DEPARTMENT OF THE ARMY
OFFICE OF THE ASSISTANT CHIEF OF STAFF FOR INSTALLATION MANAGEMENT
600 ARMY PENTAGON
WASHINGTON, DC 20310-0600

January 11, 2017

MEMORANDUM FOR: DIEDRE LLOYD (USEPA-Region 4) and JAMIE WOODS (TDEC)

SUBJECT: Uniform Federal Policy-Quality Assurance Project Plan Membrane Interface Probe Survey Work Plan, Dunn Field, Defense Depot Memphis, Tennessee, Rev. 1

1. The purpose of this memorandum is to submit the Uniform Federal Policy-Quality Assurance Project Plan Membrane Interface Probe Survey Work Plan, Dunn Field, Defense Depot Memphis, Tennessee, Rev. 1. The report has been revised to incorporate the approved responses to comments from USEPA and TDEC.

2. For additional information please contact the Trinity ADC project manager, Mr. Todd Calhoun, at (850)588-1001, (tcalhoun@trinityadc.com).

Joan Hutton for

JAMES C. FOSTER
Program Manager,
Base Realignment and Closure Division

REV. 1

Uniform Federal Policy-Quality Assurance Project Plan
Membrane Interface Probe Survey Work Plan
Dunn Field
Defense Depot Memphis, Tennessee

Prepared for:

Department of the Army

Under Contract to:

U.S. Army Corps of Engineers
Mobile District
109 St. Joseph Street
Mobile, Alabama 36602

Prepared by:

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Shalimar, Florida 32579
850-613-6800

Contract No. W9128F-11-D-0029, Task Order CK01

December 2016

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Attachment 4 Responses to USEPA and TDEC Comments

Acronyms and Abbreviations

ACSIM-ODB	Assistant Chief of Staff for Installation Management, Base Realignment and Closure Division
AS	air sparge
BEC	BRAC Environmental Coordinator
bls	below land surface
BRAC	Base Realignment and Closure
CALIBRE	CALIBRE Systems, Inc.
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CESAM	U.S. Army Corps of Engineers, Mobile District
CLP	Contract Laboratory Program
CTL	CT Laboratories, LLC
CVOC	chlorinated volatile organic compound
DCE	dichloroethene
DDMT	Defense Depot Memphis, Tennessee
DL	detection limit
DoD	Department of Defense
DOT	Department of Transportation
DPT	direct push technology
DQI	data quality indicator
DQO	data quality objective
e2M	engineering-environmental Management, Inc.
ECD	electron capture detector
EDD	electronic data deliverable
ELAP	Environmental Laboratory Accreditation Program
FD	field duplicate
FID	flame ionization detector
FTL	Field Team Leader
GC/MS	gas chromatograph/mass spectrometer
HAZWOPER	hazardous waste operations
HDR	HDR Inc.
HSO	Health and Safety Officer
ICAL	initial calibration
ICV	initial calibration verification
IDW	investigation derived waste
IRA	interim remedial action
LCL	lower confidence limit

LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LDC	Laboratory Data Consultants, LLC
LIMS	Laboratory Information Management System
LOD	limit of detection
LOQ	limit of quantitation
LTM	long-term monitoring
LUC	Land Use Control
MCL	maximum contaminant level
MDL	method detection limit
MIP	membrane interface probe
MPC	measurement performance criteria
MS	matrix spike
MSD	matrix spike duplicate
N/A	not applicable or not available
OSHA	Occupational Safety and Health Administration
PARCCS	precision, accuracy, representativeness, comparability, completeness, and sensitivity
PCE	tetrachloroethene
PID	photoionization detector
PLS	professional land surveyor
PPE	personal protective equipment
ppm	parts per million
QA	quality assurance
QC	quality control
QSM	Quality Systems Manual
RA	remedial action
RB	equipment rinsate blank
RI	Remedial Investigation
RPD	relative percent difference
RSD	relative standard deviation
RT	retention time
RW	recovery well
SB	soil boring
SDG	sample delivery group
SOP	standard operating procedure
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
TBD	to be determined

TCE	trichloroethene
TCLP	toxicity characteristic leaching procedure
TDEC	Tennessee Department of Environment and Conservation
Trinity	Trinity Analysis & Development Corp.
UCL	upper confidence limit
UFP-QAPP	Uniform Federal Policy-Quality Assurance Project Plan
USEPA	U.S. Environmental Protection Agency
VOC	volatile organic compound
WC	waste characterization
ZVI	zero valent iron

Worksheet 1 - Title and Approval Page

Document Title:	Uniform Federal Policy-Quality Assurance Project Plan (UFP-QAPP) Membrane Interface Probe (MIP) Survey Defense Depot Memphis, Tennessee (DDMT) Shelby County, Tennessee
Lead Organization:	Department of the Army
Lead Regulatory Organization:	U.S. Environmental Protection Agency (USEPA) Region 4 Tennessee Department of Environment and Conservation (TDEC)
Preparer's Name and Organizational Affiliation:	Todd Calhoun, Trinity Analysis & Development Corp. (Trinity)
Preparer's Contact Information:	1002 N. Eglin Pkwy Shalimar, Florida 32579 850-588-1001 tcalhoun@trinityadc.com
Preparation Date:	December 2016

Todd Calhoun — 12/22/2016

Todd Calhoun, PG
Trinity Project Manager

Date

Laura Roebuck — 12/22/2016

Laura Roebuck
U.S. Army Corps of Engineers, Mobile District (CESAM) Technical Manager

Date

Carolyn Jones — 12/22/2016

Carolyn Jones
Assistant Chief of Staff for Installation Management, Base Realignment and Closure Division (ACSIM-ODB) Program Manager

Date

Diedre Lloyd — 1/10/2017

Diedre Lloyd
USEPA Region 4 Remedial Project Manager

Date

Jamie Woods — 10/21/2016

Jamie Woods
TDEC Remedial Project Manager

Date

Worksheet 2 - QAPP Identifying Information

Project Name:	Membrane Interface Probe Survey Dunn Field, Defense Depot Memphis, Tennessee
Site Location:	Memphis, Shelby County, Tennessee
Site Number/Code:	TN4210020570
Contractor Name:	Trinity Analysis & Development Corp. (Trinity)
Contract Number:	W9128F-11-D-0029
Work Assignment Number:	Task Order CK01
Task Order Title:	Environmental Restoration Support 2016 Defense Depot Memphis, Tennessee (DDMT)
Guidance Used to Prepare UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans Part 1: UFP-QAPP Manual, EPA-500-B-04-900A, Intergovernmental Data Quality Task Force, March 2005
Dates of Scoping Sessions	No task specific scoping sessions were held. Monthly project team calls held to provide updates as necessary.
Identify Regulatory Programs:	Comprehensive Environmental Response, Compensation, and Liability Act, as amended by the Superfund Amendments and Reauthorization Act (CERCLA) National Priorities List
Generic or Project Specific QAPP:	This is a project-specific QAPP
List organizational partners (stakeholders) and connection with lead organization:	USEPA Region 4 TDEC CESAM
List Data Users:	ACSIM-ODB, U.S. Army Corps of Engineers, Mobile District (CESAM), USEPA Region 4, TDEC, Trinity
List dates and titles of work plan documents written for previous site work, if applicable:	Dunn Field Record of Decision (CH2M Hill, 2004) Memphis Depot Dunn Field Source Areas Final Remedial Design (CH2M Hill, 2007) *Dunn Field Record of Decision Amendment* (engineering-environmental Management, Inc. [e2M], 2009a) Remedial Action Operations and Long Term Monitoring Quality Assurance Project Plan (HDR Inc. [HDR], 2014) Analysis of Tennessee Department of Environment and Conservation (TDEC) Environmental Reports to Evaluate the Source of Chlorinated Solvents in Dunn Field Upgradient Wells (CALIBRE Systems, Inc. [CALIBRE], 2015 Annual Long-Term Monitoring Report – 2015 Defense Depot Memphis, Tennessee (HDR, 2016)
Preparation Date:	December 2016

QAPP Element(s) and Corresponding QAPP section(s)	Required Information	Crosswalk to UFP-QAPP Worksheet #
Project Management and Objectives		
2.1 Title and Approval Page	- Title and Approval Page	Worksheet 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	 Worksheet 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	 Worksheet 3 Worksheet 4; Table 1
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 - Special Personnel Training Requirements	 Worksheet 5; Figure 1 Worksheet 6, Table 2 Worksheet 7, Table 3 Worksheet 8; Table 4
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 - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site maps	 Worksheet 9 Worksheet 10; Figures 2-8
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 Project Quality Objectives - Measurement Performance Criteria Table	 Worksheet 11; Table 6 Worksheet 12; Table 7
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table	Worksheet 13; Table 8
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	Worksheet 14 Worksheet 15; Table 9 Worksheet 16; Table 10

Measurement/Data Acquisition		
3.1 Sampling Tasks 3.1.1 Sampling Process Design and Rationale	- Sampling Design and Rationale	Worksheet 17
3.1.2 Sampling Procedures and Requirements	- Sample Location Map	Figure 9 (Site Layout Map), Worksheet 14
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 Locations and Methods/ Standard Operating Procedure (SOP) Requirements Table - Analytical Methods/SOP Requirements Table - Field Quality Control (QC) Sample Summary Table - Sampling SOPs - Field Equipment Calibration, Maintenance, Testing, and Inspection Table	Worksheet 18; Table 11; Field SOPs in Attachment 1; Site Safety and Health Plan (SSHP) (separate document) Worksheet 19; Table 12 Worksheet 20; Table 13 Worksheet 21; Table 12; Field SOPs in Attachment 1 Worksheet 22; Table 15
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 SOPs References table - Analytical Instrument Calibration Table - Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	Worksheet 23; Table 16; Analytical SOPs in Attachment 3. Worksheet 24; Table 17, Worksheet 25; Table 18
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	Worksheet 26; Table 19; Field SOPs in Attachment 1 Worksheet 27; Field Forms in Attachment 2
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	Worksheet 28 (Lab QC samples) Not required for this project.

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	Worksheet 29; Table 20; Field Forms in Attachment 2 Worksheet 30 Worksheet 14 and 29
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 Actions Responses	Worksheet 31; Table 22 Worksheet 32; Table 23
4.2 Quality Assurance Management Reports	- Quality Assurance Management Reports Table	Worksheet 33; Table 24
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	Worksheet 34; Table 25 Worksheet 35; Table 26 Worksheet 36; Table 27-28 Worksheet 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	Not applicable	Not applicable

Worksheet 3 - Distribution List

Document Title:	Uniform Federal Policy-Quality Assurance Project Plan (UFP-QAPP) Membrane Interface Probe Survey, Defense Depot Memphis, Tennessee, Shelby County, Tennessee
Contract Number:	W9128F-11-D-0029/CK01

Recipient	Title	Organization	Telephone Number	Email Address
Carolyn Jones	Program Manager	ACSIM-ODB	703-545-2508	carolyn.a.jones28.civ@mail.mil
Joan Hutton	BRAC Environmental Coordinator (BEC)	CALIBRE Systems, Inc. (CALIBRE)	770-317-4323	joan.hutton@calibresys.com
Laura Roebuck	CESAM Technical Manager	CESAM	251-690-3480	laura.w.roebuck@usace.army.mil
Diedre Lloyd	Remedial Project Manager	USEPA Region 4	404-562-8855	lloyd.diedre@epa.gov
Jamie Woods	Remedial Project Manager	TDEC Division of Remediation	901-371-3041	jamie.woods@tn.gov
Todd Calhoun	Project Manager	Trinity	850-588-1001	tcalhoun@trinityadc.com
Robyn Peterson	Project Engineer	Trinity	850-613-6800	rpeterson@trinityadc.com
Tom Holmes	Project Manager	HDR	404-295-3279	thomas.holmes@hdrinc.com
Project File	---	Trinity	---	---

Worksheet 4 - Project Personnel Sign-Off Sheet

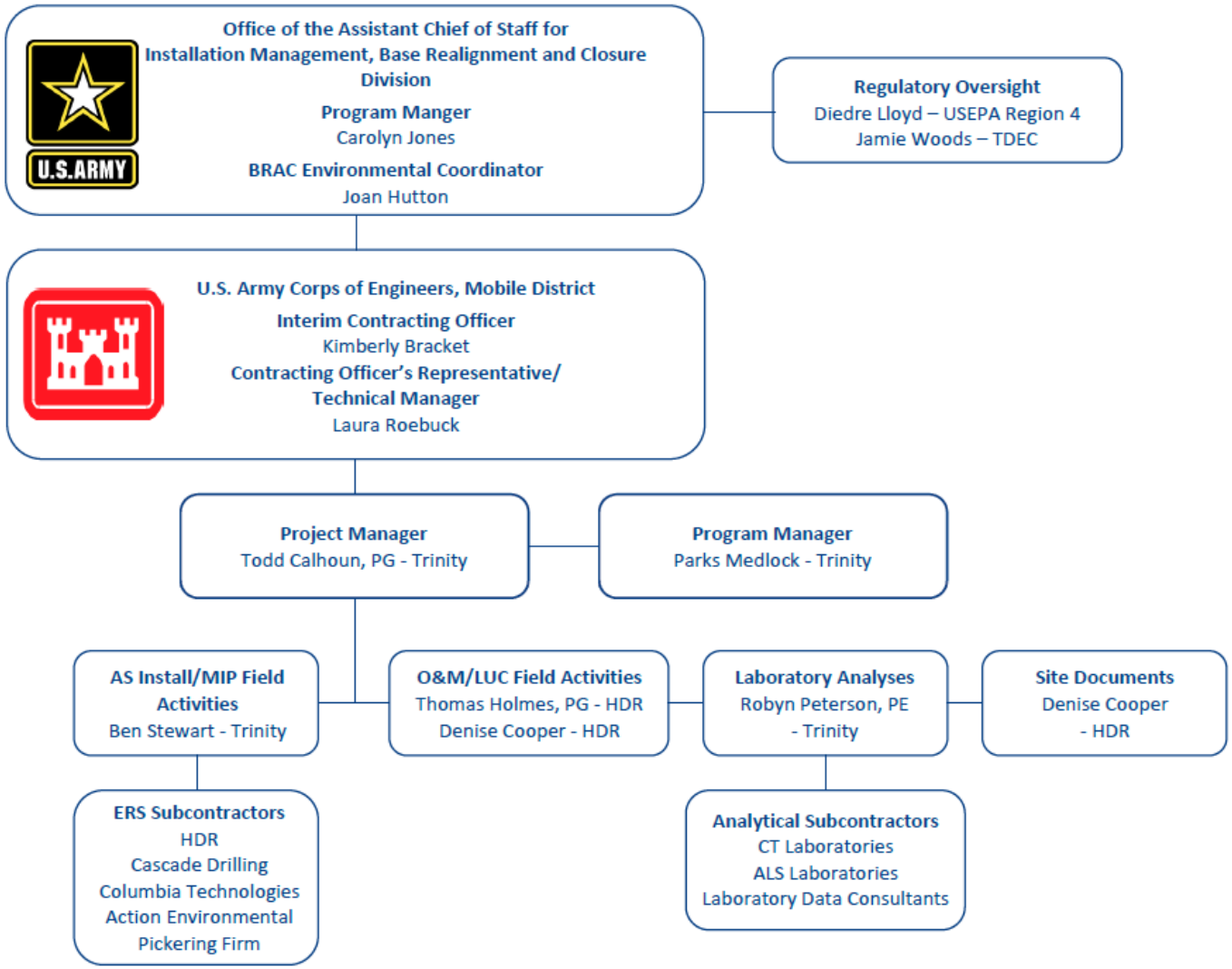
Copies of this form to be signed by key project personnel from each organization to indicate that they have read the applicable sections of this UFP-QAPP and will perform the tasks as described.

Table 1 Personnel Sign-Off Sheet

Project Personnel	Project Title	Telephone Number	Signature/Date QAPP Read Email Receipt
ACSIM-ODB			
Carolyn Jones	Program Manager	703-545-2508	
BEC			
Joan Hutton	BRAC Environmental Coordinator	770-317-4323	
CESAM			
Laura Roebuck	CESAM Technical Manager	251-690-3480	
USEPA Region 4			
Diedre Lloyd	USEPA Remedial Project Manager	404-562-8855	
TDEC			
Jamie Woods	TDEC Remedial Project Manager	901-371-3041	
Trinity			
Parks Medlock	Program Manager	850-588-0706	
Todd Calhoun	Project Manager	850-588-1001	
Robyn Peterson	Project Engineer	850-613-6800	
Ben Stewart	Project Geologist/Field Team Leader (FTL)/Site Safety and Health Officer (SSHO)	850-312-6576	
Jeanette Baldwin	Corporate Health and Safety Officer (HSO)	850-547-6243	

Worksheet 5 - Project Organizational Chart

Figure 1 Project Organizational Chart



Worksheet 6 - Project Communication Pathways

Table 2 Communication Pathways

Communication Drivers	Responsible Entity	Name	Contact Information	Procedure (Timing, Pathways, Documentation, etc.)
Contract Execution	CESAM Technical Manager	Laura Roebuck	laura.w.roebuck@usace.army.mil 251-690-3480	Email/communication with Trinity Project Manager.
Manage all Task Order phases	Trinity Project Manager	Todd Calhoun	tcalhoun@trinityadc.com 850-588-1001	All project information will be copied to the BEC and CESAM Technical Manager. Trinity Project Manager will notify BEC and CESAM Technical Manager of field related problems by phone, email, or fax by close of business the day of the event if possible and no later than noon Central Daylight/Standard Time the following day.
Regulatory agency interface	BEC	Joan Hutton	joan.hutton@calibresys.com 770-317-4323	Coordination and communication with regulatory agencies will be completed by the BEC. All regulatory interactions will be documented.
Field progress reports	Trinity FTL/ Trinity Project Manager	Ben Stewart/ Todd Calhoun	bstewart@trinityadc.com 251-709-6509 (cell) tcalhoun@trinityadc.com 850-588-1001	Daily Quality Control Reports (DQCRs) will be prepared by the FTL and provided to the Trinity Project Manager for review and issuance to the BEC and CESAM Technical Manager.
Field corrective actions	Trinity FTL/ Trinity Project Manager	Ben Stewart/ Todd Calhoun	bstewart@trinityadc.com 251-709-6509 (cell) tcalhoun@trinityadc.com 850-588-1001	Corrective actions will be issued in writing by the FTL to the Project Manager for review and approval.
QAPP changes prior to field work	Trinity FTL/ Trinity Project Manager	Ben Stewart/ Todd Calhoun	bstewart@trinityadc.com 251-709-6509 (cell) tcalhoun@trinityadc.com 850-588-1001	Change pages for the QAPP will be issued to all stakeholders via email) for approval and followed up by hard copy, where applicable.

Communication Drivers	Responsible Entity	Name	Contact Information	Procedure (Timing, Pathways, Documentation, etc.)
QAPP changes during project execution	Trinity FTL/ Trinity Project Manager	Ben Stewart/ Todd Calhoun	bstewart@trinityadc.com 251-709-6509 (cell) tcalhoun@trinityadc.com 850-588-1001	Change pages for the QAPP will be issued to all stakeholders via email for approval and followed up by hard copy, where applicable.
Laboratory QC variances	Trinity Project Engineer	Robyn Peterson	rpeterson@trinityadc.com 850-613-6800	The laboratory will be required to repeat the determination of the limit of detection (LOD) if there are significant changes to the method or instrumentation prior to analysis of the first sample. The limit of quantitation (LOQ) will be verified quarterly; if the method is modified or major changes made to the instrumentation, the LOQ will be verified and reported
Analytical corrective action	Trinity Project Engineer	Robyn Peterson	rpeterson@trinityadc.com 850-613-6800	Determines the need for corrective action for analytical issues; reviews data and technical deliverables as needed.
Data verification issues	Trinity Project Engineer	Robyn Peterson	rpeterson@trinityadc.com 850-613-6800	Confirms that scientifically sound data is used in making project decisions via a three step data review.
Data validation issues	Trinity Project Engineer/ Laboratory Data Consultants, LLC (LDC) Project Manager	Robyn Peterson/ Stella Cuenco	rpeterson@trinityadc.com 850-613-6800 scuenco@lab-data.com 760-827-1100	Evaluate whether the collected data comply with project requirements by comparing the data collected with criteria established based on data quality objectives (DQOs).
Data review corrective action	Trinity Project Engineer	Robyn Peterson	rpeterson@trinityadc.com 850-613-6800	If corrective action is deemed necessary, the review of supporting raw data to verify accuracy may be involved.

Communication Drivers	Responsible Entity	Name	Contact Information	Procedure (Timing, Pathways, Documentation, etc.)
Health and Safety issues	Trinity FTL/ Trinity Corporate HSO	Ben Stewart/ Jeanette Baldwin	bstewart@trinityadc.com 251-709-6509 (cell) jbaldwin@trinityadc.com 850-547-6243	The on-site FTL/SSHO will verbally report any issue to the HSO and notify the CESAM Technical Manager verbally, at a minimum. An incident form must be completed within 24 hours by the SSHO/FTL and submitted to the Trinity HSO for review and approval.
Stop Work Authority	All Site Workers		jbaldwin@trinityadc.com 850-547-6243	All site workers can issue a stop work order for issues that present immediate and imminent danger. The HSO will be consulted after the Stop Work verbally and then with a follow-up documented report per the Site Safety and Health Plan (SSHP).

Worksheet 7 - Personnel Responsibilities

Table 3 Personnel Qualifications

Project Personnel	Project Title	Organizational Affiliation	Responsibilities	Education and Experience
Joan Hutton	BEC	CALIBRE	Oversees project and responds to USEPA and TDEC	Master of Science, Marine Science, 30 yrs. Experience
Parks Medlock	Program Manager	Trinity	Contract management and provides resource support	Bachelor of Science, Chemistry, 23 yrs. Experience
Todd Calhoun, PG	Project Manager	Trinity	Manages project and provides technical direction	Bachelor of Science, Geology, 21 yrs. Experience
Robyn Peterson, PE	Project Engineer	Trinity	Coordinates analytical and data validation	Bachelor of Science, Biological Engineering, 21 yrs. Experience
Ben Stewart		Trinity	Supervises field activities	Bachelor of Science, Geology, 5 yrs. Experience
Brent Szymanski		CT Laboratories, LLC	Manages laboratory analyses	Bachelor of Arts, Management & Human Resources, 8 yrs. Experience
Stella Cuenco		LDC	Conducts independent analytical data validation	Bachelor of Science, Chemistry, 25 yrs. Experience

Worksheet 8 - Special Personnel Training Requirements Table

Table 4 Special Personnel Training Requirements

Project Function	Specialized Training – Title or Description of Course	Training Provider	Training Date	Personnel/ Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/ Certificates
Field Investigation Activities	40-Hour Hazardous Waste Operations (HAZWOPER) Training	Cooey Environmental	03/20/2011	Ben Stewart	FTL	Trinity Shalimar, Florida
	8-Hour HAZWOPER Refresher	U.S. Air Force	02/23/2016			
	8-Hour Occupational Safety and Health Administration (OSHA) Supervisor Training	ABAG Training Center	05/14/2013			
	First Aid/Cardiopulmonary Resuscitation	American Red Cross	06/26/2016			
	OSHA Excavation Safety Training for Competent Persons	ABAG Training Center	04/24/2015			
	Department of Transportation (DOT) HazMat Carrier Requirements (Highway)	Compliance Training Online	10/21/2013			

Worksheet 9 - Project Scoping Session Participants Sheet

Routine monthly team meetings are held with the following participants. Agendas and post-meeting notes are submitted to the team members.

Name	Organization	Title/Role	Email/Phone
Carolyn Jones	ACSIM-ODB	Program Manager	carolyn.a.jones28.civ@mail.mil 703-545-2508
Joan Hutton	CALIBRE	BEC	joan.hutton@calibresys.com 770-317-4323
Laura Roebuck	CESAM	Technical Manager	laura.w.roebuck@usace.army.mil 251-690-3480
Diedre Lloyd	USEPA Region 4	Remedial Project Manager	lloyd.diedre@epa.gov 404-562-8855
Jamie Woods	TDEC	Remedial Project Manager	jamie.woods@tn.gov 901-371-3041
Todd Calhoun	Trinity	Project Manager	tcalhoun@trinityadc.com 850-588-1001
Tom Holmes	HDR	Project Manager	thomas.holmes@hdrinc.com 404-295-3279

Worksheet 10 - Problem Definition

Site Location and History

DDMT is in southeastern Memphis, Shelby County, Tennessee approximately 5 miles east of the Mississippi River and 2 miles north of Memphis International Airport (**Figure 2**). DDMT originated as a military facility in the early 1940s. It received, warehoused, and distributed supplies common to all United States military services and some civil agencies located primarily in the southeastern United States, Puerto Rico, and Panama. Stocked items included food, clothing, petroleum products, construction materials, and industrial, medical, and general supplies. In 1995, DDMT was placed on the list of the Department of Defense (DoD) facilities to be closed under BRAC. Storage and distribution of material continued until the facility closed in September 1997.

