD are properly indicated in the folder.	
			All non-analytical tasks (encores, EDDs, etc.) are logged in and priced correctly.	
			Client has been notified regarding holding time exceedences and sample receipt discrepancies. Notified by email □ verbally □ voicemail □	By: Date:
			Login Approved (red button)	By: Date:

Yes	No	N/A	Client Services Folder Approval	
			Pricing is correct and approved. (Prepaid work is properly indicated with check or credit card.)	
			Hazardous waste designation has been set properly for each sample.	
			Report and/or EDD are complete.	
			Folder Release	By: Date:

Comments:

Attachment 5

Sample Acknowledgement Form

ACCREDITED IN ACCORDANCE WITH nelac

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

Confirmation of Sample Receipt

To:	Chaney Humphrey	From:	Chaney Humphrey
Email:	chaney.humphrey@alsglobal.com	Email:	Chaney.Humphrey@alsglobal.com
Fax:		Fax:	805-526-7270
Phone:	805-526-7161	Phone:	805-526-7161 x231

Samples for analysis have been received by ALS Environmental on 12/23/13 and assigned our Service Request number **P1305682**. **Please verify the following information and notify me of any corrections as soon as possible.**

The estimated completion date for this work is: 2/3/14

Client: ALS Environmental - Simi Valley
Project: IHPAT Round 196

EDD Required: No

Tier: I

Report To: Chaney Humphrey
ALS Environmental - Simi Valley
2655 Park Center Drive, Suite A
Simi Valley, CA 93065

Billing Address: Chaney Humphrey
ALS Environmental - Simi Valley
2655 Park Center Drive, Suite A
Simi Valley, CA 93065

Comments: Industrial Hygiene PT Samples (PAT Round)

Thank you for your business!

A - Test is Authorized H - Test is On Hold HP - Test is On Hold Pending Input P - Test is Authorized for Prep Only C - Test has been Cancelled * - Test has assigned QC

				OSHA 07 OV C Tube CS2
P1305682-001	MOH-196-1	Air	12/19/13 0000	A
P1305682-002	MOH-196-2	Air	12/19/13 0000	A
P1305682-003	MOH-196-3	Air	12/19/13 0000	A
P1305682-004	MOH-196-4	Air	12/19/13 0000	A
P1305682-005	Blank	Air	12/19/13 0000	A
P1305682-006	Blank	Air	12/19/13 0000	A
P1305682-007	Blank	Air	12/19/13 0000	A
P1305682-008	Blank	Air	12/19/13 0000	A
P1305682-009	Blank	Air	12/19/13 0000	A

Test Comments:

Group	Test/Method	Samples	Comments
Semivoa GC	OSHA 07/OV C Tube CS2	1-9	Methanol