The property consists of approximately 632 acres and includes the Main Installation and Dunn Field. The Main Installation covers approximately 567 acres and had open storage areas, warehouses, military family housing, and outdoor recreational areas. Dunn Field, which is located across Dunn Avenue from the north-northwest portion of the Main Installation, covers approximately 65 acres and had mineral storage and waste disposal areas (HDR, 2015). The northeastern portion of Dunn Field is the study area for this investigation (**Figure 3**).

In October 1992, DDMT was added to the National Priorities List (57 Federal Register 47180 No. 199). Responsibility for environmental restoration at DDMT transferred from the Defense Logistics Agency to the Department of the Army in December 2010. The regulatory oversight agencies are USEPA Region 4 and TDEC.

The R*ecord 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 along the western boundary of Dunn Field. The system became operational in November 1998. Based on reduction in CVOC concentrations in groundwater following implementation of the Dunn Field Source Areas remedial action (RA), five RWs were shut down in June 2009 and the remaining RWs were shutdown in January 2009. The IRA system was removed and the RWs abandoned in July 2010.

The groundwater remedial action objectives (RAOs) established in the *Dunn Field Record of Decision* (CH2M Hill, 2004) are:

- to prevent human exposure to contaminated groundwater (i.e., exceeding protective target concentrations)
- to prevent further off-site migration of volatile organic compounds (VOCs) in excess of protective target levels
- to remediate Fluvial Aquifer groundwater to drinking water quality to be protective of the deeper Memphis Aquifer

The remedies were implemented in three phases: Disposal Sites, Source Areas, and Off Depot. The selected remedies for the Source Areas and Off Depot were modified through the *Dunn Field Record of Decision Amendment* (e2M, 2009a).

Disposal Sites RA included excavation and off-site disposal of soil and waste material from five sites and was completed in 2006.

Source Areas RA included soil vapor extraction (SVE) in the vadose zone and injection of zero valent iron (ZVI) in groundwater. The Fluvial SVE system was operated from July 2007 to July 2012 and removed approximately 4,000 pounds of VOCs. The Fluvial SVE system was shut down after soil remediation goals were met. Thermal SVE was performed in the loess from May to December 2008 and removed approximately 12,500 pounds of VOCs. ZVI injection was not required due to success of SVE in reducing groundwater impacts. Excavation and off-site disposal of soil and waste material in two additional areas were also conducted in the Source Areas RA.

The Off Depot RA included installation of an air sparge (AS)/SVE system and implementation of Land Use Controls (LUCs) on Dunn Field. The AS/SVE system with 90 AS points and 12 SVE wells began operation in December 2009. LUCs were implemented through deed restrictions, zoning regulations, and Notice of Land Use Restrictions recorded in June 2009, and annual inspections since 2009. The AS/SVE system was installed to reduce individual CVOC concentrations in the treatment area below 50 micrograms per liter (µg/L) and to continue operation until the upgradient concentrations of individual CVOCs in the Dunn Field plume do not exceed 50 µg/L. AS/SVE in combination with natural attenuation processes is expected to reduce groundwater concentrations to USEPA maximum contaminant levels (MCLs) in accordance with RAOs in the Dunn Field Record of Decision. From December 2009 through December 2015, it was estimated that the AS/SVE system had removed approximately 84 pounds of VOCs (HDR, 2016).

Previous Investigations

Long-term monitoring (LTM) of groundwater has resulted in detections of 1,1-dichloroethene (DCE), tetrachloroethene (PCE) and trichloroethene (TCE) above MCLs in background wells MW-07, MW-08, MW-129, MW-130, and MW-230. These monitoring wells are categorized as “Background-NE” and are located on or upgradient of the northeast section of Dunn Field. This area of Dunn Field is the target for this MIP Survey.

1,1-DCE, PCE, and TCE concentrations from the last four annual sampling events (2012 – 2015) for the five Background-NE monitoring wells are shown on **Figure 4.** Contaminant concentrations for PCE, TCE, and 1,1-DCE along with their MCLs from the April 2015 sampling event are provided in the table below. No other CVOCs were detected at concentrations above their MCLs. Total CVOC concentrations from the April 2015 sampling event are shown on **Figure 5**.

Table 5 Maximum CVOC Concentrations in Background-NE Monitoring Wells – April 2015

CVOC	MCL	Maximum Concentration	Location of Maximum Concentration
1,1-dichloroethene	7	17.2	MW-07
tetrachloroethene	5	43.8	MW-07
trichloroethene	5	64.7	MW-130
All units in micrograms per liter			

The Dunn Field Remedial Investigation (RI) included surface and subsurface soil sampling in a portion of Dunn Field identified as the “Northeast Open Area” to investigate several historic sites as possible sources of contaminant releases to the environment (CH2M Hill, 2002). VOCs were identified in surface and subsurface soils at locations west and southwest of the MIP Survey study area. TCE was reported at

a low level (estimated) in a shallow subsurface soil sample collected from a boring equivalent to the current location of MW-08. No soil samples were collected within the boundaries of the MIP Survey study area.

A review of boring logs show that soil cores collected during the installation of MW-129 and MW-130 in 2003 were screened for headspace readings with a photoionization detector (PID) but no samples were collected for laboratory analysis. Elevated headspace readings were noted in soil cores from both borings. Readings of up to 999 parts per million (ppm) were observed at depths up to 25 feet below land surface (bls) in MW-129 and up to 70 ppm at depths up to 20 feet bls in MW-130. The boring logs for the other Background-NE wells did not note headspace readings.

Data Gaps

CVOCs continue to be detected at concentrations above MCLs in monitoring wells that are upgradient or cross-gradient of areas where RAs have been implemented. The RI did not identify waste disposal or other activities that would impact soil or groundwater in the Northeast Open Area of Dunn Field and no investigation has been performed within the limits of the MIP Survey study area. TDEC has performed several investigations of off-site areas northeast (upgradient) of Dunn Field for the potential source of CVOCs in groundwater, but no source areas were clearly identified.

The primary CVOCs for Dunn Field have been identified as carbon tetrachloride, chloroform, 1,1-DCE, 1,1,2,2-tetrachloroethane, PCE, TCE, trans-1,2-DCE, cis-1,2-DCE, and vinyl chloride. Most notably, 1,1-DCE has been identified in groundwater samples collected from Background-NE monitoring wells located upgradient (off-site) and downgradient in the northern portion of Dunn Field; 1,1-DCE is not present in other groundwater CVOC contaminant plumes observed on Dunn Field suggesting the potential for an off-site source.

Discussion of the CVOCs in these wells in previous reports has focused on two concepts:

1. Since these CVOCs are present in wells upgradient of Dunn Field, there must be an off-site source NE of Dunn Field.
2. Since there were no reported environmental impacts in the NE area of Dunn Field, CVOC concentrations decrease downgradient from MW-130 and 1,1-DCE was not detected in soil or groundwater elsewhere on Dunn Field, the groundwater contamination in the Background-NE wells has been considered to result solely from the suspected off-site source.

In order to support the absence of a CVOC source in the NE area of Dunn Field contributing to the off-site groundwater plume, further investigation is needed.

The study area for this project is the northeastern portion of Dunn Field where the Background-NE wells are located. A MIP survey in this area will be conducted to determine if previously unidentified storage, burial or disposal areas may be contributing to the persistent groundwater contamination in the downgradient Background-NE wells (MW-07, MW-08, MW-230). Given the east to west groundwater flow direction, the data will also be used to determine if the groundwater contaminants identified in the upgradient Background-NE monitoring wells (MW-129, MW-130) originate from an unidentified source within the northeastern portion of Dunn Field or from an off-site source not attributable to past Army operations.

Project Objectives

Due to the lack of substantive soil data for the northeastern section of Dunn Field, the Department of the Army's objective of this investigation is to exercise due diligence to investigate the study area for potential unknown or unidentified source materials which may contribute to elevated CVOC concentrations in groundwater in this area of Dunn Field. The MIP Survey and associated soil sampling are being performed to achieve the goal of confirming the absence or presence of a contaminant source within the study area.

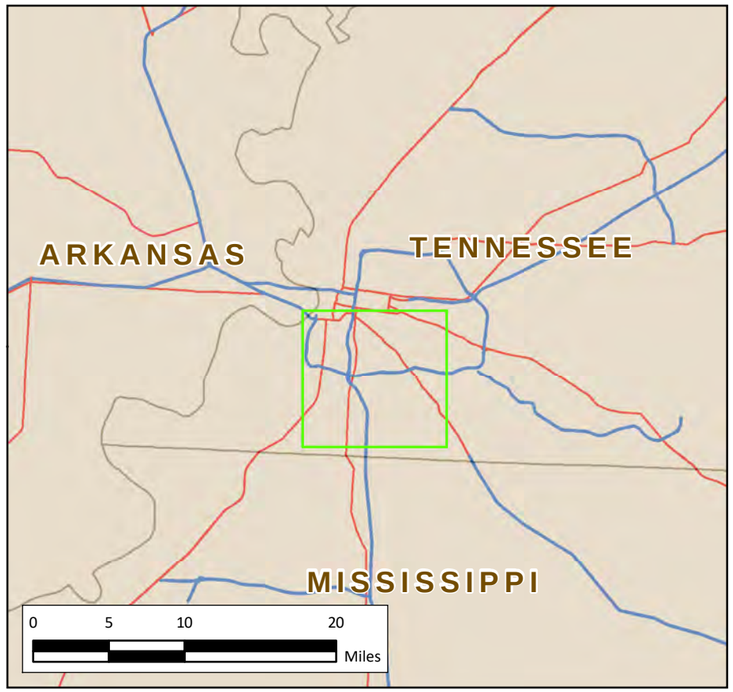
Topography, Geology, and Hydrogeology

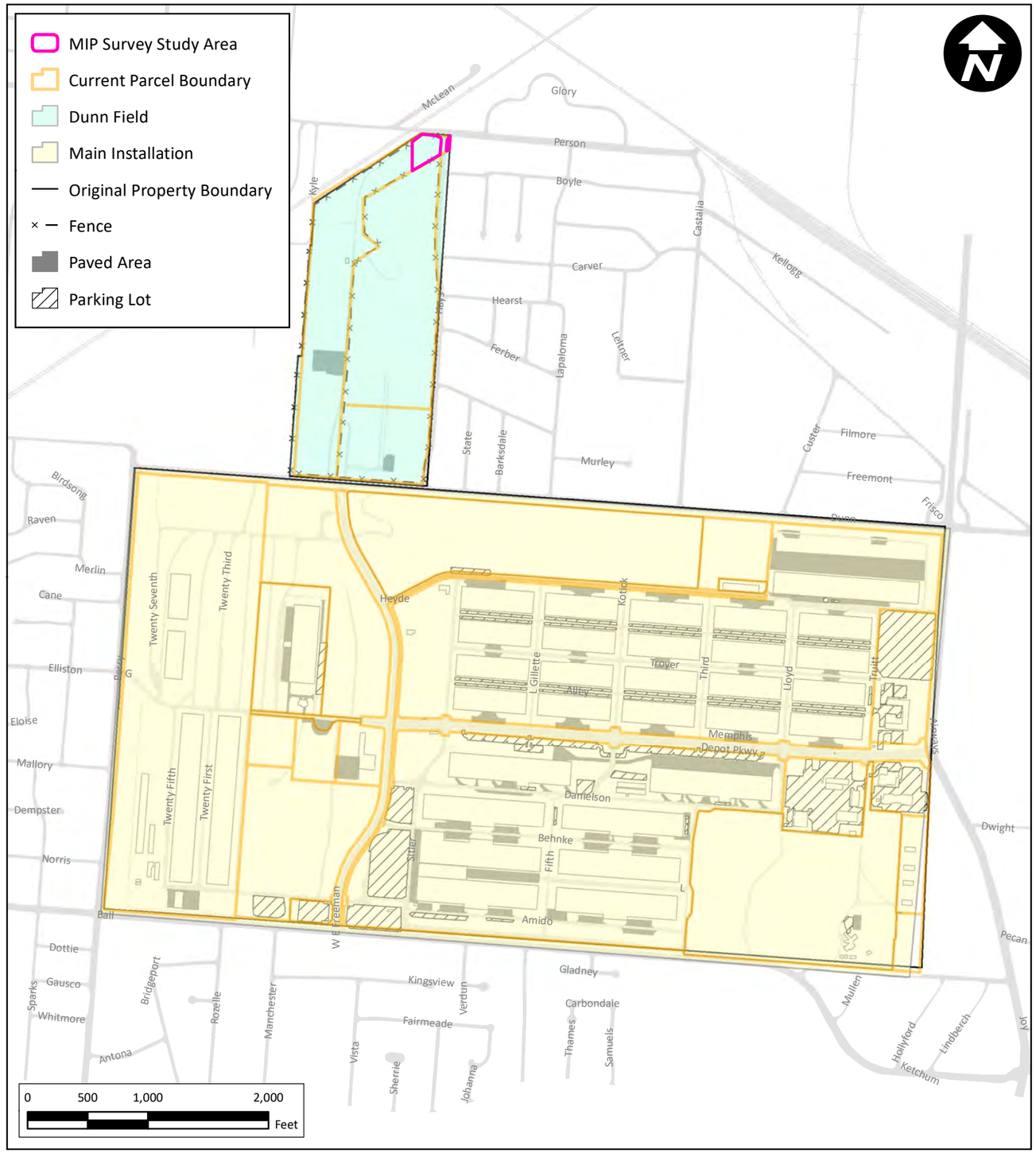
The Northeast Open Area of Dunn Field consists of mowed and wooded areas. The Dunn Field MIP study area ranges in surface elevation from approximately 286 to 272 feet above mean sea level with the lower elevations crossing through the center of the study area (**Figure 6)**.

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 unsaturated, about 20 to 30 feet thick, and are continuous throughout the Dunn Field area. In previous investigations, the fine-grained soil in the loess was found to bind CVOCs, release them over time and impact groundwater. The loess deposits are the target area of the MIP investigation.

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 interbedded 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. 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. Groundwater in the fluvial aquifer is not a drinking water source for area residents. A generalized lithologic cross-section of Dunn Field is provided as **Figure 7**. Groundwater flow direction of the unconfined fluvial aquifer is to the west and as depicted on **Figure 8**.

Additional details on previous investigations and site conditions can be found in HDR, 2015 and HDR, 2016.





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Trinity Analysis & Development Corp.

Figure 2
Site Location

Dunn Field
Defense Depot Memphis, Tennessee
Memphis, Shelby County, Tennessee

DRAWN BY:	DATE:	SCALE:	PROJECT NO:
JTC	11/18/2016	AS SHOWN	W9128F-11-D-0029 CK01

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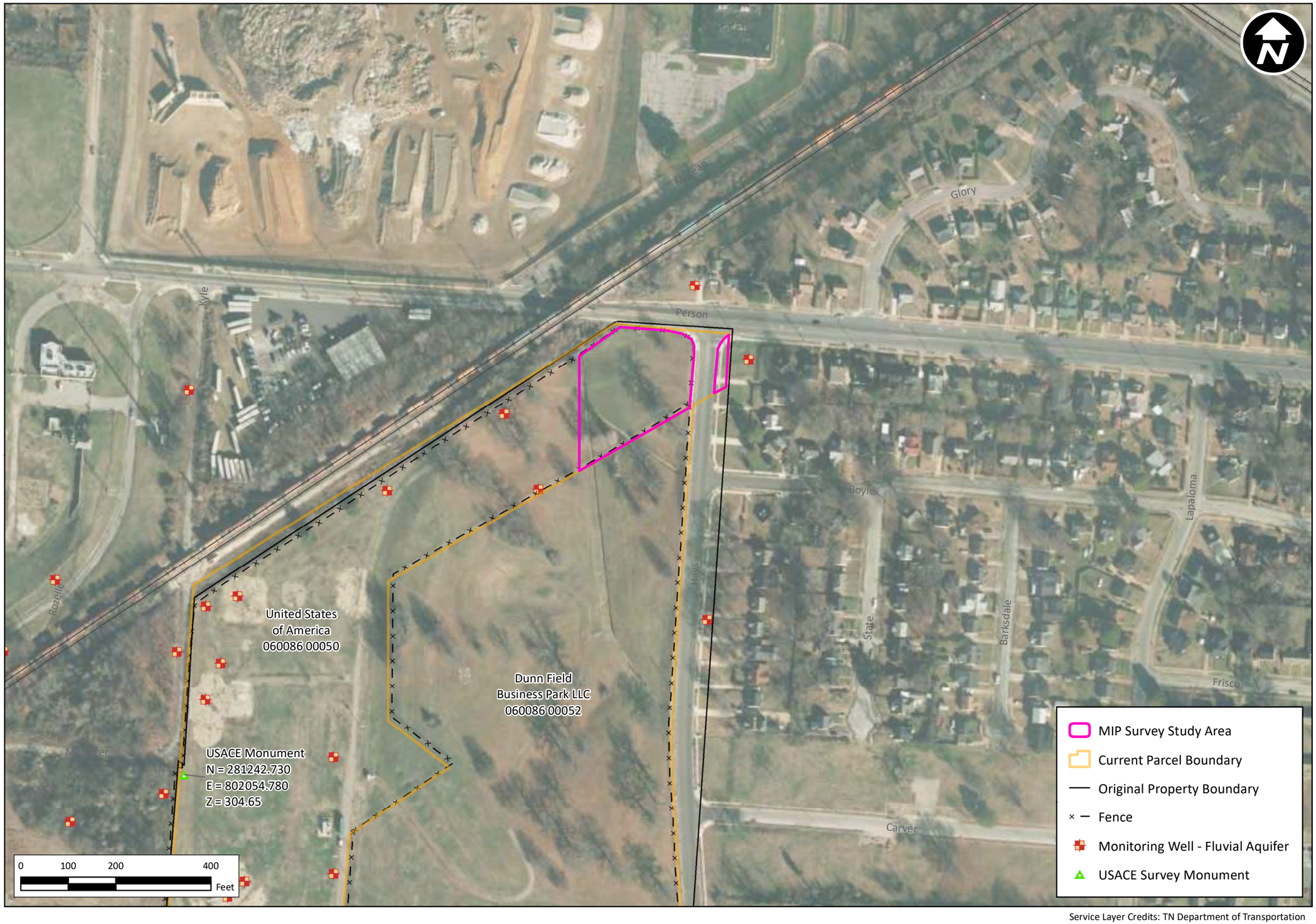

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Trinity Analysis & Development Corp.

Figure 3
Site Layout

Dunn Field
Defense Depot Memphis, Tennessee
Memphis, Shelby County, Tennessee

PROJECT NO:	DATE:	DRAWN BY:
W9128F-11-D-0029 CK01	11/18/2016	JTC

SCALE:
AS SHOWN

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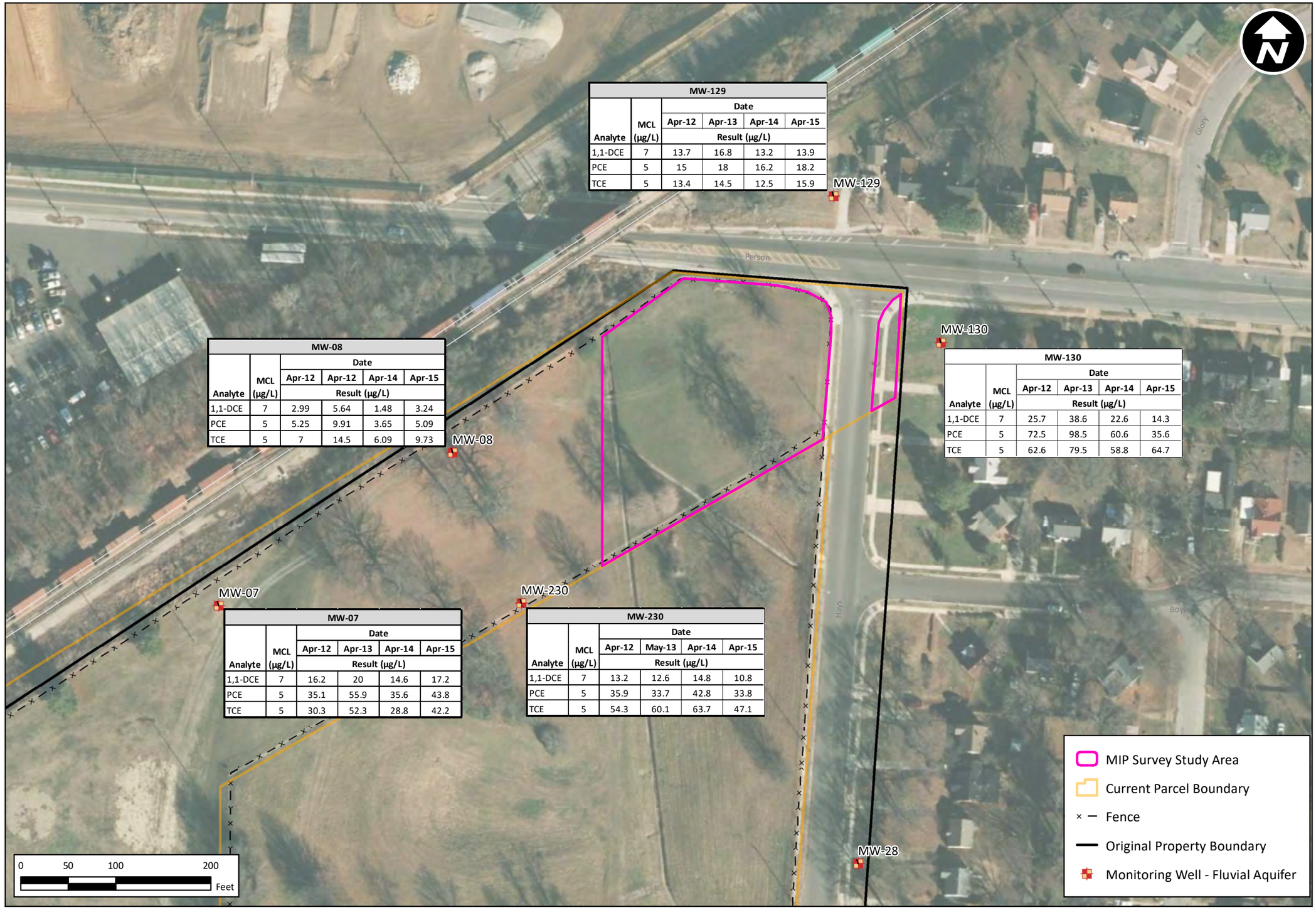

MW-129

Analyte	MCL (µg/L)	Date			
		Apr-12	Apr-13	Apr-14	Apr-15
		Result (µg/L)			
1,1-DCE	7	13.7	16.8	13.2	13.9
PCE	5	15	18	16.2	18.2
TCE	5	13.4	14.5	12.5	15.9

MW-08

Analyte	MCL (µg/L)	Date			
		Apr-12	Apr-12	Apr-14	Apr-15
		Result (µg/L)			
1,1-DCE	7	2.99	5.64	1.48	3.24
PCE	5	5.25	9.91	3.65	5.09
TCE	5	7	14.5	6.09	9.73

MW-130

Analyte	MCL (µg/L)	Date			
		Apr-12	Apr-13	Apr-14	Apr-15
		Result (µg/L)			
1,1-DCE	7	25.7	38.6	22.6	14.3
PCE	5	72.5	98.5	60.6	35.6
TCE	5	62.6	79.5	58.8	64.7

MW-07

Analyte	MCL (µg/L)	Date			
		Apr-12	Apr-13	Apr-14	Apr-15
		Result (µg/L)			
1,1-DCE	7	16.2	20	14.6	17.2
PCE	5	35.1	55.9	35.6	43.8
TCE	5	30.3	52.3	28.8	42.2

MW-230

Analyte	MCL (µg/L)	Date			
		Apr-12	May-13	Apr-14	Apr-15
		Result (µg/L)			
1,1-DCE	7	13.2	12.6	14.8	10.8
PCE	5	35.9	33.7	42.8	33.8
TCE	5	54.3	60.1	63.7	47.1

Service Layer Credits: TN Department of Transportation; HDR, 2016

Trinity Analysis & Development Corp.

Figure 4
Background-NE Monitoring Well
CVOC Concentrations, 2012 - 2015

Dunn Field
Defense Depot Memphis, Tennessee
Memphis, Shelby County, Tennessee

DRAWN BY: JTC

DATE: 11/18/2016

SCALE: AS SHOWN

PROJECT NO: W9128F-11-D-0029 CK01

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Figure 5
Dunn Field Total CVOC Concentrations, April 2015

Dunn Field
Defense Depot Memphis, Tennessee
Memphis, Shelby County, Tennessee

DRAWN BY: JTC

DATE: 11/18/2016

SCALE: AS SHOWN

PROJECT NO: W9128F-11-D-0029 CK01

MIP Survey Study Area

Legend

Total CVOC Ranges (µg/L)
- 0 - 10
- 11 - 50
- 51 - 100
- 101 - 500

Total CVOC Isopleth (µg/L)
- 10
- 50
- 100
- 200

- Groundwater Flow Direction
- Potentiometric surface of the Fluvial Aquifer 5-ft. contour
- Potentiometric surface of the Fluvial Aquifer 1-ft. contour
- Property Boundary
- Air Sparge Area

Figure 26
Dunn Field Total CVOC Concentrations, April 2015

Annual Long Term Monitoring Report-2015

Defense Depot Memphis, Tennessee

0 100 200
Feet

Notes:
1. Analytical results from the Long Term Monitoring sample event (4/8-11/15).
2. Highest concentration at well pairs used for contour.
3. Results from IAQ and MAQ wells (shown in Italics) were not used for isopleths.

Service Layer Credits: HDR, 2016

Trinity Analysis & Development Corp.

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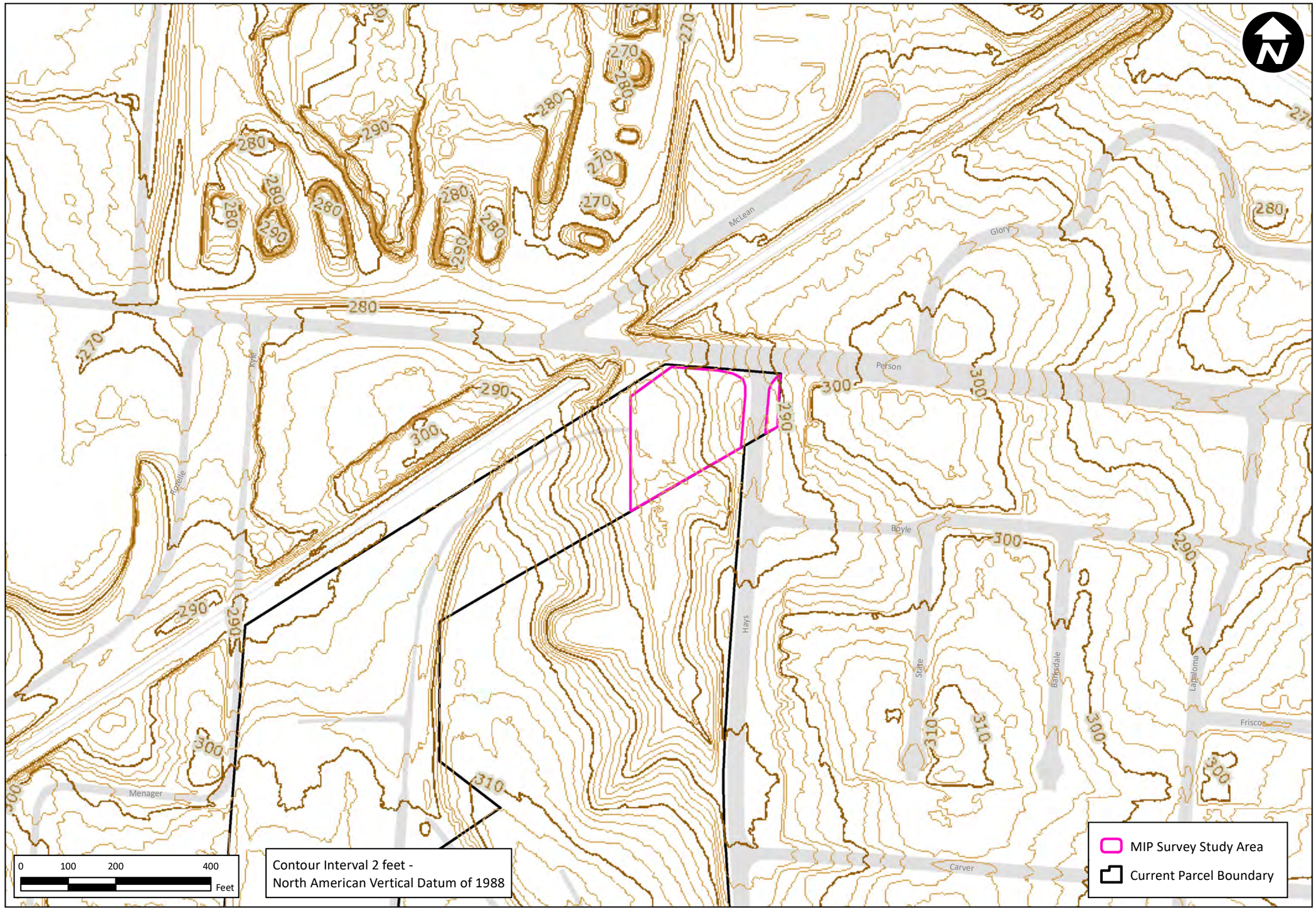

Service Layer Credits: OIR GIS

Trinity Analysis & Development Corp.

Figure 6
Site Topography

Dunn Field
Defense Depot Memphis, Tennessee
Memphis, Shelby County, Tennessee

PROJECT NO:	SCALE:	DATE:	DRAWN BY:
W9128F-11-D-0029 CK01	AS SHOWN	11/18/2016	JTC

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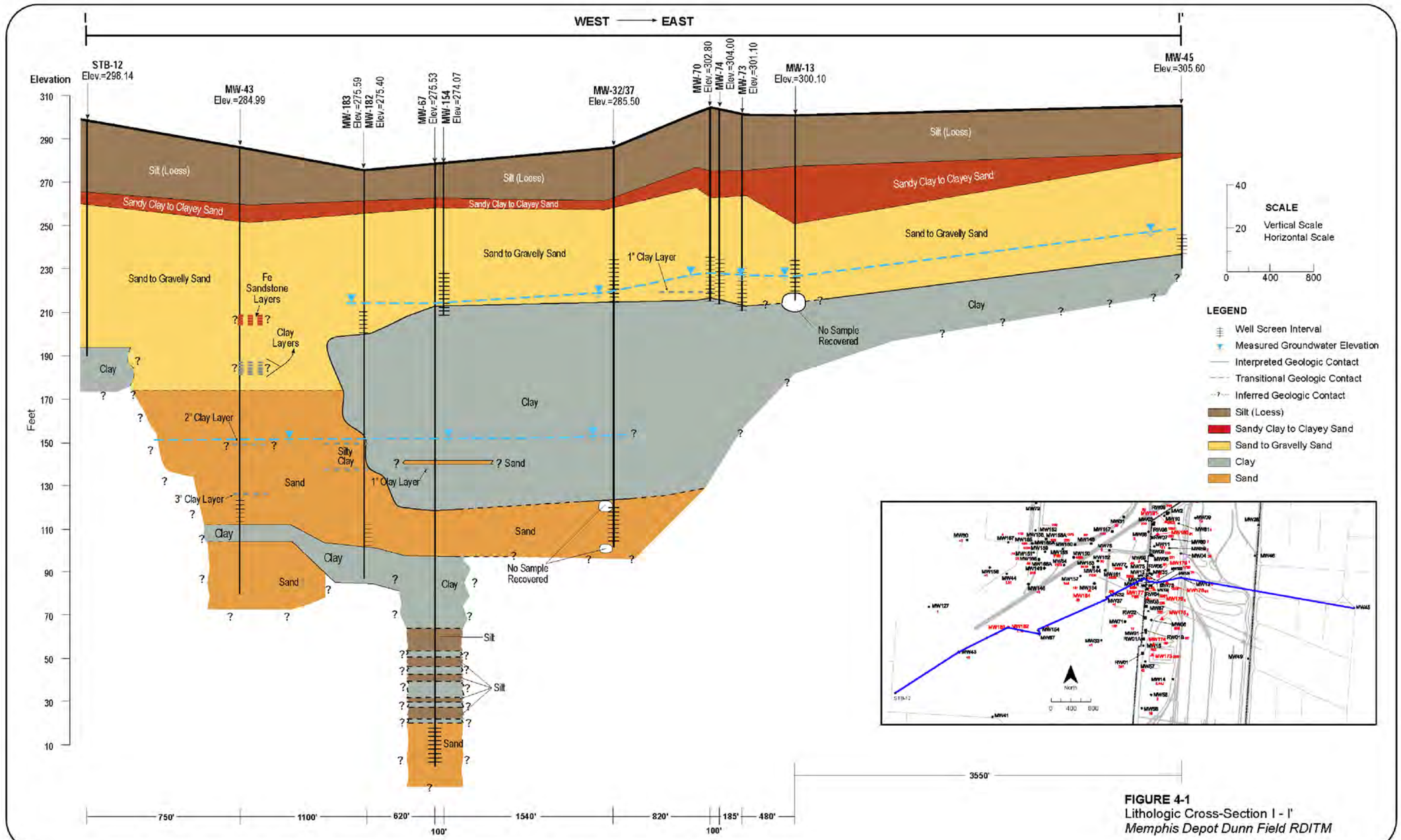

FIGURE 4-1
Lithologic Cross-Section I - I'
Memphis Depot Dunn Field RDITM

CH2MHILL

ES022006012ATL\119

Service Layer Credits: HDR, 2016

Trinity Analysis & Development Corp.

Figure 7
Dunn Field Lithologic Cross-Section

Dunn Field
Defense Depot Memphis, Tennessee
Memphis, Shelby County, Tennessee

DRAWN BY: JTC
DATE: 10/31/2016
SCALE: AS SHOWN
PROJECT NO: W9128F-11-D-0029 CK01

U.S.ARMY

Figure 8
Dunn Field Groundwater Elevations, April 2015

Dunn Field
Defense Depot Memphis, Tennessee
Memphis, Shelby County, Tennessee

DRAWN BY: JTC
DATE: 11/18/2016
SCALE: AS SHOWN
PROJECT NO: W9128F-11-D-0029 CK01

MIP Survey Study Area

Legend

- Monitoring Well Screened in the Fluvial Aquifer
- Monitoring Well Screened in the Transition Zone
- Monitoring Well Screened in the Intermediate Aquifer
- Monitoring Well Screened in the Memphis Aquifer
- Groundwater Flow Direction
- Potentiometric surface of the Fluvial Aquifer 5-ft. contour
- Potentiometric surface of the Fluvial Aquifer 1-ft. contour
- Air Sparge Area

MW-03 100.12 Blue: Fluvial Aquifer, used for groundwater contours
MW-107 202.34 Lt Blue: Transition Zone, used for groundwater contours
MW-237 100.15 Orange: Intermediate Aquifer, not contoured
MW-254 156.83 Green: Memphis Aquifer, not contoured

Figure 14
Dunn Field Groundwater Elevations, April 2015

Annual Long Term Monitoring Report-2015

Defense Depot Memphis, Tennessee

0 100 200
Feet

Service Layer Credits: HDR, 2016

Trinity Analysis & Development Corp.

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Worksheet 11 - Data Quality Objectives

This worksheet is used to develop and document project data quality objectives (DQOs) using the systematic planning process outlined in *Guidance on Systematic Planning Using the Data Quality Objectives Process* (USEPA, 2006). The site-specific DQOs were developed using the USEPA seven-step process and are summarized below.

Table 6 Data Quality Objectives

1	Problem Statement	LTM has identified CVOCs at concentrations above MCLs in monitoring wells categorized as Background-NE, some of which are located off-site (upgradient) of Dunn Field. This area is outside of any previously implemented RAs and source investigations.
2	Identify the Goals	Utilize the acquired data to determine if an on-site source of contamination does exist in the northeast corner, within the boundaries of the MIP Survey at Dunn Field, that may be impacting groundwater.
3	Inputs to the Decision	The data collected during the MIP and associated soil sampling activities will be used to decide whether an on-site source of groundwater contamination in the northeastern portion of Dunn Field exists.
4	Study Area Boundaries	The study area is limited to the northeast portion of Dunn Field. MIP boring locations will initially be evenly gridded over a 300-foot x 300-foot area. Due to property transfer for realignment of Hays Road, a portion of the study area exists outside the current Dunn Field fenced property. No borings will be advanced within Hays Road. MIP borings will be advanced through the unsaturated overlying clayey deposits (loess) estimated to range in thickness of up to approximately 30 feet bls. The MIP electrical conductivity sensor and the temperature sensor will be monitored to track lithologic changes from the loess into the underlying sands and for the presence of groundwater, respectively, to identify the vertical limits of the investigation.
5	Analytical Approach	The initial MIP boring placement was designed using a non-statistical sampling approach with a predetermined number of locations based on the study area dimensions. Boring placement method was based on a fixed spacing which allocated one boring to each area of the systematic grid. As real-time results are obtained, spacing and frequency will be refined, as necessary, to focus on specific areas of interest identified by elevated MIP responses. Soil samples will be collected and analyzed to quantitate MIP results. MIP: Field screening data will be collected utilizing a direct push technology (DPT) rig to push MIP instrumentation into the soil to collect and analyze gas samples with 3 instruments: photoionization detector (PID), flame ionization detector (FID), and electron capture detector (ECD). The ECD sensor is designed to detect CVOCs to a detection limit of 0.20-2.0 parts per million. DPT Soil: Soil borings will be collected by advancing tooling to specified depths based on the findings of the MIP Survey. Soil sample intervals within the selected boring locations will be based on observed MIP results, specifically from the ECD component of the MIP detectors which targets CVOC detection. Target intervals for soil samples will be selected to include the full range of ECD readings in the

		study area, but with a bias toward high readings. Soil samples will then be collected from the target intervals in new and clean acetate liners and sample specimens collected with Terra Core® kits for off-site laboratory analysis of CVOCs. Soil analytical results will be screened against Remediation Goals for Site-Specific Soil Screening Levels to be Protective of Groundwater (Loess Specific Values) established in the Dunn Field Record of Decision (CH2M Hill, 2004).
6	Acceptable Limits on Decision Error	Contaminant concentrations cannot be directly determined from the MIP detector responses due to changes in subsurface conditions and influences that are encountered which cannot be reproduced at the surface. MIP technology is a screening tool that aids in determining presence or absence of VOCs in soil. Additionally, variables such as soil type, water content, membrane wear, and chemical and mix of chemicals present can influence detector responses. Field measurements obtained during the MIP survey provide screening level data that are sufficient to use as judgments in selecting soil sample locations/intervals to determine the absence or presence of a source for the groundwater contamination identified in the northeastern portion of Dunn Field. Soil data analyzed at an off-site laboratory will be considered definitive data for the confirmation of MIP responses and evaluation of specific target analytes in soil, if present.
7	Develop the Plan	The specific project tasks will be conducted as described in Worksheet 14. This is a stand-alone UFP-QAPP for execution of the Dunn Field MIP Survey.

Worksheet 12 - Measurement Performance Data

This worksheet documents the quantitative measurement performance criteria (MPC) in terms of precision, bias, and sensitivity for both field and laboratory measurements and is used as guidance for selecting appropriate techniques and analytical methods. In conjunction with Worksheet 11, these MPC ensure data will satisfy the Project Quality Objectives (PQOs) and DQOs.

MPC were established for each analytical parameter. Refer to the following worksheets for the required information in this worksheet:

- Worksheet 15 (Reference Limits and Evaluation) for data quality indicators (DQIs) consisting of precision and accuracy
- Worksheet 24 (Analytical Instrument Calibration)
- Worksheet 28 (Laboratory Quality Control Sample Summary)
- Worksheet 36 (Validation [Stage 3] Summary) for data review and validation process; and
- Worksheet 37 (Usability Assessment) for precision, accuracy, representativeness, comparability, completeness, and sensitivity (commonly referred to as precision, accuracy, representativeness, comparability, completeness, and sensitivity [PARCCS] parameters)

The quality of the data to be collected for this project will be verified using appropriate MPC established for both sampling procedures and analytical methods. The criteria will relate to the DQIs in the table below. The MPC follow those defined in the DoD Quality Systems Manual (QSM), Version 5.0 (DoD, 2013). The sampling procedures and the quality of the laboratory results will be evaluated for compliance with the project-specific DQOs through a review of overall PARCCS, in accordance with procedures described in Worksheet 37 (Usability Assessment).

Table 7 Quantitative Measurement Performance Criteria

QC Sample	Analytical SOP	Frequency	Data Quality Indicators	Measurement Performance Criteria	QC Sample Assesses Error*
Matrix: **Analytical Group/Method:** **Concentration Level:**		**Soil** **VOCs/8260C** **Low/medium**			
Field Duplicates	VO 004	One per every 10 field samples	Overall Precision	For Values > 5X LOQ, RPD ≤ 30%	Sampling and Analytical
Laboratory control sample (LCS)/Laboratory control sample duplicate (LCSD)		At least one per batch	Analytical Precision/ Accuracy/ Bias	Recovery within LCS limits (see Worksheet 15); RPD ≤ 20%	Analytical
MS/MSDs		One per 20 sample matrix	Analytical Accuracy/ Bias (matrix interference)	Recovery same as for LCS (see Worksheet 15); RPD ≤20%	Sampling and Analytical
Equipment Blanks		As required per sampling event	Overall Accuracy/ Bias (contamination)	No target analytes ≥ LOQ	Sampling
LOQ		N/A	Sensitivity	See Worksheet 15	Analytical
Data Completeness		N/A	Data Completeness	95% Overall	Sampling and Analytical

LCS/LCSD – laboratory control sample/laboratory control sample duplicate
LOQ – limit of quantitation
MS/MSD – matrix spike/matrix spike duplicate
N/A – not applicable or not available
RPD – relative percent difference
SOP – standard operating procedure

Worksheet 13 - Secondary Data Criteria and Limitations

Secondary data refer to historical data and background information previously collected at the site. The source(s) of the data, date of collection, planned uses, and limitations of the secondary data are summarized in the following table.

Table 8 Secondary Data Criteria Limitations

Secondary Data Source	Source	Date of Collection	How Data Will Be Used	Limitations on Data Use
Annual Long-Term Monitoring Report-2015	HDR, 2016	2015	Guidance for field investigation design	None
Annual Long-Term Monitoring Report-2014	HDR, 2015	2014	Guidance for field investigation design	None
Analysis of Tennessee Department of Environment and Conservation (TDEC) Environmental Reports to Evaluate the Source of Chlorinated Solvents in Dunn Field Upgradient Wells	CALIBRE, 2015	2004-2008	Guidance for field investigation design	None
Main Installation Source Area Investigation	e^2m, 2009b	2008	Guidance for field investigation design	None
Memphis Depot Dunn Field Source Areas Final Remedial Design	CH2M Hill, 2007	2005	Guidance for field investigation design	None
Dunn Field Record of Decision	CH2M Hill, 2004	Not Applicable	Guidance for field investigation design	None

Worksheet 14 - Summary of Project Tasks

This worksheet includes specific tasks and responsible parties. The planned start and end dates for the project tasks are provided in Worksheet 16. The proposed activities are based on the project Statement of Work (CESAM, 2016). A discussion of project activities is presented in the following sections. Field SOPs are included in **Attachment 1** and example field forms are included in **Attachment 2**.

Pre-Investigation Requirements

A portion of the investigation area will be conducted on private property and within the City of Memphis right of way. All property owners will be notified prior to mobilization for approval. Clearance of all underground utilities will be performed in the areas of subsurface intrusive activities prior to field mobilization. A pre-investigation walk through will be conducted by the FTL to inspect site conditions for equipment access, equipment staging, decontamination area(s), potential site hazards, and emergency evacuation routes.

MIP Borings

MIP borings will be advanced via DPT methods within a 300-foot by 300-foot study area located in the northeast portion of Dunn Field to confirm the absence or presence of CVOCs that may be associated with unknown burial or disposal sites, or other environmental contamination on Army property. The initial design will evenly distribute 60 boring locations in a systematic grid across the area (**Figure 9**). The borings will be advanced through the unsaturated loess, ranging in thicknesses of 20 to 30 feet, and move outward from the initial boring location at the northeast corner of the study area. The minimum depth of the MIP borings will be the base of the loess as determined by field measurements and the maximum depth will be based on vertical extent of contamination as indicated by field instruments or DPT boring refusal. As MIP results are returned and data evaluated in real-time, the boring locations may be shifted to optimize the design and maximize time in the field.

Target operation of the MIP tooling will be as follows based on manufacturer recommendation. The MIP probe will be advanced in 1-foot increments at a rate of approximately 0.5 feet/second. The probe will be stopped at each 1-foot interval for 45 seconds for the MIP block and membrane to heat up and for consistent sample collection. The membrane will also be exposed for 45 seconds in the response test and which is approximately the length of time required to add a drill rod. Stopping at each interval for 45 seconds keeps the advancement and membrane exposure consistent. The professional judgement of the MIP operator will be used to determine if a positive response above instrument detection levels exists.

Soil Borings/Sampling

DPT soil borings will be advanced at locations based on the intervals of interest identified during the MIP investigation and within 1-foot of or as close as possible to the MIP boring location. Continuous soil cores will be collected in new and clean acetate liners during the advancement of each boring and logged in the field by the FTL. Soil cores will be collected from the target intervals and sample specimens collected with Terra Core® kits for off-site laboratory analysis. The locations and target depths of the soil borings will be based on the MIP results and will be used to confirm both absence and presence of detected results. The number and locations of borings advanced will be determined in the field based on the observed MIP results which could allow for the collection of soil samples from multiple intervals within the same boring.

The field geologist in communication with the project team will review the "real-time" MIP data and concur on soil sample locations and depths.

Boring Abandonment

Upon completion of MIP and DPT soil borings, they will be abandoned by grouting to the surface with a neat Portland cement grout with 5% bentonite from the bottom up using a tremie pipe. Additional grout will be added as necessary to fill the boring if settling occurs.

Land Surveying

A professional land surveyor (PLS) licensed in the State of Tennessee will identify and stake all proposed MIP boring locations prior to MIP mobilization. Upon completion of the MIP and DPT soil investigations, the PLS will resurvey all completed borings. 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 0.1 foot for elevations and horizontal coordinates. The surveyor's data will be compatible with existing surveys using referenced benchmark located on Dunn Field. The location of the benchmark is identified on **Figure 3**.

Investigative Derived Waste Management

Waste generated during MIP and DPT soil sampling activities will be classified as either non-investigative waste or investigative derived waste (IDW). Non-investigative waste such as packing materials, personal PPE, and other inert refuse will be collected and placed in a dumpster for disposal as municipal waste. The IDW will consist of decontamination water and excess soil cuttings. Decontamination water will be stored in 55-gallon drums or polyethylene totes and excess soil cuttings will be stockpiled on plastic sheeting at designated locations within Dunn Field. Each medium will be sampled for waste characterization to determine final disposition.

If soil results are below remediation goals set forth in the Dunn Field Record of Decision (CH2M Hill, 2004), the soil will be spread on the ground at Dunn Field. If soil VOC concentrations are above remediation goals, off-site disposal will be arranged. Containerized decontamination water will be disposed off-site after receipt and review of waste characterization profile.

Laboratory Analysis Tasks

Soil samples will be submitted to an off-site laboratory for analysis of VOCs by USEPA Method 8260C.

Quality Control Tasks

In addition to quality assurance (QA)/QC and maintenance procedures recommended by the MIP detector manufacturers and the American Society for Testing and Materials, on-site QA/QC procedures will be performed using pre- and post-boring standard sensitivity response tests using solutions of CVOCs. Field SOPs for sample collection, sample packaging and shipping, and analysis will be followed by the FTL.

Data Management Tasks

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

Documentation and Records

All sample locations will be identified by a PLS upon completion of the investigation, field measurements and sample data noted in field records and maintained in project files. Sample results and data validation will be presented in the summary report.

Data Packages

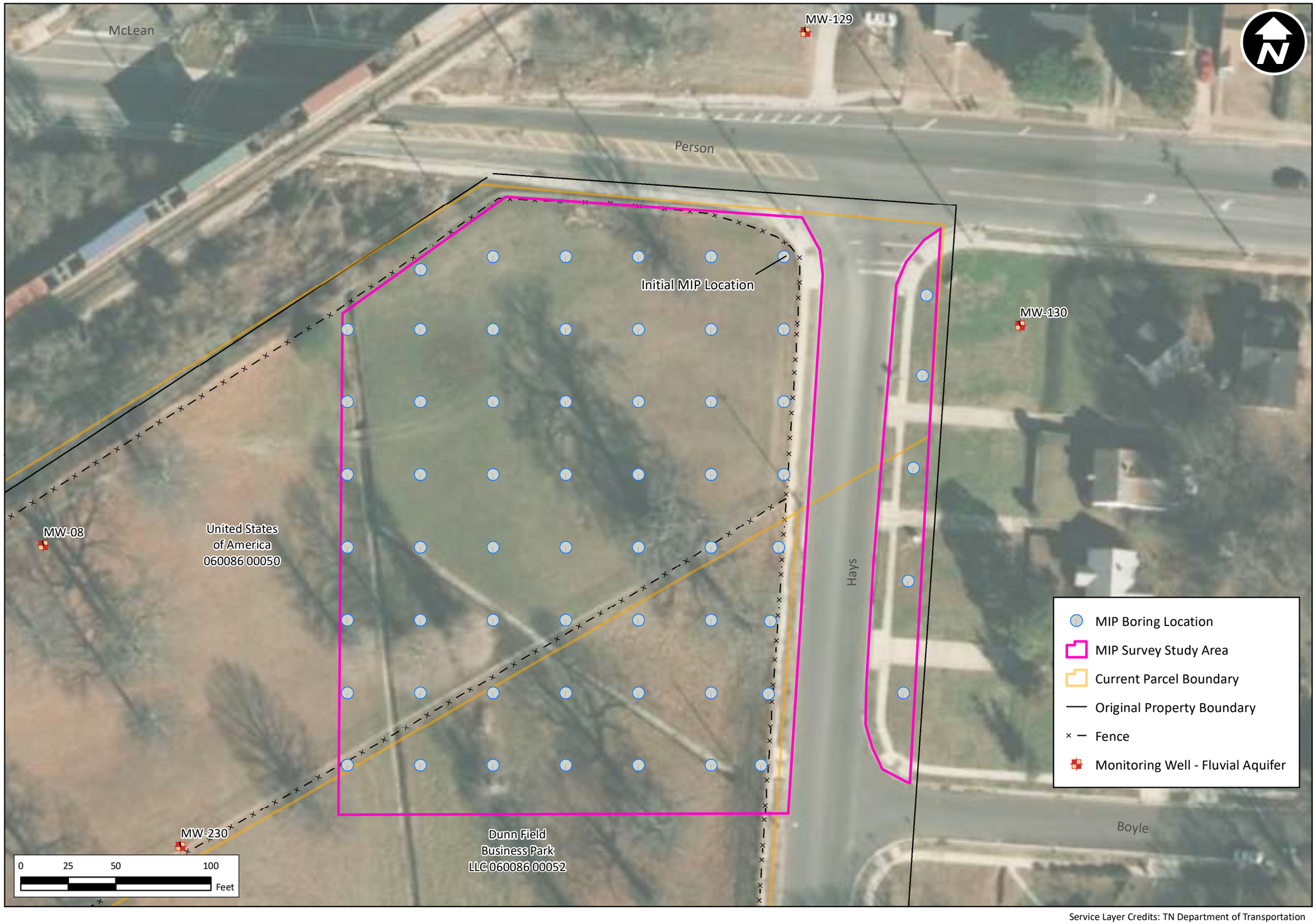
CTL will provide complete analytical data packages including raw data (Level IV) for soil samples in accordance with Appendix E, SW-846 Reporting Requirements, of the DoD QSM (DoD, 2013).

Assessment/Audit Tasks

Field sampling procedures will be reviewed by the Trinity Project Manager. Annual laboratory audits are performed through the DoD Environmental Laboratory Accreditation Program (ELAP).

Data Review Tasks

The off-site laboratory will verify that all data are complete for the samples received. All data package deliverable requirements will be met. Data will be reviewed by LDC at the Step I (Verification)/Steps IIa and IIb (Validation) level as described in Worksheets 34, 35, and 36. Achievement of all project-specific MPC identified in Worksheet 12 will be evaluated during the data verification and validation, and the analytical measurement error will be assessed. A Stage 3 Data Validation report (DoD, 2013) will be produced for each sample delivery group (SDG).



Service Layer Credits: TN Department of Transportation

Trinity Analysis & Development Corp.

Figure 9
MIP Boring Layout

Dunn Field
Defense Depot Memphis, Tennessee
Memphis, Shelby County, Tennessee

DRAWN BY: JTC

DATE: 12/8/2016

SCALE: AS SHOWN

PROJECT NO: W9128F-11-D-0029 CK01

U.S.ARMY

Worksheet 15 - Reference Limits and Evaluation

This worksheet includes laboratory quality control data for each matrix and analytical method. The goal is that the laboratory and method can provide accurate data at the project screening criteria. Note that this table only includes analytes identified above screening criteria in groundwater samples collected in upgradient and downgradient monitoring wells.

Table 9 Reporting Limits and Screening Objectives

Analyte	Soil Screening Objective (µg/kg)	Chemical Abstract Service No.	LOQ (µg/kg)	LOD (µg/kg)	Detection Limit (DL) (µg/kg)	Accuracy Control Limit (%R)	Precision Control Limit RPD (%) Lab QC / Field QC
Matrix: Soil Analytical Group/Method: VOCs/8260C Concentration Level: Low/medium							
1,1,2,2-tetrachloroethane	11.2	79-34-5	2	1	0.5	70 – 124	20 / 50
1,1,2-trichloroethane	62.7	79-00-5	2	1	0.4	78 – 121	20 / 50
1,1-dichloroethene	150	75-35-4	2	1	0.4	70 – 131	20 / 50
1,2-dichloroethane	32.9	107-06-2	2	1	0.5	73 – 128	20 / 50
carbon tetrachloride	215	56-23-5	2	1	0.3	70 – 135	20 / 50
chloroform	917	67-66-3	2	1	0.3	78 – 123	20 / 50
tetrachloroethene	180.6	127-18-4	2	1	0.4	73 – 128	20 / 50
trichloroethene	182	79-01-6	2	1	0.3	77 – 123	20 / 50
cis-1,2-dichloroethene	755	156-59-2	2	2	0.4	77 – 123	20 / 50
trans-1,2-dichloroethene	1520	156-60-5	2	1	0.4	74 – 124	20 / 50
vinyl chloride	29.4	75-01-4	2	1	0.5	56 – 135	20 / 50

Soil Screening Objective = Remediation Goals for Site-Specific Soil Screening Levels to be Protective of Groundwater, Loess Specific Values (CH2M Hill, 2004)
Lab QC = LCS/LSD and MS/MSD
Field QC = field duplicate (FD)

Worksheet 16 - Project Schedule/Timeline Table

Figure 9 presents the anticipated schedule for this project, which includes the timeframes for the major activities and deliverables, as well as the individual tasks and their interrelationships.

Table 10 Project Schedule

Activity	Organization	Anticipated Date(s) of Initiation	Anticipate Date(s) of Completion	Deliverable	Deliverable Due Date
Work Plan Preparation	Trinity	5/6/2016	1/10/2017	UFP-QAPP Work Plan	1/10/2017
MIP Survey Field Work	Trinity	2/6/2017	2/15/2017	Samples to laboratory, field reports to file	---
Soil Sample Analysis	CTL	2/15/2017	2/28/2017	Level IV report and electronic data deliverable (EDD)	---
Data Validation	LDC	2/28/2017	3/6/2017	Data narrative report	---
Report Preparation	Trinity	2/21/2017	8/9/2017	Summary report	8/9/2017

Worksheet 17 - Sampling Design and Rationale

This worksheet describes the sampling design/field investigation activities and basis for its selection. The field activities will be conducted in accordance with the project Statement of Work (CESAM, 2016) and the field SOPs provided in Worksheet 21 and **Attachment 1**. The number of samples and the analytical parameters planned are summarized in Worksheet 18.

Physical Boundaries for the Area Under Study

The boundaries of the study area are shown in **Figure 8**. Investigation activities will take place within the fenced northeastern corner of Dunn Field and private property located adjacent to the Dunn Field property both bordered by Hays Road to the east and East Person Avenue to the north. Additionally, based on the realignment of Hays Road and shifting of the Dunn Field property boundary, the physical boundaries will encompass City of Memphis right of way.

Basis for the Placement and Number of MIP Borings and Soil/Boring Sample Locations

MIP Boring Layout

The MIP investigation will be conducted within an approximately 300-foot by 300-foot area based on the presence of contaminants, specifically 1,1-DCE, identified in groundwater samples collected from nearby, off-site and upgradient monitoring wells. Within this area, approximately 60 borings will be advanced through the loess to depths of approximately 20 to 30 feet below land surface. Based on the review and evaluation of the real-time data responses, the sampling grid may be modified to optimize the location of borings.

Soil Boring/Sample Locations

Soil samples will be collected from intervals identified during the MIP investigation. Up to 15 soil samples will be collected for VOC analysis by USEPA Method 8260C by CTL to provide definitive analytical results at 25% of the MIP locations. The goal of 25% was selected to be consistent with that stated for the Main Installation Source Area Investigation (e^2m, 2009b). The analytical results will be used to correlate CVOC concentrations to MIP-ECD results. The analytical results will be used to verify the absence or presence of soil contamination, and quantify, if present, so samples will be collected across a range of MIP responses, including no response. Detection of the target CVOCs above remediation goals in soil samples or correlated ECD results will serve as indication of a contaminants in soil and will be used to make recommendations for further actions, as necessary.

Worksheet 18 - Sampling Locations and Methods/SOP Requirements

The following table summarizes the sampling matrix, number of samples to be collected, analytical parameters, and the rationale for sampling locations described in Worksheet 17 (Sampling Design and Rationale).

Table 11 Sampling Locations and Sampling SOP Requirements

Sampling Location	Number of Locations/ Samples	Matrix	Depth (feet)	Analytical Group	Concentration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference[1]	Rationale for Sampling Location
Dunn Field MIP Boring	Up to 60	Soil gas	TBD	CVOCs	N/A	N/A	Technical Bulletin No. MK3010	Potential on-site source
Dunn Field Soil Boring	Up to 15	Soil	TBD	CVOCs	No response to highest response	15 primary 2 field duplicate 1 MS 1 MSD	SOP 02	Based on MIP results
IDW	Up to 2	Soil/aqueous	N/A	VOCs	N/A	1 composite soil/ 1 composite aqueous	SOP 05	N/A

TBD – to be determined

The sample nomenclature for soil samples will be as follows:

DF-####-dd-YYMM

Where,

- DF = Dunn Field
- ### = sample location type and identifier)
- dd = bottom of sample depth in feet bls
- YYMM = Year/Month

Example,

DF-SB01-20-1611: soil sample collected from Dunn Field soil boring (SB) SB01 from a bottom depth of 20 feet bls, November 2016

The following are reserved for waste characterization (WC) and QC sample type identifiers and will be used in the place of the soil boring location ID and will be sequentially numbered; WC samples will be composited and therefore will not have a depth identifier included; equipment rinsate blanks (RB) and trip blanks (TB) will be aqueous samples and therefore not have a depth identifier included:

FD = field duplicate

RB = equipment rinsate blank

TB = trip blank

WC = waste characterization

FDs will be collected at a frequency of 10 percent, MS/MSD samples will be collected at a frequency of approximately 5 percent. FD samples will only be collected if a sufficient volume of soil exists and field conditions allow. MS/MSD samples will be identified exactly as the primary sample and will not receive a special designation. The collection of MS/MSD will be noted on the chain of custody.

Worksheet 19 - Analytical SOP Requirements Table

This worksheet summarizes the analytical methods for each sampling matrix, including the required sample volume, containers, preservation, and holding time requirements. Further information on the analytical SOPs is provided in Worksheet 23 (Analytical SOP References).

Table 12 Sample Containers, Preservation, and Hold Times

Matrix	Analytical Group	Preparation and Analytical Method/SOP Reference	Container	Preservation Requirement	Maximum Holding Time
Soil	VOCs	5035A/8260C VO 004	Terra Core® or other sampling device (4) 40-mL glass vials plus 2-ounce jar	Cool to 0-6°C, methanol (2 40-mL vials) and sodium bisulfate (2 40-mL vials)	14 days
Aqueous	VOCs	5035A/8260C VO 004	(3) 40-mL glass vials	Cool to 0-6°C; no headspace; hydrochloric acid to a pH less than 2	14 days
Soil	TCLP VOCs	1311 PR 002	(1) 8-ounce glass jar	Cool to 0-6°C	14 days from collection until start of extraction
Aqueous	TCLP VOCs	1311 PR 002	(1) 1-liter glass jar	Cool to 0-6°C	14 days from collection until start of extraction

TCLP - toxicity characteristic leaching procedure

Worksheet 20 - Field Quality Control Sample Summary

This worksheet summarizes the field QC samples to be collected from the site. The number of field QC samples for each sampling matrix and analytical parameter is provided in the table below.

Table 13 Field QC Sample Summary

Matrix	Analytical Group	Analytical Method[1]	No. of Normal Field Samples	No. of FDs	No. of RB[2]	No. of TBs[3]	No. of MS/MSD (Total)	Total No. of Samples to Lab
Soil	VOCs	8260C	15	2	0	0	2	19
Aqueous	VOCs	8260C	0	0	4	4	0	8

[1] - The analytical and preparation SOP references are provided in Worksheet 23 (Analytical SOP References).
[2] – One RB will be collected each day of soil sample collection for VOC analysis.
[3] – One TB will be shipped in each cooler containing samples designated for VOC analysis.

Worksheet 21 - Project Sampling Standard Operating Procedure Reference

The field SOPs associated with the project sampling (including, but not limited to, sample collection and sample handling and custody) are listed in the following table. Corporate SOPs were prepared based on guidance from the Quality System and Technical Procedures established by the USEPA Science and Ecosystem Support Division (http://www.epa.gov/region4/sesd/fbqstp/). The referenced field SOPs are provided in **Attachment 1**.

Table 14 Field SOP References

Reference Number	Title	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)
SOP 01	Field Records and Documentation	Trinity	N/A	N
SOP 02	Drilling and Soil Sampling	Trinity	Field sampling equipment	N
SOP 03	Field Screening Techniques	Trinity	Field measurement equipment	N
SOP 04	Equipment Decontamination	Trinity	N/A	N
SOP 05	Investigation Derived Waste Management	Trinity	N/A	N
Technical Bulletin No. MK3010	Geoprobe Membrane Interface Probe	Geoprobe	MIP equipment	N

Worksheet 22 - Field Equipment Calibration, Maintenance, Testing, and Inspection

This worksheet lists the field equipment and instruments to be used during the MIP investigation and DPT soil sampling that will require calibration, maintenance, testing, or inspection.

Field equipment and instruments are identified in the table below.

Table 15 Field Equipment and Instruments

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	Field SOP Reference
MIP (PID, FID, ECD)	Before each use per manufacturer's specifications and as needed	Per manufacturer's specifications	Analyze reference standard as per manufacturer's specifications	Beginning of day (before use) and as needed	See manufacturer's specifications	Repeat calibration	MIP Operator	Technical Bulletin No. MK3010

Worksheet 23 - Analytical Standard Operating Procedure References

The laboratory SOP references identified in the table below were provided by CTL in Baraboo, Wisconsin. The laboratory SOPs are supplemented by internal communication systems within the laboratory to disseminate the project requirements to technical staff. Analytical SOPs are provided in **Attachment 3**.

Table 16 Analytical SOP References

Reference Number	Title, Revision Number, and Date	Definitive/ Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
VO 004	Analysis of Volatile Organic Compounds by GC/MS (8260C)	Definitive	VOCs 8260C	Hewlett Packard Gas Chromatographs (5890 & 6890) Columns, Supelco (SPB-624), Agilent (DB-624UI), or Zebron (ZB-624)	CTL	N
PR 002	TCLP and SPLP Extraction, Volatile Fraction	Definitive	VOCs TCLP	Refer to SOP	CTL	N

GC/MS - gas chromatograph/mass spectrometer

Worksheet 24 - Analytical Instrument Calibration

To confirm that the analytical methods and the selected instrumentation meet the project requirements, each analytical instrument will be calibrated according to the procedures outlined in Worksheet 28 (Laboratory QC Sample Summary) and the following table.

Specific analytical method SOP references are provided in Worksheet 23 (Analytical SOP References). Full method QA/QC tables are provided for ease of use to the Project Engineer and the laboratories. This information provides documentation on corrective actions, flagging criteria for laboratory services and expectations for analytical services, meets the requirements outlined in Worksheet 28, and reflects the requirements of the DoD QSM, Version 5.0 (DoD, 2013).

Table 17 Analytical Instrument Calibration

Instrument/ Method	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/MS / 8260C	CCV	CCV daily, before sample analysis, and every 12 hours of analysis time, and at the end of analytical run	All targets ≤ 20%D / within 50%D for end of analytical batch CCV	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification	Analyst / Supervisor	VO 004 Rev 0
	ICAL for all analytes (including surrogates)	Initial calibration prior to sample analysis	RSD ≤30 for RFs of the CCVs; Average %RSD ≤ 15% for all compounds, linear or quadratic curve fit with coefficient of determination ≥ 0.99	Repeat calibration if criterion is not met	Analyst / Supervisor	
	Second source calibration verification	Once after each initial calibration	All analytes within ± 20% of expected value	Remake standard, recalibrate if necessary.	Analyst / Supervisor	

Instrument/ Method	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
	Evaluation of Relative Retention Times	Prior to sample analysis	Set at mid-point of ICAL; +/- 30 seconds each CCV	CCV fails, perform column maintenance, inspect pumps, and leak checks	Analyst / Supervisor	
	Tuning	Every 12-hours before calibration	SOP criteria for ion abundance.	Perform instrument maintenance	Analyst / Supervisor	
	LOD/LOQ verification	Quarterly	LOD meets method qualitative requirements or is at least 3X higher than noise; LOQ is within LCS/LCSD criteria.	Perform instrument maintenance and repeat failed LOD or LOQ study passing two consecutive tests or perform new DL study.	Analyst / Supervisor	

CCV = continuing calibration verification
ICAL = Initial calibration
RFs = Response factors
RSD = relative standard deviation

Worksheet 25 - Analytical Instrument and Equipment Maintenance, Testing, and Inspection

To confirm that the analytical instrument and equipment are available and in working order when needed, all laboratory analytical equipment will be maintained and tested in accordance with procedures described in the analytical method SOPs as listed on Worksheet 23. The analytical instrument and equipment maintenance, testing and inspection activities and acceptance criteria are provided in the following table.

Table 18 Analytical Instrument and Equipment Maintenance, Testing, and Inspection

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Replace septa, clean injection port, clip column, check auto sampler, clean source	VOC	Detector, injection port, column, autosampler	As needed	Must meet initial and/or continuing calibration criteria	Repeat maintenance activity or remove from service	Lab Section Supervisor	VO 004

Worksheet 26 - Sample Handling System

To verify sample authenticity and data defensibility, a proper sample handling system will be followed from the time of sample collection to final sample disposal.

The FTL or designee will be responsible for the sample collection, sample packing, and coordination of sample shipment. Samples will be sent to the analytical laboratory via FedEx Priority overnight.

A laboratory representative will acknowledge receipt of the sample coolers upon arrival. The laboratory technicians will prepare and analyze the field samples in accordance with the analytical methods and SOPs. The field samples will be stored at the laboratory for 60 days after a final report has been submitted to Trinity. The Laboratory Hazardous Waste Manager will be responsible for the final sample disposal upon notice from the Trinity Project Manager.

Table 19 Sample Handling System

Sample Collection, Packaging, and Shipment	
Sample Collection (Personnel/Organization):	Ben Stewart (FTL) / Trinity
Sample Packaging (Personnel/Organization):	Ben Stewart (FTL) / Trinity
Coordination of Shipment (Personnel/Organization):	Ben Stewart (FTL) / Trinity
Type of Shipment/Carrier:	FedEx Priority Overnight service
Sample Receipt and Analysis	
Sample Receipt (Personnel/Organization):	Brett Szymanski / CTL
Sample Custody and Storage (Personnel/Organization):	Brett Szymanski / CTL
Sample Preparation (Personnel/Organization):	Brett Szymanski / CTL
Sample Determinative Analysis (Personnel/Organization):	Brett Szymanski / CTL
Sample Archiving	
Field Sample Storage (number of days from sample collection):	60 days
Sample Extract/Digestate Storage (number of days from extraction/digestion):	60 days
Sample Disposal	
Personnel/Organization:	Brett Szymanski / CTL
Number of Days from Analysis:	60 days

Worksheet 27 - Sample Custody Requirements

Proper sample handling, shipment, and maintenance of chain of custody forms are key components of building the documentation and support for data that can be used to make project decisions. The sections below summarize the field and laboratory sample custody procedures to be followed during the project.

Field Sample Custody Procedures

Field work for sampling activities will be conducted in accordance with the field SOPs listed in Worksheet 21 and located in **Attachment 1**. These SOPs outline the methodologies for equipment decontamination (Trinity SOP 02) and soil sampling and subsurface investigation (Trinity SOP 05). Sample nomenclature will follow previous investigation designations as described in Worksheet 18. Sample packaging, shipment, and delivery to laboratory activities will be conducted as described below.

Sample Handling and Shipping Procedures

Upon collection, all samples will be placed on ice immediately. Once all samples are collected or the cooler is filled with samples, it will be taped with a custody seal (example provided in **Attachment 2**) and secured in a sampling vehicle until the completion of the day's sampling activities. Prior to shipping, the contents of all coolers will be checked against the chain of custody and re-packaged for shipment so that bottles will not dislodge and/or break during shipment but arrive within temperature requirements. The chain of custody will be placed in a waterproof plastic bag and placed just under the lid of the cooler. The cooler lid will be secured with custody seals at a minimum of two locations and the seals will be covered with clear tape. All efforts will be made to ship samples each day they are collected. If necessary due to time constraints or work taking place over a weekend, all samples may be held for a short period of time (Worksheets 19 and 30).

The packaged samples will meet all applicable DOT and International Air Transportation Authority requirements prior to shipment. The samples will be classified as environmental samples which includes groundwater and soil. Shipping of environmental samples will be in accordance with the DOT *Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Laboratory Samples* (DOT, 1981).

Laboratory Sample Custody Procedures

Until the laboratory accepts delivery of samples by notation on a chain of custody document or otherwise in writing, the laboratory is not responsible for loss of or damage to samples. The laboratory, at its sole discretion, reserves the right to refuse or revoke Acknowledgment of Receipt for any sample due to insufficient sample volume, improper sample container, or risk of handling for any health, safety, environmental, or other reason. The laboratory does not accept samples that contain asbestos, biohazards, or radiological materials. Regardless of prior acceptance, the laboratory may return samples at its sole discretion if it is determined that the samples may pose a risk in handling, transport or processing, for any health, safety, environmental or other reason.

All samples must be scanned each time custody of the container is changed. This information is stored in the Laboratory Information Management System (LIMS), and includes a complete record of the sample custody from receipt to disposal. Information includes the location of the sample, the date and time of each custody transfer, unique initials of each person assuming custody, and a reason for the transfer.

Sample Archiving

The laboratory will retain all records related to sample analysis including raw test data, calculations, derived data, calibrations and copies of test reports. These records are archived in accordance with regulatory requirements for a minimum of 10 years. If the laboratory is going out of business, Trinity will be notified at least 60 days (if time permits) prior to closure of the laboratory and will receive a final report for all submitted samples. The notification will request instructions on the retention or distribution of laboratory records and will provide contact information for after the closure. Software/hardware permitting the access of electronic data must be maintained.

The laboratory will store copies of analytical reports in a location with access restrictions and all reports must be signed out using the archived reports logbook. Analytical reports and chains of custody are also scanned for electronic storage. All archived logbooks, corrective actions, training records, and other QA/QC reports are stored in a locked storage closet and only members of the QA/QC Department have access to these records. Written and printed data records (bench sheets, logbooks, electronic printouts, etc.) are scanned before being boxed and placed in storage. Electronic data are stored on a dedicated server backed-up daily. Approximately 1 year of electronic data are accessible at workstations. Data removed from the servers and stored on tapes can be reloaded by submitting a request. The laboratory's safety officer keeps safety and disposal information.

The laboratory stores archived data on site until capacity is met. The oldest archived data are then moved to a secure storage facility. The storage and on-site facility are monitored and protected from fire and theft. Electronic data storage is free from magnetic sources. It is the goal of the analytical laboratories to have redundant copies (hard and electronic) to prevent loss of records due to being misplaced or environmental deterioration or catastrophe.

Sample Disposal

Samples are stored in the appropriate cooler for 60 days after receipt. After 60 days, samples are moved to a waste area. The samples are scanned out for disposal on the LIMS. The samples are then stored in the waste staging area until disposal into appropriate drums. Laboratory waste is segregated by laboratory personnel into waste streams, which have been established by the laboratory Regulatory Compliance Officer. The waste streams are determined by analysis of the waste and through process knowledge. All laboratory wastes are disposed of in the proper container. No waste is placed in regular trash containers or poured down the drain. Waste is stored in drums in satellite accumulation areas and then in the central accumulation facility. Waste disposal service is provided by approved vendors who will incinerate, landfill, treat, or reclaim the waste based on the characteristics.

Samples not consumed in testing will normally be retained for a maximum of 60 days before disposal. Samples will be returned to Trinity when requested in writing or when they would pose a disposal problem as a hazardous waste as determined by each analytical laboratory, at their sole discretion.

Worksheet 28 - Laboratory Quality Control Sample Summary

This worksheet presents analytical QC requirements relevant to analysis of environmental samples that will be followed by laboratories producing definitive data. The purpose of the laboratory QC activities is to produce data of known quality that satisfy the project-specific DQOs. Laboratory QC samples will follow method specific requirements of the DoD QSM Version 5.0 (Appendix B of the QSM).

Laboratory QC samples must be included in an analytical batch with the field samples. An analytical batch is a group of samples (not exceeding 20 environmental samples plus associated laboratory QC samples) similar in composition (matrix) that are extracted or digested at the same time and with the same lot of reagents and analyzed together as a group. The analytical batch also extends to cover samples that do not need separate extraction or digestion. The identity of each analytical batch will be clearly reported with the analyses so that a reviewer can identify the laboratory QC samples and the associated environmental samples. The type of laboratory QC samples and the frequency of use of these samples are discussed below and in method-specific analytical SOPs.

Method Detection Limits

The method detection limit (MDL), as defined by Title 40 Code of Federal Regulations Part 136, Appendix B, is the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The MDL will be considered the DL for the purposes of this project in accordance with the DoD QSM Version 5.0 (DoD, 2013). The laboratory has established DLs for each analyte, and provided them to Trinity. The DL is used along with other measurements of sensitivity, such as the LOD and LOQ.

The laboratory participating in this work effort, CTL, will demonstrate the capability to achieve the MDLs for each instrument by presenting data for the most recent and comprehensive method detection limit studies for each instrument to be used to analyze project samples. If multiple instruments are used, the DL used for reporting purposes will represent the least sensitive instrument response for each analyte spiked.

Limit of Detection

The DL will be used to determine the LOD for each analyte and for all preparatory and cleanup methods routinely used on samples. The in-house LOD for each analyte is listed in the tables on Worksheet 15. The laboratories will be required to repeat the determination of the LOD if there are significant changes to the method or instrumentation prior to analysis of the first environmental samples for this project. The laboratories will maintain documentation for all DL and LOD determinations and verifications.

Limit of Quantitation

The laboratory in-house LOQ for each target analyte is presented in the tables on Worksheet 15. During analysis of the project environmental samples, the laboratories will verify LOQs by including a standard equal to or below the LOQ as the lowest point on the initial calibration curve.

If a result is greater than the LOD but less than the LOQ, the result will be reported as a detected concentration and flagged “J”. If no detected concentration is determined down to the LOD, the result will be reported as not detected (flagged “U”) at the LOD. The LOD will be adjusted for each sample based on dilution, final sample volume and sample weight. A detected result greater than or equal to the LOQ will be reported without a qualifying flag unless a specific QA/QC failure is associated with the data. For this project and for purposes of evaluation and reporting the LOQ will be considered equivalent to the reporting limit.

At a minimum, the LOQ must be verified quarterly. The LOQ and associated precision and bias must meet project-specific requirements and must be reported after verification. If the method is modified or major changes are made to the instrumentation, the LOQ must be verified and reported.

Sample dilution because of target and or non-target analyte concentrations or matrix interference could prevent LOQs from being achieved. Each sample must be initially analyzed while undiluted when reasonable. If dilution is necessary, both the original and diluted sample results must be reported and the dilution noted in the case narrative. Any samples that are not initially analyzed undiluted must have the express written approval of the Project Engineer within extraction and analysis holding time. Justification must be supported by matrix interference documentation such as sample texture, color, odor or results from other analyses of the same sample, to show that undiluted analysis is not possible. Appropriate cleanup procedures must be followed to minimize matrix effects on LOQs.

Calibration

All analytes reported must be present in the initial and continuing calibrations. The calibrations must meet the acceptance criteria specified in Worksheet 24 (Analytical Instrument Calibration). All results reported must be within the calibration range. Samples will be diluted, if necessary, to bring analyte responses within the calibration range and properly identified in the case narratives. Records of standard preparation and instrument calibration will be maintained and are available upon request. Records must clearly trace the standards and their use in calibration and quantitation of sample results.

Instrument calibration will be performed by beginning with the simplest approach first, the linear model through the origin and then progressing through other options until the acceptance criteria are met. In cases where an analyte has more than one acceptable calibration model, results from the simplest calibration model will be reported. If more than the minimum number of standards is analyzed for the initial calibration verification (ICV), all of the standards analyzed will be included in the ICV. The only exception to this rule is that a standard at either end of the calibration curve can be dropped from the calibration curve, providing that the requirement for the minimum number of standards is met and the low point of the calibration curve is at or below the quantitation limit for each analyte.

The CCV cannot be used as the LCS. A CCV will be performed daily before sample analysis, unless an initial calibration and second-source standard verification is performed immediately before sample analysis, and as required by the method.

Laboratory Control Samples

A LCS is a sample of known composition that is spiked with all target analytes. The LCS is used with each analytical batch to determine whether the method is in control. Each analyte in the LCS will be spiked at a level less than or equal to the midpoint of the calibration curve, which is defined as the median point of the curve instead of the middle of the range. The LCS will be carried through the complete sample preparation and analysis procedure.

At least one LCS will be included in each analytical batch. If more than one LCS is analyzed in an analytical batch, results from all LCSs will be reported. Failure of an analyte in any LCS will necessitate appropriate corrective action, including qualification of the failed analyte in all of the samples as required.

The in-house LCS control limits, which meet DoD QSM specifications, will be used for the project (Worksheet 15) until and unless new in-house limits are developed and approved for the project. When an analyte in the LCS is outside the acceptance limit, corrective action will be required. If an analyte in

the LCS exceeds the upper or lower control limit and no corrective action is performed, or the corrective action taken is deemed to be ineffective, an appropriate data qualifier may be applied during data validation to all associated sample results.

Marginal Exceedance

The laboratory may not use marginal exceedances as part of their data review process but are encouraged to contact the Project Engineer to discuss the problem and corrective action to be taken.

Matrix Spike and Matrix Spike Duplicate Samples

An MS or MSD is an aliquot of sample collected in the field and spiked in the laboratory with known masses and concentrations of all target analytes. The spiking will occur before sample preparation and analysis. Each analyte in the MS and MSD must be spiked at a level less than or equal to the midpoint of the calibration curve for that analyte. The MS/MSD is used to document potential matrix effects associated with a site and will not be used to control the analytical process. The FTL will select the samples for MS/MSDs.

The performance of the MS/MSD will be evaluated against the accuracy and precision limits (Worksheet 12, Worksheet 15). If either the MS or the MSD is outside the acceptance limits, the data will be evaluated to determine whether there is a matrix effect or analytical error. The determination will be made during data validation. If matrix effect is determined, the analytes in the parent sample will be qualified accordingly.

If the sample concentration exceeds the spike concentration by a factor of four or more, the associated parent sample data will not be qualified. The laboratory should communicate potential matrix difficulties to the Project Engineer so that an evaluation can be made with respect to the project-specific DQOs.

Surrogates

Surrogates are compounds similar to the target analytes in chemical composition and behavior in the analytical process, but not normally found in environmental samples. Surrogates are used to evaluate accuracy, method performance and extraction efficiency. Surrogates will be added to all environmental samples, controls, and blanks in accordance with method requirements.

The acceptance limits for surrogate recoveries are presented on Worksheet 15. If a surrogate recovery is outside the acceptance limit, corrective action must be performed. After the system problems have been resolved and system control has been re-established, the sample will be re-prepared and re-analyzed. If the surrogate outlier persists after re-analysis, the sample results from both the original and the re-analysis runs will be reported and discussed in the case narrative. The reported results will be evaluated during data validation and a decision on qualification of the affected data will be made.

Internal Standards

Internal standards are known amounts of standards that are added to a portion of a sample or sample extract and carried through the entire determination procedure. Internal standards are used as a reference for calibration and for controlling the precision and bias of the analytical method. Internal standards will be added to environmental samples, controls, and blanks, in accordance with the method requirements.

If the results of the internal standards are outside of the acceptance limits, corrective actions will be performed. After the system problems have been resolved and system control has been reestablished,

all samples analyzed while the system was malfunctioning will be re-analyzed. If corrective actions are not performed or are ineffective, an appropriate flag will be applied to the sample results.

Retention Time Windows

Retention time (RT) windows are used in GC analysis for qualitative identification of analytes. They are calculated from replicate analyses of a standard on multiple days. The procedure and calculation method are given in each method. The center of the RT window is established for each analyte and surrogate using the RT of the midpoint standard of the initial calibration.

If the RT is outside of the acceptance limits, corrective action will be performed. This applies to all continuing calibration verification subsequent to the ICV and to LCSs. If corrective actions are not performed or are ineffective, an appropriate flag will be applied to the sample results.

Method Blank

A method blank (MB) is an analyte-free matrix carried through the complete sample preparation and analytical procedure, and is used to assess potential contamination resulting from the analytical process.

A MB will be included in every analytical batch. The presence of analytes in a MB at concentrations greater than the LOD indicates the need for further assessment of the data. The source of contamination will be investigated and measures will be taken to correct, minimize, or eliminate the problem if the concentration exceeds one-half the LOQ. No analytical data will be corrected for the presence of analytes in the blanks.

If an analyte is detected in the MB and in the associated samples and corrective actions are not performed or are ineffective, the data will be evaluated during data validation and a decision on qualification of data will be made at that time.

Quality Control Checks

Holding Time Compliance

All sample preparation and analyses will be performed within the method-required holding times (see **Table 12**, Worksheet 19). For the target analytes (VOCs) not requiring sample preparation, holding time is calculated from the time of sample collection to the time of completion of all analytical runs.

Control Charts

Control charts are used to track laboratory performance over time. All analytes spiked into the LCS will be tracked via control charts by the laboratory. These charts are useful for identifying trends and problems in an analytical method and the laboratory uses these charts to establish in-house LCS control limits. The control charts will be updated as needed (for example, when there is a significant change to the analytical system). At a minimum, the charts will be updated annually and reviewed each time a data point is generated so that corrective action can be taken in a timely manner. These charts can also be used to benchmark a laboratory's performance against QAPP requirements to determine possible areas for improvement.

Standard Materials

Standard materials (including second source materials) used in calibration and sample preparation must be traceable to National Institute of Standards and Technology, USEPA, American Association of Laboratory Accreditation, or other equivalent approved source, if available. If a traceable standard

material is not available, the standard material proposed for use must be included in an addendum to this QAPP and approved before use.

The standard materials must be current, and the following expiration policy must be followed:

- Expiration dates for ampullated solutions should not exceed the manufacturer's expiration date or one year from the date of receipt, whichever comes first
- Expiration dates for laboratory-prepared stock and diluted standards must be no later than the expiration date of the stock solution
- Expiration dates for pure chemicals will be established by the laboratory and be based on chemical stability, possibility of contamination, and environmental and storage conditions
- The laboratory will label standard and QC materials with expiration dates and expired standard materials will be discarded

A second source standard will be used to independently confirm the initial calibration. A second source standard is a standard purchased from a vendor different from that supplying the material used in the initial calibration. The second source material can be used for the continuing calibration standards and/or for the LCS. Two different lot numbers from the same vendor do not normally constitute a second source. However, when a project requires analyses for which there is not a separate vendor source available, the use of different lot numbers from the same vendor will be acceptable to verify calibration.

Worksheet 29 - Project Documents and Records

The required data package deliverables during every aspect of the project are identified in this worksheet. These include, but are not limited to: 1) sample collection and field measurement records, 2) analytical records, and 3) QC records.

Sample Collection and Field Measurement Records

Sample collection and field measurement records generally include field log books, photo documentation, equipment decontamination records, sampling instrument calibration records, test pit logs, soil sampling logs, chain of custody forms, and air bills. Blank field forms are provided in **Attachment 2**.

Analytical Records

All records and data will be maintained in laboratory notebooks, summary tables, and/or other electronic or paper media.

Hardcopy and Analytical Data Deliverables

Hardcopy deliverables must be provided with a summary format forms package, equivalent to those specified in DoD QSM Version 5.0. Other delivery formats are also acceptable such as those from the latest versions of USEPA CLP Statements of Work for Organic and Inorganic Analyses or CLP-like as long as the format provides summarized, form oriented reporting and are fully validated. Other reporting formats require approval from the Project Engineer.

All hardcopy deliverables will be Level IV deliverables. All Level IV analytical data packages will be delivered using computer readable files (such as the Adobe Portable Document File [PDF] format). One PDF file will be submitted for SDG. The PDF files may be submitted electronically via compact disc. The laboratory data report must be organized in a format that facilitates identification and retrieval of data.

Electronic Data Deliverables

The laboratory will submit an EDD for each SDG. The EDD will contain all final laboratory data complete with the flags as applied by validation. EDDs to be submitted as a part of the final report will include the raw EDDs submitted by the laboratory, the post validation EDDs (for each SDG), the final validated data summary tables, complete with the validation flags applied during validation, and the laboratory PDF files for each SDG. Details regarding the data verification and validation procedures are presented in Worksheet 35 and Worksheet 36.

Quality Control Records

Table 20 Sample Collection and Field Records

Record	Generation	Verification	Storage location
Field logs	FTL	Project Manager	Project file
Chain of custody	FTL	FTL	Project file/Laboratory

Worksheet 30 - Analytical Services

Table 21 Analytical Services

Analyses:	VOCs (8260C)
Matrix:	Soil
Concentration Level:	Low/medium
Analytical SOP	VO 004
Primary Laboratory:	CTL
Laboratory Contact, Title:	Brett Szymanski, Project Manager
Laboratory Address	1230 Lange Court Baraboo, Wisconsin 53913
Laboratory Telephone Numbers:	Main: 608-356-2760 Fax: 608-356-2766
Certification:	DoD ELAP (3806.01)
Accreditation Expiration:	06/30/2016 (DoD ELAP) (currently under renewal)
Sample Delivery Method:	FedEx Overnight services
Data Deliverable:	14 Calendar Days for results/21 days for Level IV

Worksheet 31 - Planned Project Assessments

Periodic assessments will be performed during the course of the project so that the planned project activities are implemented in accordance with this document. The type, frequency, and responsible parties of planned assessment activities to be performed for the project are summarized in the table below.

Table 22 Planned Project Assessments

Assessment Type	Frequency	Report for Documenting Assessment Findings	Person(s)/Organization Responsible for Performing Assessment	Person(s) Responsible for Identifying and Implementing Corrective Actions
Field Document Review	Daily	Findings to be included in Project Reports	Ben Stewart, Trinity FTL	Todd Calhoun, Trinity Project Manager
Field Procedure Assessment	As work progresses	Findings to be included in Project Reports	Ben Stewart, Trinity FTL	Todd Calhoun, Trinity Project Manager
Safety & Health Audit	As needed	Findings to be included in Project Reports	Jeanette Baldwin, Trinity Corporate HSO	Jeanette Baldwin, Trinity Corporate HSO

Worksheet 32 - Assessment Findings and Corrective Action Responses

Periodic assessments will be performed during the course of the project so that the planned project activities are implemented in accordance with this document. The type, frequency, and responsible parties of planned assessment activities to be performed for the project are summarized in the table below.

Table 23 Assessment Findings and Corrective Action Reponses

Assessment Type	Frequency	Report for Documenting Assessment Findings	Person(s)/Organization Responsible for Performing Assessment	Person(s) Responsible for Identifying and Implementing Corrective Actions
Field Document Review	Daily	Findings to be included in Project Reports	Ben Stewart, Trinity FTL	Todd Calhoun, Trinity Project Manager

Worksheet 33 - Quality Assurance Management Reports

Periodic assessments will be performed during the course of the project so that the planned project activities are implemented in accordance with this document. The type, frequency, and responsible parties of planned assessment activities to be performed for the project are summarized in the table below.

Table 24 Periodic Assessment Schedule

Type of Report	Frequency	Projected Delivery Date(s)	Person(s)/Organization Responsible for Report Preparation	Report Recipient(s) (title and organizational affiliation)
Field Daily Quality Control Report (DQCR)	Daily MIP/DPT soil activities	Daily	Ben Stewart, Trinity FTL	Todd Calhoun, Trinity Project Manager
Data Verification and Validation Report	After receiving data from the laboratory	TBD	LDC	Robyn Peterson, Trinity Project Engineer
Internal Project Report Review	Once per report	TBD	Todd Calhoun, Trinity Project Manager	Laura Roebuck, CESAM Technical Manager Joan Hutton, BEC Tom Holmes, HDR Project Manager
External Project Report Review	Once per report	TBD	Todd Calhoun, Trinity Project Manager	Diedre Lloyd, USEPA Region 4 Remedial Project Manager Jamie Woods, TDEC Remedial Project Manager Laura Roebuck, CESAM Technical Manager Joan Hutton, BEC Tom Holmes, HDR Project Manager

Worksheet 34 - Data Verification and Validation Inputs

To confirm that scientifically-sound data of known and documented quality are used in making project decisions, the following three-step data review will be performed:

- Step I (verification) will confirm that all sampling and analytical requirements have been completed and documented.
- Step II (validation) will assess whether the sampling and analytical processes conform to stated requirements including those in the contract, method and QAPP.
- Step III (usability assessment) will determine whether the resulting data are suitable as a basis for the decision being made.

Worksheet 35 (Data Verification Procedures) and 36 (Data Validation Procedures) describe the processes to be followed for the above steps, respectively. This worksheet establishes the procedures that will be followed to verify project data including, but are not limited to, sampling documents and analytical data package.

Table 25 Data Verification Worksheet

Item	Description	Verification (completeness)	Validation (conformance to specifications)
Planning Documents/Records			
1	Approved QAPP	X	
2	Contract	X	
3	Field SOPs	X	
4	Analytical SOPs	X	
Field Records			
5	Field logbooks	X	X
6	Equipment calibration records	X	X
7	Chain of custody forms	X	X
8	Sampling logs	X	X
9	Relevant correspondence	X	X
10	Change orders/deviations	X	X
11	Field audit reports	X	X
12	Field corrective action reports	X	X
Analytical Data Package			
13	Cover sheet (laboratory identifying information)	X	X
14	Case narrative	X	X
15	Internal laboratory chain of custody	X	X
16	Sample receipt records	X	X

Item	Description	Verification (completeness)	Validation (conformance to specifications)
17	Sample chronology (dates and times of receipt, preparation, and analysis)	X	X
18	Communication records	X	X
19	DL/LOD/LOQ establishment and verification	X	X
20	Instrument calibration records	X	X
21	Definition of laboratory qualifiers	X	X
22	Results reporting forms	X	X
23	QC sample results	X	X
24	Corrective action reports	X	X
25	EDD	X	X

Worksheet 35 - Data Verification Procedures

Data verification is a completeness check to confirm that all required activities were conducted, all specified records are present, and the contents of the records are complete. It applies to both field and laboratory records.

Table 26 Data Verification, Validation, and Usability Assessment Responsibilities

Records Reviewed	Description	Person(s) Responsible
Field SOPs	Verify that the sampling SOPs were followed	Ben Stewart/Trinity FTL
Analytical SOPs	Verify that the analytical SOPs were followed	Laboratory QA Officer Robyn Peterson/Trinity Project Engineer Todd Calhoun/Trinity Project Manager
Method QC Results	Verify that the required QC samples were run and met required limits	Laboratory QA Officer Robyn Peterson/Trinity Project Engineer Todd Calhoun/Trinity Project Manager
Data Validation	Validate 100 % of the data to confirm quality as defined on Worksheet 14 (Summary of Project Tasks)	Stella Cuenco/LDC
Data Usability Evaluation	Evaluate data based on precision, accuracy, representativeness, comparability and completeness for project objectives	Robyn Peterson/Trinity Project Engineer Todd Calhoun/Trinity Project Manager
Field Documentation	Verify accuracy and completeness of field notes	Todd Calhoun/Trinity Project Manager Robyn Peterson/Trinity Project Engineer

Worksheet 36 - Data Validation Procedures

The objective of the data validation is to assess the performance associated with the analysis in order to determine the quality of the data. This will be accomplished by evaluating whether the collected data comply with the pre-defined project requirements (including method, procedural, or contractual requirements) and by comparing the collected data with criteria established based on the project DQOs.

All types of data, including screening data and definitive data, are relevant to the usability assessment. The following sections focus on the data review requirements for definitive data only.

Table 27 Validation (Stage 3) Summary

Stage	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator
3	Soil	VOCs	No response to highest response	Defined below	Stella Cuenco/LDC

Data Review Requirements for Definitive Data

Scientifically sound data of known and documented quality that meet the PQOs are essential to the decision-making process. Data will be examined and evaluated to varying levels of detail and specificity by multiple personnel who have different responsibilities within the data management process. Data review includes verification, validation, and usability assessment. The data review process will be documented to facilitate efficient and accurate assessment of data quality and usability. The overall usability of the data is indicated with appropriate qualifiers.

Data verification is used to confirm that the specified requirements have been performed.

Data validation extends data verification and is used to confirm that the requirements for a specific intended use are fulfilled. Data validation is the systematic approach of evaluating the compliance of the data with the pre-defined requirements of the project (including method, procedural, or contractual requirements) and compliance of the data against criteria based on the quality objectives documented in this document. The purpose of data validation is to assess the performance associated with the analysis in order to determine the quality of the data. Data validation includes a determination, to the extent possible, of the reasons for any failure to meet performance requirements, and an evaluation of the impact of such failures on the usability of the data.

Data usability assessment is an evaluation based on the results of data verification and validation in the context of the overall project decisions or objectives. The assessment is used to determine whether the project execution and resulting data meet the PQOs. Both sampling and analytical activities must be considered, with the ultimate goal of assessing whether the final, qualified results support the decisions to be made with the data.

Laboratory Requirements

Each analytical data package must contain adequate information and be presented in a clear and concise manner. The contents of each package must be equivalent to a CLP-like Level IV data package. Minimum requirements include the following:

- Cover sheet, which identifies the laboratory generating the data and the project for which the data were generated and signed by the appropriate laboratory personnel

- Table of contents
- Case narrative, which summarizes samples, analyses, and discusses any issues that may affect data usability
- Analytical results
- Laboratory LODs, and LOQs
- Sample management records
- CLP-like QC summary forms for the QC elements (including tuning, calibration, surrogates, LCS, MS/MSD, etc.)
- All supporting raw data for project, field, and lab QC samples (including chromatograms, quantitation reports, formulas and example calculations and mass spectral data)

Each laboratory data package should represent a group of samples received, prepared, and analyzed together in an analytical batch, with associated laboratory quality control samples (i.e., SDG). The complete data package for each SDG will be submitted electronically as a computer readable file (such as Adobe's PDF). In addition to the PDF, an EDD in a Chemlab flat file will be submitted with each SDG. The EDD deliverables will be used to perform the automated data review for the definitive data.

The data management platform will evaluate each EDD to determine the PARCCS of the data, based on the information contained in the data package. Additional manual evaluation of the data package may be required for QC requirements not covered by this software.

A schedule will be established so that laboratory data deliverables (including the PDF and EDD) for each SDG are provided in a timely manner for data review, validation, assessment, and use. The data deliverables for each SDG will not be considered complete until the Project Engineer has evaluated them for completeness and compliance. Any deliverable found to be non-compliant will be returned to the laboratory for correction and re-submittal.

Laboratory Data Reporting Requirements

The case narrative of each analytical data package will include but is not be limited to the following:

- Table summarizing samples received, correlating field sample numbers, laboratory sample numbers, and laboratory tests completed
- Discussion of any and all issues that may affect data usability (such as temperature, preservation, sample containers, air bubbles, and multi-phases)
- Samples received but not analyzed and the reasons why
- Discussion of holding time exceedances for sample preparation and analyses
- Summary of any and all instances of outliers and corrective actions taken
- Identification of samples and analytes for which manual integration was necessary
- Discussion of all qualified data and definition of qualifying flags

The following requirements should also be met for the reporting:

- MDLs, LODs, LOQs and sample results should be reported with the appropriate number of significant figures for the measurement.

Samples will be analyzed undiluted if possible. Non-detects will be reported to the LODs. MDLs, LODs, and LOQs for minority chemicals in highly-contaminated samples may have to be adjusted because of dilutions.

Manual Integrations

Manual integrations are an integral part of the chromatographic analysis process and will be done only as a corrective action measure. Examples of instances where manual integration would be warranted include, but are not limited to, co-eluting compounds resulting in poor- peak resolution, a misidentified peak, an incorrect retention time, or a problematic baseline.

When manual integrations are used, they must follow the procedures outlined in the laboratory's SOP for the method. Any and all instances of manual integration must be identified in the case narrative.

Laboratory Data Review Requirements

All definitive data will be reviewed first by the laboratory analyst and then by the laboratory supervisor of the respective analytical section using the same criteria before they are submitted to Trinity. This internal data review process, which is multi-tiered, should include all aspects of data generation, reduction, and QC assessment. Elements for review or verification at each level must include, but are not limited to, the following:

- Sample receipt procedures and conditions
- Sample preparation
- Appropriate analytical SOPs and methodologies
- Accuracy and completeness of analytical results
- Correct interpretation of all raw data, including all manual integrations
- Appropriate application of QC samples and compliance with established control limits
- Verification of data transfers
- Documentation completeness
- Accuracy and completeness of data deliverables (hard copy and electronic)

Laboratory Data Evaluation

All definitive data will be reviewed, reduced, and validated by the laboratory following the procedures specified in the laboratory's SOPs for data reduction and validation.

Data qualifiers should be applied by the laboratory as part of their internal validation activities. The allowable data qualifiers for definitive data are Q, M, J, B, UJ, U, and 4. The definitions of the data qualifiers are provided on the table below. Flagging criteria apply when acceptance criteria are not met and corrective actions were not successful or not performed. The data qualifiers must be reviewed by the supervisor of the respective analytical sections.

The laboratory QA section should perform a 100 percent review of 10 percent of the completed data packages. The third-party validator will subsequently evaluate the flags applied by the laboratory as part of their data validation and usability assessment activities. The flags may be accepted, modified, or rejected. For all data qualifiers that are changed, clear justification will be provided. All Q-flagged data will be evaluated and either accepted without qualification, accepted with qualification, or rejected.

Table 28 Laboratory Data Qualifiers

Qualifier	Description
Q	This indicates that one or more QC criteria fail. Data must be carefully assessed by Trinity (or project team) with respect to the project-specific requirements and evaluated for usability. Subsequent assessment by DoD may result in rejection of data.
M	Manually integrated compound
J	The analyte was positively identified and result is between the DL and LOQ; the quantitation is an estimate because of discrepancies in meeting certain analyte-specific QC criteria.
B	The analyte was found in an associated blank above one half the LOQ, as well as in the sample.
U	The analyte was analyzed for but not detected.
UJ	The analyte was not detected; however, the result is estimated because of discrepancies in meeting certain analyte-specific QC criteria.
4	MS, MSD: The analyte present in the original sample is 4 times greater than the matrix spike concentration; therefore, control limits are not applicable.

Method Blank Evaluation Guidance

For MBs, the source of contamination should be investigated and documented by the laboratory. The results of the investigation should be included in the case narrative. If all samples associated with MB contamination are not reanalyzed, the results will be reported, by the Laboratory, with a B-flag, along with any other appropriate data qualifier. If an analyte is found only in the MB, but not in any batch samples, no flagging is necessary. Sample results affected by the MB contamination will be evaluated during data validation and the final result qualified accordingly.

Data Verification Guidelines

The Project Engineer will review the data verification performed by the laboratory for completeness and accuracy. Data verification may be done both electronically and manually. Data verification may include but is not limited to the following:

- Sampling documentation (such as the chain-of-custody form)
- Preservation summary and holding times
- Presence of all analyses and analytes requested
- Use of required sample preparation and analysis procedures
- LODs and LOQs
- Correctness of concentration units
- Case narrative

Data Validation Guidelines

Raw Data Review

The data validation process builds on data verification. The third-party validator will review and evaluate the laboratory results. Following automated review of the data, the third-party validator will review the

results and qualifiers applied by the software. Manual validation of analytical data packages for all SDGs to address QC requirements not addressed by the software (such as tuning verification, initial and continuing calibration, quantitation, multiple run samples, instrument performance, and sample preservation) will be performed. The Project Engineer will request a Level IV data package from the Laboratory in order to perform the manual review to address any problems found during the electronic data validation.

Based on the review of the data results and manual evaluations, the Project Engineer may change or add qualifiers to the data and document reasons for the changes within the data software. Any additions or changes to the data validation qualifiers based on the manual data review will be incorporated into the final data deliverables and discussed in the Quality Control Summary Report.

Data validation guidelines have been developed in accordance with the method requirements, USEPA's National Functional Guidelines for Organic Data Review, professional judgment, and DoD QSM (Version 5.0) requirements. The following information will be reviewed as part of a Stage 3 type summary data validation performed using the data management system platform:

- Chain of custody documentation
- Holding time
- QC sample frequencies
- MBs
- LCS
- Surrogate spikes
- MS/MSD
- Field and laboratory duplicate precision
- Initial and continuing calibration information
- Internal standards
- Case narrative review and other method specific criteria

Blank Evaluation Guidelines

The Project Engineer will evaluate laboratory B-qualified data such as MBs, as well as other field blanks based on the concentration of the analyte in the samples in relation to the concentration in the blank. The B-flag may not be used if the analyte concentrations in the samples are much higher (≥ 5X) than in the blank (≥ 10X in case of common laboratory contaminants). Any blank contamination that may impact data usability must be discussed in conjunction with project-specific goals. When a data set contains low-level detects in field samples and has associated field or laboratory blanks that have detects at similar concentrations, this suggests that the low-level detects in these field samples may be artifacts because of either field or laboratory practices. A sample detect that is ≤ 5X the blank concentration (≤ 10X for common lab contaminants) shall be considered a non-detect and flagged "U".

Duplicate Evaluation Guidance

QC measures for precision include field duplicates, laboratory duplicates, MSDs, analytical replicates, and surrogates. These measures will be evaluated by the laboratory and qualified according to applicable procedures, with the exception of the field duplicates. Specifically, field duplicates should be sent to the laboratory as blind samples and should be given unique sample identification numbers. These sample results can be used to assess field sampling precision, laboratory precision, and,

potentially, the representativeness of the matrix sampled. Flagging of results associated with field duplicates should be assigned such that the level of uncertainty required, as provided by the project-specific objectives, is taken into account.

Poor overall precision may be the result of one or more of the following: field instrument variation, analytical measurement variation, poor sampling technique, sample transport problems, or heterogeneous sample matrices. To identify the cause of imprecision, the project team should evaluate the field sampling design rationale and sampling techniques, and review both field and analytical duplicate sample results. If poor precision is indicated in both the field and analytical duplicates, then the laboratory may be the source of error. If poor precision is limited to the field duplicate results, then the sampling technique, field instrument variation, sample transport, and/or nature of the matrix may be the source of error.

Matrix Interference Evaluation Guidance

In the case of matrix interference, data validation qualifiers may be applied to additional samples from the same site and same matrix, based on the professional judgment of the data validator. In this case, it is the responsibility of the validator to document the reasons for the additional qualifiers.

Flagging Conventions

The allowable final data qualifiers for definitive data and the hierarchy of data qualifiers, listed in order of the most severe through the least severe, are R, J and UJ. Their definitions are summarized in below.

Table 29 Usability Assessment Data Qualifiers

Qualifier	Description
R	The data are rejected because of deficiencies in meeting QC criteria and may not be used for decision making.
J	The analyte was positively identified and result is between the DL and LOQ; the quantitation is an estimate because of discrepancies in meeting certain analyte-specific quality control criteria.
U	The analyte was analyzed for but not detected. The analyte was also detected in an associated laboratory or field blank at a concentration comparable to the concentration in the sample. The reported result has been re-qualified as non-detected.
UJ	The analyte was not detected; however, the result is estimated because of discrepancies in meeting certain analyte-specific quality control criteria.

The following two tables present the specific guidelines for applying these data usability qualifiers.

Table 30 General Data Qualifying Conventions

QC Requirement	Criteria	Flag	Flag Applied To
Holding Time	Time exceeded for extraction or analysis	J for the positive results; R or UJ for non-detects*	All analytes in the sample
Sample Preservation	Water; not preserved >7 days Water; preserved >14 days Non aqueous; preserved or not	J positive results; R or UJ for non-detects* Use professional judgment	Sample
Temperature out of control	> 6° Celsius	Professional judgment or if grossly outside; J for positive results; R or UJ for non-detects*	Sample
Instrument Tuning	Ion abundance method-specific criteria not met	R for all results	All associated samples in analytical batch
Initial Calibration	All analytes must be within method-specified criteria	J for positive results; non-detects (use professional judgment)	All associated samples in analytical batch
Second Source Check or Continuing Calibration	All analytes must be within method-specified criteria	High Bias: J for positive results, no flag for non-detects Low Bias: J for positive results, UJ for non-detects	All associated samples in analytical batch
Low Level Calibration Check or Interference Check Sample	All analytes must be within 20% of expected value	High Bias: J for positive results, no flag for non-detects Low Bias: J for positive results, UJ for non-detects R for all non-detects greater than twice the control criteria	All associated samples in analytical batch
LCS	%R > UCL %R < LCL	J for the positive results; no qualification for the non-detects; J for the positive results; UJ for the non-detects	The specific analyte(s) in all samples in the associated analytical batch
Internal Standards	Area > UCL Area < LCL Sample is re-extracted and reanalyzed and recovery outside of criteria is confirmed as a matrix effect	J for positive results J for positive results; UJ for the non-detects	Sample

QC Requirement	Criteria	Flag	Flag Applied To
Surrogate Spikes	%R > UCL	J for positive results	Sample
	%R < LCL and >10%	J for positive results; UJ for non-detects	
	%R <10%	J for positive results; R for non-detects	
	Excessive dilution*	No flag required	
Blanks (MB, RB, or TB)	Analyte(s) detected > 1/2 LOQ (use the blank of the highest concentration)	U for positive sample results < 5x highest blank concentration (<10x for common lab contaminants)	All samples in preparation, field or analytical batch, whichever applies
FDs or field replicates	RPD >30%	J for the positive results	The specific analyte(s) in both parent and duplicate
MS/MSD	MS or MSD % R>UCL	Cross reference with LCS. Possible J for positive results	The specific analyte(s) in the parent sample
	MS or MSD % R<LCL or MS/MSD RPD>Control Limit;	Cross reference with LCS. Possible J for positive results; UJ for non-detects	
	Sample concentration > 4x spike concentration;	No flag required	
	Excessive dilution*	No flag required	
Post-Digestion Spike (metals)	All analytes must be within 25% of expected value	High Bias: J for positive results Low Bias: J for positive results; UJ for non-detects	The specific analyte(s) in the parent sample
Retention Time Window	Analyte within established window	R for all results	Sample

* = Based on analyte-specific review

LCL – lower confidence limit
LCS – laboratory control sample
LOQ – Limit of quantitation
MS – matrix spike
MSD – matrix spike duplicate
%R – percent recovery
QC – quality control
RPD – relative percent difference
RSD – relative standard deviation
UCL – upper confidence limit

Table 31 Data Qualifying Conventions - Quantitation

Criteria	Flag
< LOD	U, UJ at the LOD
> LOD < LOQ	J
> LOQ	As needed
> high standard/linear range	J

Examples:
LOD = 2, LOD = 4, LOQ = 15, sample is undiluted.
Example #1: Analytical result: not detected; reported result: <4U.
Example #2: Analytical result: 10; reported result: 10J.
Sample #3: Analytical result: 15; reported result: 15.

Worksheet 37 - Data Usability Assessment

The data usability assessment is an evaluation based on the results of data verification and validation in the context of the overall project decisions or objectives. The assessment is used to determine whether the project execution and resulting data meet the project DQOs. Both the sampling and analytical activities must be considered, with the ultimate goal of assessing whether the final, qualified results support the decisions to be made with the data.

The following sections summarize the processes to determine whether the collected data are of the right type, quality, and quantity to support the environmental decision-making for the project, and describe how data quality issues will be addressed and how limitations of the use of the data will be handled.

Personnel Responsible for Participating in the Data Usability Assessment

- Robyn Peterson, PE – Trinity Project Engineer
- Todd Calhoun, PG – Trinity Project Manager
- Ben Stewart – Trinity FTL

Summary of Usability Assessment Processes

Data gaps may result if:

- A sample is not collected
- A sample is not analyzed for the requested parameters
- The data are determined to be unusable

If data gaps exist, the need for further investigation will be determined by the project leaders.

The Project Engineer and each laboratory's QA Officer will confirm that the collected data meet the LODs, LOQs, and laboratory QC limits specified in this document. During the data validation assessment, non-conformances will be documented, and data will be qualified accordingly. The Project Engineer will determine whether the data are usable based on the requirements specified in this document.

All data as qualified during data validation are considered usable, with the exception of rejected data ("R" qualified data). Estimated results are considered usable.

Usability Summary Documentation

To ensure that quality data are continuously produced during analysis, and to enable the subsequent compliance review, systematic QC checks are incorporated into the sampling and analyses to show that procedures and test results remain reproducible and that the analytical method is without unacceptable bias. Systematic QC checks include the comparability of field and laboratory duplicates as well as the laboratory performance for each batch of samples. Discussion will cover PARCCS.

Precision

Total precision is the measurement of the variability associated with the entire sampling and analytical process. The required levels of precision for each method, matrix and analyte are provided in Worksheet 15 (Reference Limits and Evaluation). Laboratory precision is measured by the variability associated with duplicate (two) analyses. The field precision will be evaluated through the use of field duplicates, while the laboratory precision will be evaluated through the use of spike duplicates. For duplicate sample results, the precision is evaluated using the RPD.

If calculated from duplicate measurements:

$$\left(\frac{x_1 - x_2}{(x_1 + x_2)/2} \right) \times 100$$

Where:

X_1= larger of the two observed values

X_2 = smaller of the two observed values

Accuracy

Accuracy reflects the total error associated with a measurement. A measurement is considered accurate when the reported value agrees with the true value or known concentration of the spike or standard within acceptable limits. The accuracy will be evaluated through the use of LCS, MS, and surrogates. In each case the accuracy will be determined by calculating the percent recovery (%R) for each target analyte.

The formula for calculation of accuracy is included below as *%R*. Accuracy requirements are listed for each method, matrix and analyte in Worksheet 15 (Reference Limits and Evaluation).

For measurements where matrix spikes are used:

(value of spiked sample – value of unspiked sampled)/value of added spike X 100

Representativeness

Representativeness is a qualitative term that is related to the sample collection procedures. Representativeness is determined by proper program design, with consideration of elements such as sampling locations. Samples that are improperly collected or preserved, or are analyzed beyond the method required holding time, would not provide data that represent the sampling site. In addition, if the laboratory subsampling criteria were not met (i.e. proper premixing and homogenizing), the resulting data would not be representative of the initial sample collected.

Comparability

Comparability is a qualitative indicator of the confidence with which one data set can be compared to another data set. The objective is to produce data with the greatest possible degree of comparability. Comparability is achieved by using standard methods for sampling and analysis, reporting data in standard units, using standardized data collection forms and using standard and comprehensive reporting formats. In order to ensure that the data sets are comparable, the same method will be used for each sampling event.

Completeness

Completeness is calculated for the aggregation of data for each analyte measured for any particular sampling event or other defined set of samples (for example, by site). Completeness is calculated and reported for each method, matrix, and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are all results not qualified with an R-flag after data validation. The goal for completeness is 95 percent for aqueous samples.

Completeness is calculated as follows for all measurements:

%*C*=100 % x [A/T]

Where:

%C = percent completeness

A = number of individual analyte results deemed valid

T = total number of results

Sensitivity

Sensitivity is the ability of an analytical method or instrument to discriminate between measurement responses representing different concentrations. Sensitivity requirements include the establishment of various limits such as calibration requirements, instrument LODs and LOQs. The project QA/QC control on method requirements has been established to be compliant with the DoD QSM Version 5.0. Project specific LOD and LOQs are established in Worksheet 15.

References

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Attachments

Attachment 1 Field Standard Operating Procedures

1.0 SOP 01 – FIELD RECORDS AND DOCUMENTATION 1-1
1.1 Procedure 1-1
1.1.1 Documentation of Field Records 1-1
1.1.2 Field Logbook Format 1-1
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1.3 Copying and Filing of Field Records 1-3
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1.0 SOP 01 – FIELD RECORDS AND DOCUMENTATION

This Standard Operating Procedure (SOP) describes the objectives necessary to provide consistent procedures and formats for collecting clear and concise field records and field documentation of field activities conducted at the Defense Depot Memphis, Tennessee (DDMT). This SOP focuses only on the procedures for documenting field activities in the field logbook and Daily Quality Control Reports (DQCRs). Important documents used to develop this SOP are U.S. Environmental Protection Agency (USEPA) Science and Ecosystem Support Division (SESD) Procedure SESDPROC-010-R5, *Logbooks* (USEPA, 2013).

1.1 Procedure

It is the responsibility of the Field Team Leader (FTL) to scribe field activities in the field logbook. However, if necessary, it is acceptable for the FTL to designate field personnel to take this responsibility. Personnel will use only bound field logbooks that have sewn and consecutively numbered pages that meet USEPA guidance for the maintenance of field records (USEPA, 2013).

The following materials will be used for recording field records in the logbook:

- Field logbook
- Pens, containing water-proof ink
- Calculator
- Means to tell time (e.g. wristwatch, cell phone, laptop computer)

Management of the field logbooks will be based on specific conditions and requirements of each project. The Project Manager (PM), however, will ensure that all field notes can be efficiently traced, filed, and retrieved.

1.1.1 Documentation of Field Records

All field data will be recorded in waterproof ink. If errors are made in any field logbook, field form, chain of custody form, or any other field record document, corrections will be made by crossing a single line through the error, entering the correct information, and initialing.

1.1.2 Field Logbook Format

Logbook entries will be made in the following format. Documentation and reporting of events and activities will be made in chronological order. Every page will contain the date, recorded at the top, left-hand corner, followed by the site name and client of the project. At the beginning of each day, the first four entries will be “Personnel/Contractors on Site”, “Weather”, “Anticipated Scope of Work for the Day”, and the “FTL for the project”. The time of every entry will precede the field note, and be listed in columnar form down the left-hand side of each page. Military time will be used (for example, 1300, rather than 1:00 PM.)

All calculations, results, and calibration data (including the calibration media’s lot number and expiration date) for field sampling and serial numbers for field equipment will be recorded in the field logbooks or recorded on approved field forms.

All field analyses, measurements, and samples will be traceable to specific pieces of field equipment, and to the field investigator in the field logbook, or specific field form. Therefore, the reconciliation of later problems can be better resolved.

All samples collected in the field will be recorded in the logbook. Mandatory information regarding the sample are sample time, sample name, matrix, and laboratory analyses. The depth interval relative to a

measuring point for all soil, sediment, and surface water samples shall also be recorded. All QC/QA samples will also be recorded with the same mandatory information. This information must match what is recorded on the COC and the sample label. Additional information about the sample will be recorded as warranted, including unique circumstances or sample characteristics that could affect data evaluation. soil lithology, and detected odors.

Pertinent information on health and safety will also be logged in the field book. This includes time of daily health and safety meeting and safety issues discussed, times and reasons for breaks or stop work (e.g., excessive heat or cold conditions, or other inclement weather scenarios), and any other unanticipated health and safety events or issues. Resolutions to applicable health and safety issues will be recorded.

All personnel on site, visitors' names, association, and time of arrival/departure, etc., will also be recorded in the field logbook.

All phone calls to the project manager, client, etc. will be recorded with issues discussed. Additionally, any important discussions had in the field with property owner, project manager, client, etc. will be recorded.

The personnel will initial at the bottom, right-hand or left-hand corner of each completely filled out page. Additionally, at the end of each day's entry or particular event, the personnel will draw a diagonal line originating from the bottom left corner of the page to the conclusion of the entry and sign along the line indicating the conclusion of the entry of the day's activity.

If an entry is made in a non-dedicated logbook, then the date, project name, and project number will be entered left to right, respectively, along the top of the right page. All other aspects of field record keeping will follow guidelines outlined in the previous paragraphs of this section.

Once completed, these field logbooks become formal records and must be maintained as part of the project files. Periodic audits of field logbooks should be conducted by the PM to ensure compliance with this procedure.

Since field records are the basis for later written reports, the language should be objective, factual, and free of personal feelings or other terminology which might prove inappropriate. However, the personnel should not feel limited to record only the information previously outlined. Any and all information pertaining to the site or project that could affect data interpretation or management decision-making should be recorded.

1.2 Daily Quality Control Report and Format

DQCRs will be completed daily by the FTL. The purpose of the DQCRs is to provide a one-page summary of the daily field activities to applicable stakeholders (for example, client and/or contracting agency).

Different projects/clients may require specific DQCRs, but commonly require the recording of the following information:

- Project number
- Project name and location
- Date
- Temperature range
- Wind conditions
- Personnel on site

- Summary of site activities
- Level of health and safety protection
- Instruments used (including serial numbers)
- Calibrations performed (with lot numbers and expiration of calibration solutions)
- Instrument problems (and corrective actions taken)
- Soil borings/well installation details (with hand-held coordinates, if necessary)
- Samples collected
- Summary of sample collection methods
- Quality control samples collected
- Additional remarks

DQCRs will be completed on a daily basis. These reports will be reproduced and sent to the client or contracting agency, as required per the contract agreement. These records may be submitted in hardcopy or by electronic files sent via email.

1.3 Copying and Filing of Field Records

During field activities, the field logbooks will be copied on a periodic basis. The FTL is responsible for making copies of logbook pages and DQCRs. The PM is responsible for ensuring that copies are maintained as project files.

When an individual logbook is full, it will be submitted to the PM for final cataloging and filing. The logbooks will be stored at Trinity's office.

All non-bound field records (for example, DQCRs, drilling logs, well construction forms, sampling records, and chain of custody copies) will be completed on the same work day, scanned, and turned in to the Project Manager the following work day. The originals will be filed by the PM or, as designated by the PM, the FTL in the project file.

All field data collected using electronic data loggers or computer entry forms will be downloaded, as soon as practical, to the Trinity server.

1.4 References

USEPA, 2013. *Logbooks,* SESDPROC-010-R5. May.

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2.0 SOP 02 – DRILLING AND SOIL SAMPLING

This Standard Operating Procedure (SOP) describes the methods and procedural guidelines for drilling and soil sampling operations in support of environmental investigation activities at the Defense Depot Memphis, Tennessee (DDMT). Important documents used to develop this SOP are U.S. Environmental Protection Agency (USEPA) Science and Ecosystem Support Division (SESD) Guidance SESDGUID-101-R1, *Design and Installation of Monitoring Wells* (USEPA, 2013), Procedure SESDPROC-205-R3, *Field Equipment Cleaning and Decontamination* (USEPA, 2015), and SESDPROC-300-R3, *Soil Sampling* (USEPA, 2014).

2.1 Overview

The selected method for the collection of soil samples as part of the Membrane Interface Probe (MIP) survey will be via direct push technology (DPT) drilling and soil sampling. DPT drilling will provide an effective method for boring advancement and soil sampling based on geologic conditions.

Based on the proposed location of the MIP survey area being on uneven terrain, a track mounted DPT rig will be utilized. Drilling will be conducted by a Tennessee-licensed subcontractor with experience on similar projects and geology. All necessary permits (as applicable) will be obtained prior to initiation of drilling operations.

2.2 Health and Safety

Specific tasks and general safety requirements are addressed in the *Site Safety and Health Plan* (SSHP), Environmental Restoration Support, Defense Depot Memphis, Tennessee, Final (Trinity, 2016). Each individual supporting the MIP/DPT soil sampling activities is required to have read and understood the SSHP. Personal protective equipment (PPE) will be worn at all times during drilling activities and, at a minimum will consist of hard hats, steel toed shoes, safety glasses, hearing protection, and high visibility clothing.

All drilling locations will be cleared for underground and above ground utilities prior to beginning drilling activities. Prior to the start of drilling activities, the drilling subcontractor will hand auger each drilling location to a depth of 4-feet below ground surface (bgs) in order to verify that no underground utilities or objects are present.

2.3 Personnel Qualifications and Responsibilities

Field activities will be directed by the Field Team Leader (FTL), a mid- or senior level geologist with experience in the planned drilling activities. Drilling will be performed by a licensed driller and crew familiar with the requirements of this tasks. Additional details are provided in the UFP-QAPP Work Plan and the SSHP.

2.4 Start-Up Activities

After arrival on-site, but prior to beginning drilling activities, the following activities will be performed:

- Complete equipment and supply checklists and verify that required documentation and equipment are on-site and in working order
- Confirm drilling locations are clearly marked and review locations for hazards including overhead and underground utilities
- Calibrate field equipment
- Conduct team safety meeting

2.5 MIP Borings

Borings advanced for MIP soil gas screening will be performed with a DPT drilling rig. A MIP is a semi-quantitative field screening device that can detect VOCs in soils and sediment. Generally, the technique can only detect total VOCs; it cannot provide specificity of analytes. Logging with the MIP can produce a real-time vertical log of VOCs with depth. Commonly, the MIP probe also includes a tip that measures the soil or water conductivity. This information can be used to correlate contamination to soil stratigraphy.

Specific data collection will be performed with specialized MIP tooling consisting of a photoionization detector (PID), flame ionization detector (FID), and electron capture device (ECD). A MIP is commonly used for PIDs for BTEX, FIDs for other petroleum hydrocarbons (straight and branch chained alkanes), and ECD for chlorinated volatile organic compounds (CVOCs). The detection limits for these devices range from 2.5 parts per billion (ppb) for ECDs to about 1 part per million (ppm) for the PIDs and FIDs but are considered quantitative measurements.

Details on the collection and analysis of the data is provided in the SOP for *Geoprobe® Membrane Interface Probe (MIP), Technical Bulletin No. MK3010* (Geoprobe, 2015).

2.6 Soil Borings

This section describes the procedures for advancement and sampling of soil borings. Stringent protocols will be followed to ensure that geologic and chemical data produced are reliable and of high quality, that contaminant migration does not occur, and that contaminants are not introduced to the subsurface or to samples obtained. This procedure applies to all Trinity personnel who are responsible, both directly and indirectly, for obtaining and evaluating data obtained from soil borings.

Procedures employed, site-specific conditions, and other pertinent observations that may affect the evaluation and interpretation of data collected will be recorded by the on-site geologist or geotechnical engineer on the boring log, and/or in the field log book, as appropriate. The data sheets will be maintained in the project file.

2.6.1 Field Documentation

Field activities will be documented in a bound logbook for each team as outlined in SOP 01 *Field Records and Documentation*.

2.6.2 Boring Logs

The geologist will log the subsurface conditions encountered in the boring and record the information on the boring log and in the logbook. Additional pertinent information will be recorded on the boring log including the following information:

- Boring identification
- Coordinates/elevation
- Drilling method
- Drilling date(s) including start and completion times
- Weather conditions
- Geologist name
- Driller's name/Drilling subcontractor/Type of drill rig
- Diameter of outer and inner sonic drill casings

- Types of drilling fluids and depths at which they were used
- Penetration length
- Penetration rate
- Soil recovery per penetration
- Visual description of soil consistent with the Uniform Soils Classification System (USCS)
- Total depth of boring
- Final disposition of boring

2.6.3 Soil Description

The USCS will be used for soil identification. The USCS provides useful information about soil gradation and plasticity. However, critical information necessary for site interpretation and evaluation are not included in the USCS. The USCS should therefore be supplemented with additional information.

The descriptive format begins with the USCS group name and symbol, which is discussed in more detail below. A detailed description, based upon ASTM standards, follows the USCS classification. For consistency, the primary descriptive elements listed below should be included in all soil descriptions, presented in the following standardized order:

- USCS group name and symbol (underlined and capitalized)
- Color (observable and Munsell Chart designation)
- Plasticity (non-plastic, slightly plastic, or plastic)
- Moisture condition (dry, damp, moist, or wet)
- Consistency (very soft, soft, medium stiff, very stiff, or hard for cohesive soils; loose, compact, or dense for non-cohesive soils)
- Gradation (relative percentages of all soil components: 40-50% = numerous, 30%-40% = many, 20%-30% = few, 10%-20% = scattered, 0%-10% = occasional; maximum size; weathering)
- Other descriptors (roots, lenses, seams, etc.)

A description of other pertinent properties should be included, as needed, following the primary descriptive elements listed above.

Following the detailed soil description, the probable geologic origin should be provided (in capital letters as shown). Several typical descriptions are presented below:

<u>POORLY GRADED SAND (SP)</u>
Yellowish brown (10YR 5/4), non-plastic, dry, loose, mostly fine sand with occasional medium sand.

<u>LEAN CLAY (CL)</u>
Olive brown (2.5 YR 4/3), sli-plastic, moist, stiff, occasional rock fragments, rooted.

<u>POORLY GRADED SAND WITH GRAVEL (SP)</u>
Pale yellow (2.5 YR 7/3), non-plastic, wet, compact to dense, subrounded, scattered gravel up to 0.5-inch.

Soil/sediment descriptions should be as comprehensive as possible, without excessive emphasis on insignificant details. Good judgment and common sense based on an understanding of geology, engineering behavior of soils, and project requirements are required.

2.7 Drilling Procedures

Drilling activities will be completed in accordance with the planned activities presented in the project UFP-QAPP Work Plan and the following requirements.

- All borings will be advanced using DPT drilling methods in a manner that conforms to Shelby County rules and regulations, Rules of Tennessee Department of Environment and Conservation (TDEC), Division of Water Supply, Chapter 12-4-10.
- All drilling equipment will be decontaminated prior to drilling activities in accordance with SOP 04 *Equipment Decontamination*.
- All necessary precautions will be taken to prevent leakage of hydraulic fluids of other contaminants into the borehole or onto equipment that is placed in the borehole
- Soil cores will be collected continuously in 4 to 5 foot intervals, depending on the specific tooling design. The soil cores will be collected in new and clean acetate liners for each interval.
- During drilling activities, the driller will notify the Trinity geologist of any changes in drilling conditions which will be noted on the boring log.
- Any investigation derived waste (IDW) (i.e., excess soil cores, decontamination water) will be disposed as specified in the Project Work Plan and SOP 05 *Investigation Derived Waste Management*.

2.8 Soil Sampling

Soil sampling for contaminant of concern analysis will be performed for laboratory analysis of volatile organic compounds (VOCs) as part this investigation in the following methods:

- Upon removal of the tooling from the boring, the acetate liner will be removed from the core barrel and opened for visual logging by the Trinity geologist.
- The Trinity geologist will collect a representative specimen from each soil core corresponding to intervals of interest identified during the MIP investigation. Samples will be collected utilizing Terra Core® kits for analysis of VOCs. The remainder of the soil containerized in accordance with SOP 05 *Investigation Derived Waste Management*.

2.9 Boring Abandonment

Upon completion of MIP and DPT soil borings, they will be abandoned by grouting to the surface with a neat Portland cement grout with 5% bentonite from the bottom up using a tremie pipe. Additional grout will be added as necessary to fill the boring if settling occurs.

2.10 Closeout Activities

Prior to departure at the end of each day's drilling activities, the following activities will be performed:

- All equipment shall be decontaminated and checked for damage
- All debris and trash shall be collected and removed from the site
- The FTL will complete all logbook entries and provide to Trinity Project Manager for review
- The crew will ensure the site is secured for the night and/or weekend

2.11 References

Geoprobe, 2015. *Geoprobe® Membrane Interface Probe (MIP) Standard Operating Procedure Technical Bulletin No. MK3010*. January.

Trinity, 2016. *Site Safety and Health Plan*, Final. July.

USEPA, 2013. *Design and Installation of Monitoring Wells*, SESDGUID-101-R1. January.

USEPA, 2014. *Soil Sampling*, SESDPROC-300-R3. August.

USEPA, 2015, *Field Equipment Cleaning and Decontamination* SESDPROC-205-R3. December.

3.0 SOP 03 – FIELD SCREENING TECHNIQUES 3-1
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3.3 Volatile Organic Vapor Screening 3-1
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3.0 SOP 03 – FIELD SCREENING TECHNIQUES

This Standard Operating Procedure (SOP) describes the methods and procedural guidelines for conducting preliminary field screening of geochemical site conditions. The data provided by these methods are not considered to be of quality sufficient to meet regulatory requirements regarding conceptual site modeling, site characterization, and/or closure; but are to be used for gross delineation, and extent of contamination, in order to make accurate real-time field decisions and gain efficiency in field assessment and remediation activities.

3.1 Overview

Feld screening with the Membrane Interface Probe (MIP) is critical to the successful completion of this task as the advancement of additional MIP borings and placement of subsequent direct push technology (DPT) soil borings will be based on the MIP results. Additionally, any significant findings of elevated field screening measurements will be used as part of a larger evaluation of site conditions.

3.2 Health and Safety

Specific tasks and general safety requirements are addressed in the *Site Safety and Health Plan* (SSHP), Environmental Restoration Support, Defense Depot Memphis, Tennessee, Final (Trinity, 2016). Each individual supporting the MIP survey and associated activities is required to have read and understood the SSHP. Personal protective equipment (PPE) will be worn at all times during field screening activities and, at a minimum will consist of hard hats, steel toed shoes, safety glasses, hearing protection, and high visibility clothing.

3.3 Volatile Organic Vapor Screening

The objective of this section is to describe procedures to be employed when screening of volatile organic vapors. Volatile organic vapor screening may be conducted for several reasons, including collecting headspace readings for grossly delineating soil contamination and monitoring the breathing zone air quality within the work area.

3.3.1 Membrane Interface Probe Soil Gas Screening

As described in the UFP-QAPP Work Plan, a DPT drill rig will be used to advance MIP sensors into the subsurface to evaluate lithologic characteristics and relative contaminant concentrations. A MIP is a semi-quantitative field screening device that can detect VOCs in soils and sediment. Generally, the technique can only detect total VOCs; it cannot provide specificity of analytes. Logging with the MIP can produce a real-time vertical log of VOCs with depth. Commonly, the MIP probe also includes a tip that measures the soil or water conductivity. This information can be used to correlate contamination to soil stratigraphy.

The MIP tooling includes a photoionization detector (PID) used for benzene, toluene, ethylbenzene, and xylene (BTEX), flame ionization detector (FID) for other petroleum hydrocarbons (straight and branch chained alkanes), and electron capture device (ECD) for chlorinated volatile organic compounds (VOCs). The detection limits for these devices range from 2.5 parts per billion (ppb) for ECDs to about 1 part per million (ppm) for the PID and FID.

After establishing and documenting a datum point for depth measurements, the MIP will be pushed at a constant rate of about one minute per foot. However, the push constancy may depend on the soil characteristics and the VOCs of interest. For example, the constant rate can be applied to loose sands with benzene as the target analyte.

Details on the collection and analysis of the data is provided in the SOP for *Geoprobe® Membrane Interface Probe (MIP), Technical Bulletin No. MK3010* (Geoprobe, 2015).

3.3.2 Breathing Zone Monitoring

The monitoring of the breathing zone air quality during site activities requires discernment and skill. It is important to recognize that this monitoring is for health and safety purposes only and is not used to help characterize the site contamination, as is the case with the headspace screening described in Section 4.3.1. Therefore, the specific requirements for the breathing zone monitoring should be captured in the SSHP (Trinity, 2016).

Commonly this activity is performed by holding the PID tip at the approximate height of mouth and nostrils of workers and monitoring the readings accordingly. When working around heavy equipment, such as a drill rig, this activity itself can represent a potential safety hazard. Therefore, care should be taken to let workers know when monitoring personnel are coming in to take a breathing zone reading.

3.4 References

Geoprobe, 2015. *Geoprobe® Membrane Interface Probe (MIP) Standard Operating Procedure Technical Bulletin No. MK3010*. January.

Trinity, 2016. *Site Safety and Health Plan*, Final. July.

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4.0 SOP 04 – EQUIPMENT DECONTAMINATION

This Standard Operating Procedure (SOP) describes methods to be used in the decontamination of equipment used in the Membrane Interface Probe (MIP) Survey at the Defense Depot Memphis, Tennessee (DDMT). Important documents used to develop this SOP are U.S. Environmental Protection Agency (USEPA) Science and Ecosystem Support Division (SESD) Procedure SESDPROC-205-R3, *Field Equipment Cleaning and Decontamination* (USEPA, 2015).

4.1 Health and Safety

Specific tasks and general safety requirements are addressed in the *Site Safety and Health Plan* (SSHP), Environmental Restoration Support, Defense Depot Memphis, Tennessee, Final (Trinity, 2016). Each individual supporting the MIP/direct push technology (DPT) and associated activities is required to have read and understood the SSHP. Personal protective equipment (PPE) will be worn at all times during decontamination activities and, at a minimum will consist of hard hats, steel toed shoes, safety glasses, hearing protection, and high visibility clothing.

4.2 Personnel Qualifications and Responsibilities

MIP tooling and DPT soil sampling equipment decontamination will be directed by the Field Team Leader (FTL) and performed by a member of the drilling team.

4.3 Equipment and Materials List

The required equipment and supplies to complete decontamination activities will consist of a high pressure steam cleaner, potable water, and a decontamination area.

4.4 Start-Up Activities

After arrival on-site, but prior to beginning MIP survey/soil sampling activities, the FTL will confirm that decontamination equipment and supplies are on-site and in working order.

4.5 Decontamination

4.5.1 Decontamination Area

The location of the decontamination area will be determined in consultation with the drilling subcontractor personnel. The decontamination area 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 and racked steel rods.

4.5.2 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. For smaller amounts, the shop water hose can be used.

4.5.3 Decontamination Procedures

The required decontamination procedure for large pieces of equipment such as drill rigs and drilling rods/core barrels is:

1. Wash the external surface of the equipment 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

4.5.4 Equipment Rinsate Collection

No equipment rinsate samples will be collected as part of the AS well installation activities.

4.6 Closeout Activities

Prior to departure at the end of each day's activities, the following activities will be performed:

- Confirm all equipment is decontaminated and properly store all equipment
- Add decontamination rinse water to the wastewater storage tank
- All debris and trash shall be collected and removed from the site
- The FTL will complete all logbook entries and provide to Trinity Project Manager for review

4.7 References

Trinity, 2016. *Site Safety and Health Plan*, Final. July.

USEPA, 2015. *Field Equipment Cleaning and Decontamination*, SESDPROC-205-R3. December.

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5.0 SOP 05 – INVESTIGATION DERIVED WASTE MANAGEMENT

This Standard Operating Procedure (SOP) describes methods to be used in the handling, management, and disposal of investigation derived waste (IDW) generated during the Membrane Interface Probe (MIP) survey and direct push technology (DPT) soil sampling activities at the Defense Depot Memphis, Tennessee (DDMT). Important documents used to develop this SOP are U.S. Environmental Protection Agency (USEPA) Science and Ecosystem Support Division (SESD) Procedure SESDPROC-202-R3, *Management of Investigation Derived Waste* (USEPA, 2014).

5.1 Health and Safety

Specific tasks and general safety requirements are addressed in the *Site Safety and Health Plan* (SSHP), Environmental Restoration Support, Defense Depot Memphis, Tennessee, Final (Trinity, 2016). Each individual supporting the Dunn Field MIP survey/DPT soil sampling and associated activities is required to have read and understood the SSHP. Personal protective equipment (PPE) will be worn at all times during decontamination activities and, at a minimum will consist of hard hats, steel toed shoes, safety glasses, hearing protection, and high visibility clothing.

5.2 Personnel Qualifications and Responsibilities

MIP survey/DPT soil sampling IDW management will be directed by the Field Team Leader (FTL) and performed by a member of the drilling team.

5.3 Equipment and Materials List

The required equipment and supplies to complete IDW management activities will consist of plastic sheeting, drums, or other bulk containers.

5.4 Start-Up Activities

After arrival on-site, but prior to beginning MIP survey/DPT soil sampling activities, the FTL will confirm that an IDW staging area has been identified and that all containers are in acceptable condition.

5.5 Types of Investigation Derived Waste

IDW generated during MIP/DPT activities will consist of the following:

- Soil cuttings
- Decontamination water

5.6 Investigation Derived Waste Handling

Waste generated during MIP/DPT activities will be classified as either non-investigative waste or IDW. Non-investigative waste such as packaging materials, PPE, acetate soil core liners, and other inert refuse will be collected and placed in a dumpster for disposal as municipal waste. The IDW will consist of decontamination water and excess soil cuttings from the AS well install activities. Decontamination water will be stored in 55-gallon drums or polyethylene totes and excess soil cuttings will be stockpiled on plastic sheeting at designated locations within the Dunn Field fenced boundary. Each medium will be sampled for waste characterization to determine final disposition.

5.7 Investigation Derived Waste Characterization

Composite samples from both the soil and decontamination waste streams will be collected for waste characterization analysis to determine final disposition. Analysis will consist of standard (USEPA Method 8260C) and Toxicity Characteristic Leaching Procedure (TCLP) for volatile organic compounds (VOC).

5.8 Investigation Derived Waste Disposal

Based on the analytical results, a final determination for the disposal of the IDW will be made. If soil results are below remediation goals set forth in the Dunn Field Record of Decision (CH2M Hill, 2004), the soil will be spread on the ground at Dunn Field. If soil VOC concentrations are above remediation goals, off-site disposal will be arranged. Containerized decontamination water will be disposed off-site disposal after receipt and review of waste characterization profile.

5.9 Closeout Activities

Prior to departure at the end of each day's activities, the following activities will be performed:

- Confirm all IDW is properly stored and secured
- Confirm all non-investigative waste is packaged and removed from the site
- The FTL will complete all logbook entries and provide to Trinity Project Manager for review

5.10 References

CH2M Hill, 2004. *Dunn Field Record of Decision*. March.

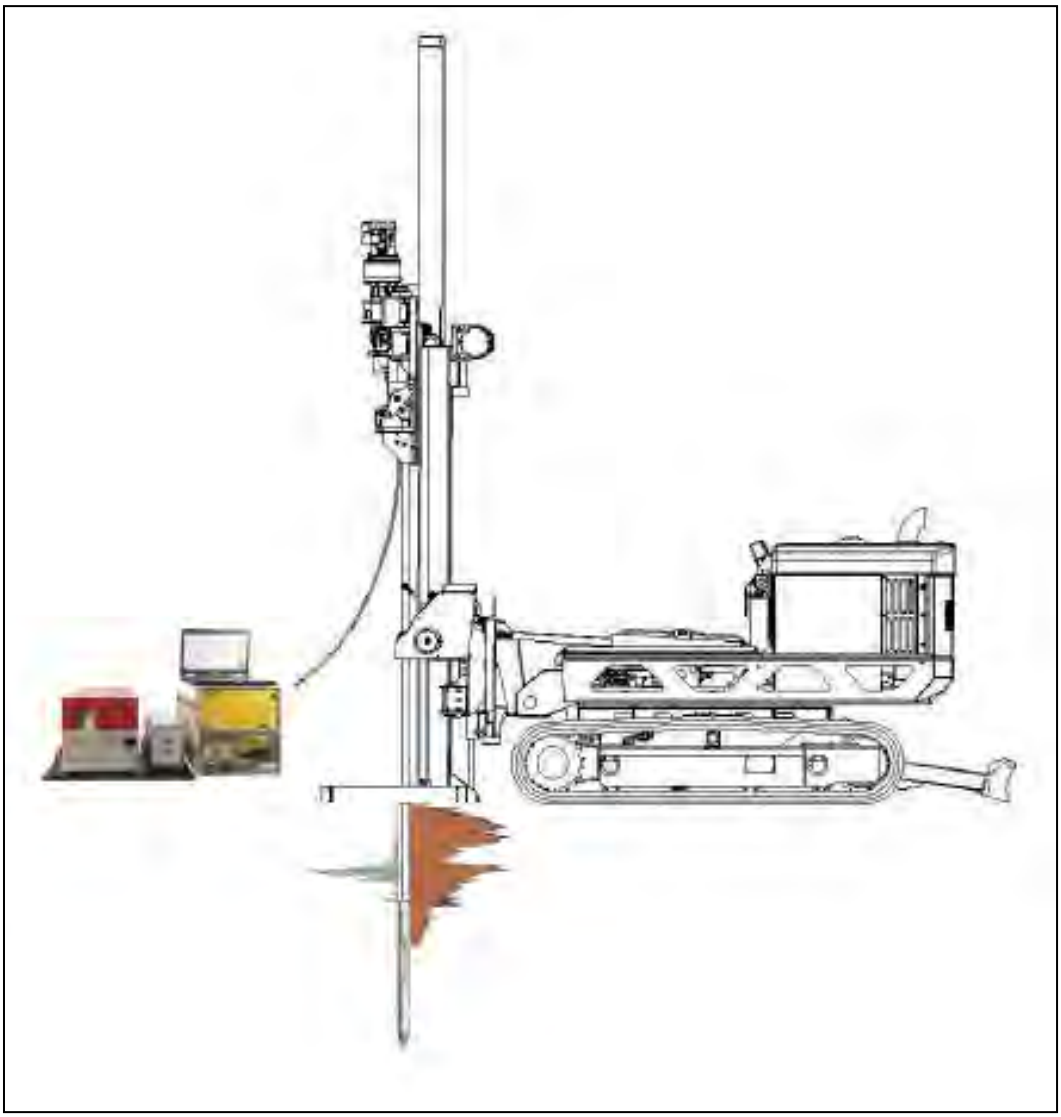
Trinity, 2016. *Site Safety and Health Plan*, Final. July.

USEPA, 2014. *Management of Investigation Derived Waste*, SESDPROC-202-R3. July.

Geoprobe® Membrane Interface Probe (MIP)

Standard Operating Procedure

Technical Bulletin No. MK3010
PREPARED: May, 2003
REVISED: January, 2015



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1.0 Objective

This document serves as the standard operating procedure for use of the Geoprobe Systems® Membrane Interface Probe (MIP) used to detect volatile organic compounds (VOCs) at depth in the subsurface.

2.0 Background

2.1 Definitions

Geoprobe®: A brand name of high quality, hydraulically-powered machines that utilize both static force and percussion to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform soil core and soil gas sampling, groundwater sampling and testing, soil conductivity and contaminant logging, grouting, and materials injection.
**Geoprobe® is a registered trademark of Kejr, Inc., Salina, Kansas.*

Membrane Interface Probe (MIP): A system manufactured by Geoprobe Systems® for the detection and measurement of volatile organic compounds (VOCs) in the subsurface. A heated probe carrying a permeable membrane is advanced to depth in the soil. VOCs in the subsurface cross the membrane, enter into a carrier gas stream, and are swept to gas phase detectors at ground surface for measurement.

2.2 Discussion

The MIP is an interface between contaminates in the soil and the detectors at ground surface. It is a mapping tool used to find the depth at which the contamination is located, but is not used to determine concentration of the compound. Two advantages of using the MIP are that it detects contamination in situ and can be used in all types of soil conditions.

The MIP is a logging tool used to make continuous measurements of VOCs in soil. Volatile compounds outside the probe diffuse across a membrane and are swept from the probe to a gas phase detector at ground surface. A log is made of detector response versus probe depth. In order to speed diffusion, the probe membrane is heated to approximately 121°C. (Refer to Figure 2.1).

Along with the detection of VOCs in the soil, the MIP also measures the electrical conductivity of the soil to give a probable lithology of the subsurface. This is accomplished by using a dipole measurement arrangement at the end of the MIP probe so that both conductivity and detector readings may be taken simultaneously. A simultaneous log of soil electrical conductivity is recorded with the detector response.

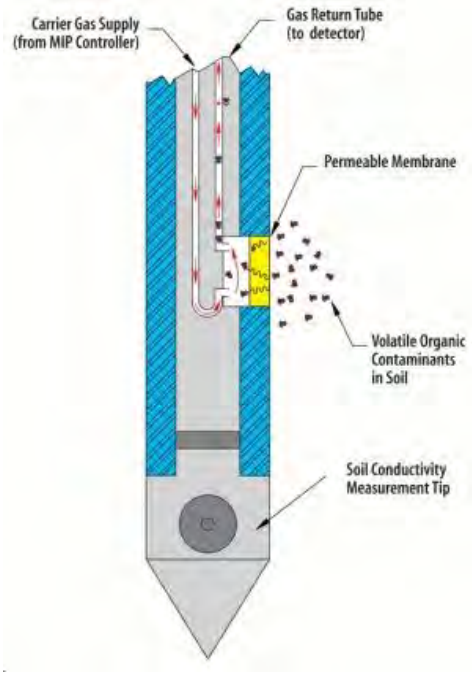

Figure 2.1: Diffusion across the membrane

Interpretation of electrical conductivity (EC) logs comes with field experience. It is very important that soil core samples are taken to confirm lithologic changes as each EC log is unique per site. As a generalization, a high conductivity reading indicates a smaller grain size and a low conductivity reading indicates a larger grain size (See Fig. 2.2).

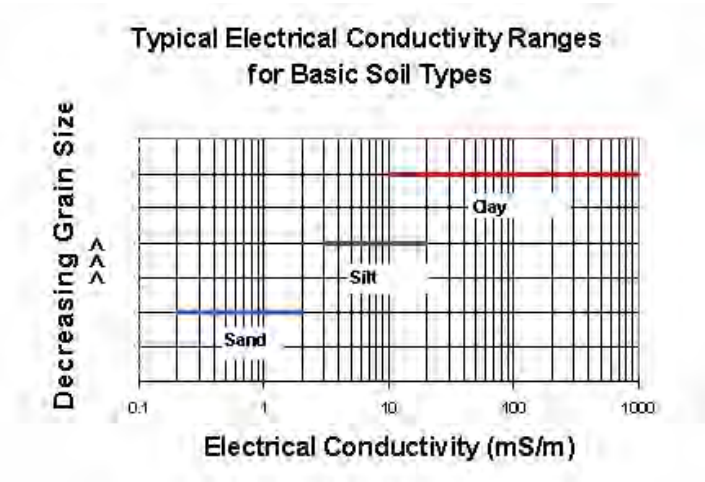

Figure 2.2: Generalized Electrical Conductivity Readings

3.0 MIP/EC Interferences

3.1 Detector saturation may require a short period of time for the detector to return to baseline after a log has been performed in higher concentrations. The MIP system can be used in free product environments with the operator monitoring and making the necessary adjustments to the detector and software gain/attenuation settings to account for the higher voltage readouts.

3.2 The MIP system can be operated in a wide range of contaminant concentrations from low dissolved phase to free phase materials. During a log and the removal of the tool string, contaminants can absorb onto the surface of the membrane and trunkline material causing elevated detector baseline signals. It is very important that the probe and trunkline system is clean enough to see the low concentrations typically used in the chemical response test. Not adequately decontaminating the probe prior to performing a response test can elevate the concentration of the standard causing an inaccurate high response to the specific concentration of standard that was prepared for the test.

3.3 Electrical conductivity can provide false positives or higher than expected readings when the soil is impacted by ionic plumes (chloride, nitrate) originating from, but not limited to: agriculture practices, seawater, salt storage, mining practices. Encountering metallic objects in the subsurface can also result in high EC readings.

3.4 Some silt and clay soils will not have the typical ionic composition that an operator may be used to for similar soils. This can result in lower than expected readings and perhaps cause misidentification of the associated soil zone based on typical response of a courser grain material. This can occasionally be found in clays that have had the minerals leached out or in intermixed silt-sand zones.

4.0 Tools and Equipment

The following equipment is needed to perform and record MIP logs. Basic MIP system components are listed in section 4.1 and shown in Figure 4.1. Additional equipment needed to run MiHPT logs is listed in section 4.2 with optional equipment listed in section 4.3. Refer to Appendix V for a detailed illustration of the GC1000 setup configuration. Appendix VI shows the common MIP probe tool string diagrams. There may be more required tools as determined by your specific model of Geoprobe® direct push machine.

4.1 Basic MIP System Components

Description	Quantity	Material Number
Field Instrument, 120V (Model FI6000)	-1-	213940
Field Instrument, 220V (Model FI6003)	*	213941
MIP Acquisition Software	-1-	214128
MIP Controller, 120V (Model MP6505)	-1-	214137
MIP Controller, 220V (Model MP6507)	*	214139
Gas Chromatograph, 120V with PID, FID and XSD	-1-	213946
Gas Chromatograph, 220V with PID, FID and XSD	*	213947
MIP Probe, 1.75 inch	-2-	214143
MIP/HPT Connection Tube	-2-	220912
MIP/HPT Drive Head 1.5in. rods	-2-	203794
Slotted 1.5” Drive Cap, Threadless	-2-	206609
MIP Probe, 2.25 inch	**	214152
2.25 Connection Tube	**	219455
2.25 Inch Water Seal Drive Head	**	212089
2.75 Inch Water Seal Drive Head	**	209796
Slotted 2.25” Drive Cap	**	211798
MIP Trunkline 100 ft.	(optional)	204077
MIP Trunkline 150 ft.	-2-	202570
MIP Trunkline 200 ft.	(optional)	204655
Agilent ADM 1000 Digital Flow Meter	-1-	600227
Hydrogen Gas Regulator	-1-	600137
Nitrogen Gas Regulator	-1-	600175
Vertical Gas Bottle Rack	-1-	214121
MIP Service Kit	-1-	214142
EC Bypass Cable	-1-	204025
Stringpot, 100-inch	-1-	214227
Stringpot Ground Plate	-1-	220775
Stringpot Cordset, 65-feet (19.8 m)	-1-	202884
Stringpot Mounting Bracket (6600/7700)	(optional)	202947
Stringpot Mounting Bracket (78 Series)	-1-	209511

Stringpot Foot Bracket (6600/7700)	(optional)	201816
Stringpot Foot Bracket (78 Series)	-1-	209533
Stringpot Piston Weight	-1-	214225
Drive Cushion (GH60)	-1-	204278
Rod Wiper, 1.25/1.5” Rods	-1-	600341
Rod Wiper Weldment	-1-	204387

4.2 Additional MiHPT System Components

Description	Quantity	Material Number
HPT Flow Module, 120V (Model K6300)	-1-	214091
HPT Flow Module, 220V (Model K6303)	*	214093
HPT Reference Tube 1.75 in HPT Probe	-1-	212689
HPT Reference Tube 2.25 in HPT Probe	**	211762
MiHPT Probe, 1.75 inch	-2-	219228
MiHPT Probe, 2.25 inch	**	214117
MiHPT Connection Tube	-2-	219594
MiHPT Drive Head for 1.5” rods	-2-	220545
MiHPT Trunkline 100 ft	(optional)	214113
MiHPT Trunkline 150 ft	-2-	214114
MiHPT Trunkline 200 ft	(optional)	214115
Coupling 1/8 to 1/8 Tube	-5-	107963
Coupling 0.135 to 0.150 Tube	-5-	220978
Oetiker Band Clamp 4.7mm x 5.7mm	-10-	220977
Oetiker Band Clamp #7	-10-	220976
HPT Sensor Module	-2-	210091
Heated Trunkline Seal Asm.	-4-	211768
HPT Test Load	-1-	206552
HPT Service Kit	-1-	205599

4.3 Optional Accessories

Description	Quantity	Material Number
Heated Trunkline Controller, 120V (Model MP7000)	-1-	214147
Heated Trunkline Controller, 220V (Model MP7003)	*	214148
MIP Heated Trunkline 100 ft.	(optional)	214150
MIP Heated Trunkline 150 ft.	-1-	214151
MIP Heated Transfer Line 8 ft.	-1-	214149
MIP Breakout Connection Panel.	-1-	211100
Roll-out Rod Rack (30-1.5in rods)	-1-	213627
Water Transport System.	-1-	203450

*Use in place of 120V components if desired.

**Use in place of 1.75 inch probe and components if desired.

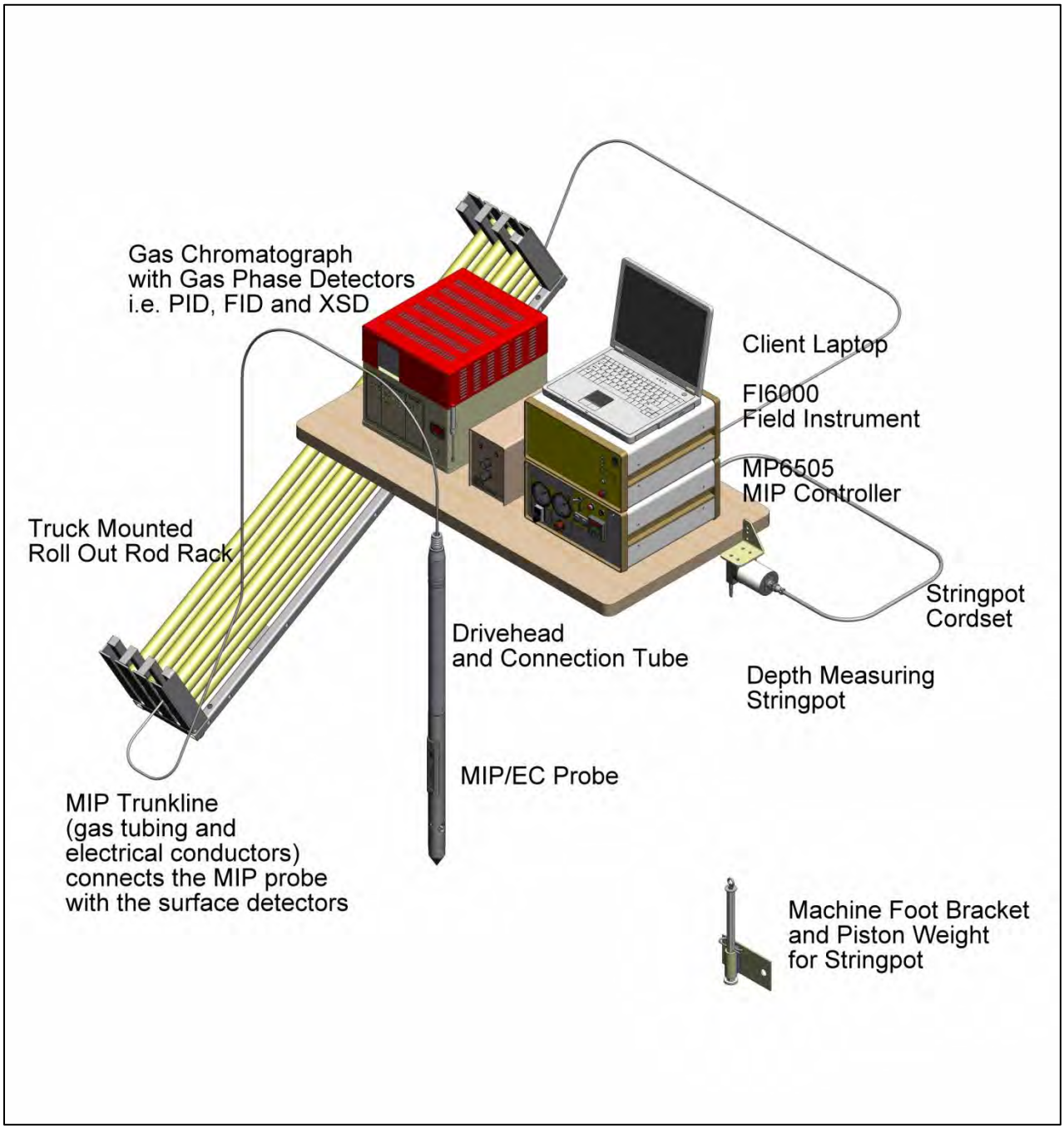

Figure 4.1: MIP System Components

5.0 Quality Assurance/Quality Control

Quality assurance (QA) testing of each of the sensors on the probe is too be performed before and after each log to validate that the equipment is capable of generating good data. The MIP tool includes chemical response tests (Section 5.1.3) which are performed to ensure that the probe membrane, trunkline and detectors are capable to providing ample signal over baseline noise to a known site contaminant at a given concentration. The electrical conductivity (EC) sensor is tested using an EC dipole test (Section 5.2) with low and high readings typical to EC values of the soil. The HPT sensor is included on the MiHPT probe and is tested using the HPT reference test (Section 5.3) which confirms the sensors ability to accurately measure a 6" column of water and provides an accurate measurement of atmospheric pressure.

Quality control (QC) is performed during and after each log is generated. Log QC will answer the following questions to ensure that the data is good and makes sense:

1. Does the log look correct? Does the elctrical conductivity appear to be in an acceptable range? Is there anything seen in the log that would make you suspect that the system wasn't working correctly, ie. a loss of temperature or gas pressure of the system.
2. Response consistency? As more logs are completed do they show general consistency of EC and contaminant response? Review a cross section of logs in the DI-Viewer (Appendix IV).
3. Repeatablity? Replicate logs may be run every 10 to 20 locations to verify repeatability.
4. Are my lithogy changes consistant with physical soil cores? Take continuous or discreet confirmation soil samples to confirm your lithogy changes in EC.
5. Do the MIP detector responses make sense for contaminant concentration. This must be verified by the collection of water or soil samples for lab analysis to confirm contaminants and their concentrations.

5.1 MIP Chemical Response Test:

Chemical response testing is an important quality assurance measure used to validate each log by proving that the integrity of the detector system is intact. With the chemical response test the operator introduces a working standard (known site contaminant of concern) at a known concentration to the membrane for a set time of 45 seconds which should match the residence or holding time at each sampling interval. Two acceptable methods of introducing the standard to the membrane are shown in Figures 5.4 and 5.5.

Typical site contaminant of concerns which are used in MIP chemical response tests include but are not limited to Benzene, Toluene, Trichloroethylene or Perchloroethylene. The stock standard should be made up from one of these or an appropriate mix of chemicals.

5.1.1 Preparation of the Stock Standard

Preparation of the stock standard is critical to the final outcome of the concentration to be placed into the testing cylinder. The following items are required for preparing the stock standard:

- Neat sample of the analyte of interest (i.e.: Benzene, Toluene, TCE, PCE, etc.) purchased from a chemical vendor
- Microliter syringes (recommended to have: 25μL, 100μL and a 500μL or 1,000μL syringes).
- 25-mL or 50-mL Graduated cylinder
- Several 40-mL VOC vials with labels
- 25mL Methanol

1. The total volume of methanol and the compound added should equal 25mL.
2. Pour methanol into graduated cylinder to the 23.5-24mL mark, the volume depends upon the compound density (Table 5.1).
3. Pour the methanol from the graduated cylinder into a 40mL VOC vial.
4. Add the appropriate volume of desired neat analyte into 40mL VOC vial containing methanol. The required volume of neat analyte for seven common compounds is listed in Column 3 of Table 5.2. The equation in table 5.1 shows how to calculate the appropriate neat analyte volume for other compounds of interest given the appropriate density.
5. Label the vial with the name of the stock standard (i.e. Benzene, Toluene, TCE, PCE), concentration (50mg/mL), date created, and created by (your name or initials).
6. Stock standards need to be kept cold in a refrigerator or freezer to ensure they can last up to one month otherwise they should be made up more frequently as often as every 3 days if there is not cooling during the summer months. The more volatile the compound the quicker it will lose its concentration.

Stock Standard Calculations
25mL (methanol) x 50mg/mL = 1250mg 1250mg x 1/density of analyte = amount of neat material to be placed with methanol to make up 25mL total volume *Example: Preparation of 50mg/mL Benzene standard.* *1250mg x 1/0.8765mg/μL = 1426μL* *Use 1426μL of neat Benzene in 23.5mL of Methanol to get a 50mg/mL stock standard.*

Table 5.1: Stock Standard Preparation Calculations

Compound	**Density (mg/μL)**	**Volume (μL) of Neat Standard Required to prepare Working Standard (0.5 L)**
Benzene	0.876	1426
Toluene	0.867	1442
Xylenes	0.860	1453
Methylene Chloride	1.335	936
Carbon Tetrachloride	1.594	784
Chloroform	1.480	845
Trichloroethylene	1.464	854
Perchloroethylene	1.623	770

Table 5.2: Density and required volumes of neat (pure ~100%) compounds used to make a 50mg/mL stock standard into 25 ml of methanol

5.1.2 Preparation of Working Standards

The following items are required to perform response testing:

- Microliter syringes (recommended to have: 10, 25, 100 & 500µL syringes).
- Freshly made 50ml/ml Stock standard
- Testing cylinder made from a nominal 2-in. PVC pipe with a length of 24 in. or 40ml vial
- 0.5 L plastic beaker or pitcher
- Supply of fresh water, 500mL needed per test
- Stopwatch

Volume of 50mg/ml Stock Standard (µL)		Final Concentration mg/L = ppm
10		1.0
100		10
1000		100

Table 5.3: Volumes of stock standard and final concentrations

Figure 5.1: Working standard

5.1.3 Performing the Chemical Response Test

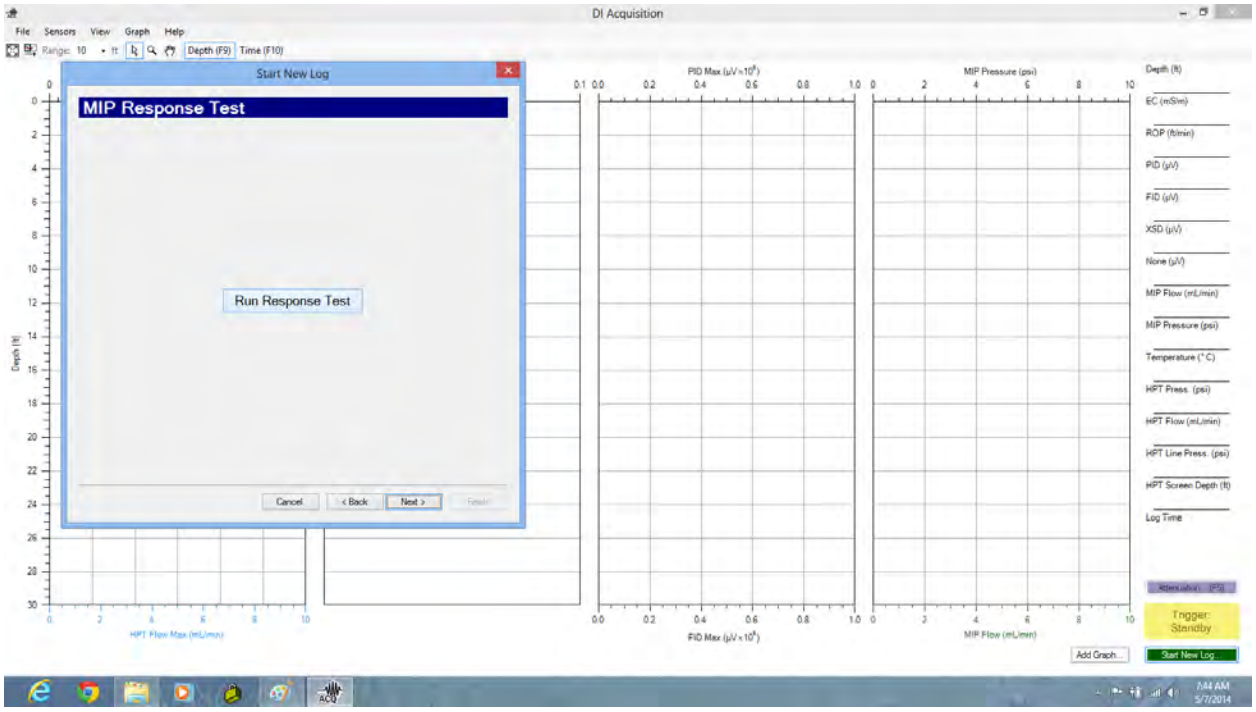

Figure 5.2: DI Acquisition Response Test Screen

1. Begin a new log in the DI-Acquisition software and proceed to the response test screen. The detector signals should be stable before proceeding with the test.
2. Measure out 500mL of tap or distilled water in a graduated beaker.
3. Using Table 5.3, determine the desired volume of stock standard to place into the 500ml measured volume of water to make up the working standard.
4. If the detector baselines have been monitored while the standard was being prepared select “Clear Response Test”.
5. When ready with the working standard prepare to run the response test by exposing the membrane to the working standard. Two acceptable methods are to pour the standard into a nominal 2-inch x 24-inch PVC pipe and insert the probe into the tube (Fig. 5.4) and the other method is to pour the working standard into a 40ml/vial and invert onto the membrane (Fig. 5.5).
6. Start the response test by clicking on the “Run Response Test” button (Fig. 5.2) and immediately expose the MIP membrane to the test solution (Figures 5.4 or 5.5).

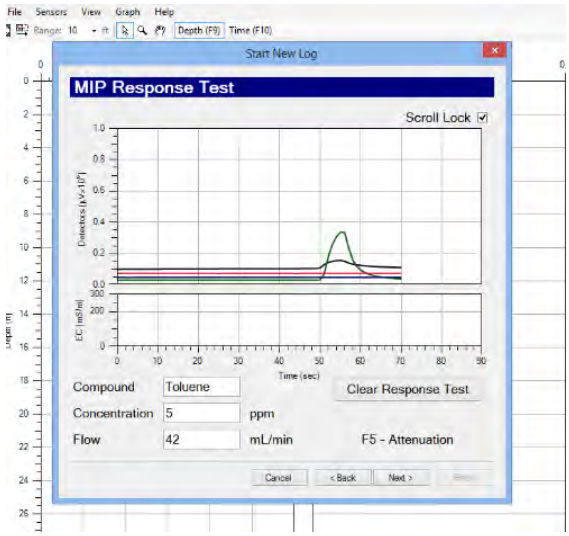

Figure 5.3: Running the Chemical Response Test

7. Leave the membrane exposed to the test solution for 45 seconds. This time is to be equal to the resonance time at each depth interval during probe advancement.
8. Starting the response test time file as the membrane is exposed to the test solution allows the trip time (Section 5.1.4) to be easily calculated by when response begins to climb which is approximately 50seconds in Fig. 5.3.
9. Fresh working standards need to be made for each test, they cannot be reused.
10. After the response has come through the detectors and adequate detector response is seen the operator may select “next” to move to the EC QA test.

Acceptable methods for performing the MIP Chemical Response Test

Figure 5.4 Probe immersed in steel or PVC pipe containing working standard

Figure 5.5: 40ml vial of working standard inverted onto membrane

5.1.4 Determination of Contaminant Trip Time:

Response testing also enables the operator to measure the chemical trip time which needs to be entered into the MIP software to accurately plot the contaminants depth position. The trip time is the time it takes for the contaminant to travel through the trunkline from the membrane to the detectors. The contaminant trip time is influenced primarily by trunkline length and carrier gas flow rates as well as the contaminant makeup specifically boiling point. The chemical response trip time can be determined from the results on the Pre-Log Response Test. Using Fig. 5.6 the Benzene trip time (TT) would be approximately 55 seconds. This response test would need to have been started right when the chemical used in the response test was exposed to the membrane. Additional typical response test graphs are located in Appendix I.

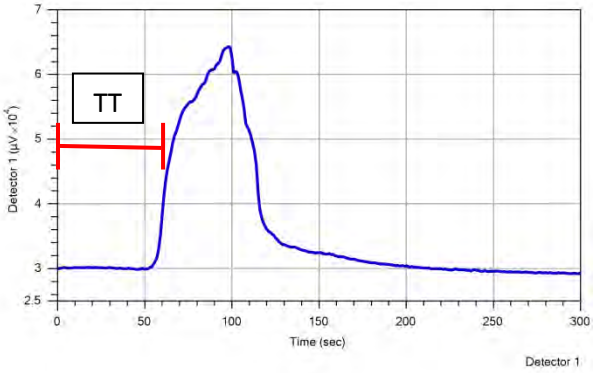

Figure 5.6: 5ppm Benzene on PID

5.1.5 Appropriate Chemical Response Test Concentrations and Response

The compound used in a chemical response test should be the site contaminant of concern or similar which will give you the most accurate response magnitude for that chemical as well as an accurate trip time. If the site objective is to delineate the extent of a dry cleaner plume then the operator should use PCE for the response tests at the lowest possible concentrations ~1ppm or less. If the site objective is to delineate the extent of the petroleum plume from a gas station then the operator should use one of the BTEX compounds or a gasoline mixture at or near the detection limit. If the site objective is to map out a plume source and high contaminant concentrations are expected then the response tests should be run at higher concentrations such as 10ppm-50ppm. This should reduce the possibility of trunkline/membrane carryover masking the chemical response tests.

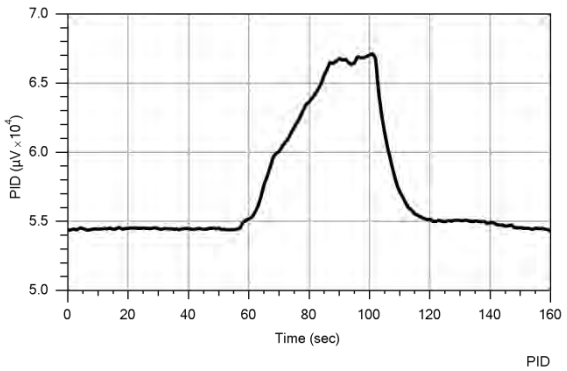

Figure 5.7: 2.5ppm Benzene on the PID

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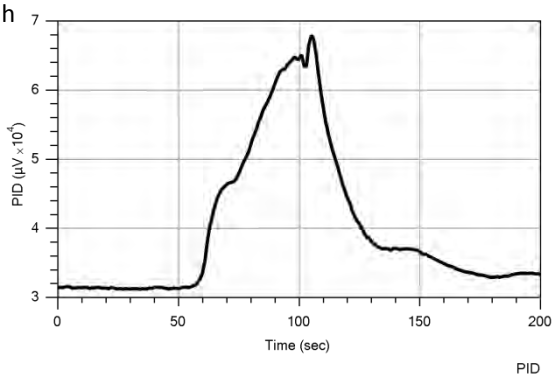


Figure 5.8: 5ppm TCE on the XSD

Figure 5.7 shows a benzene response over baseline on the PID of approximately 12,500μV on a 2.5ppm standard for a 5,000μV/1ppm. Figure 5.8 shows TCE responses over baseline on the XSD of 33,000μV on a 5ppm standard for approximately a 6,500μV/1ppm response.

5.1.6 Minimum Acceptable MIP Response Test Levels and Maintenance Tips

Geoprobe Systems specifies the following guidelines as minimum MIP response test values for performing MIP logging.

Detector systems can vary in the level of response for a given chemical concentration depending on detector age, model, and maintenance performed. However, it should be expected that the detector system would be able to provide at least a 5:1 signal to noise ratio (see Appendix I) for 1ppm of Benzene or TCE. Other compounds or concentration may be performed at the client requests however they may have different response magnitudes and signal to noise ratios at 1ppm. These specifications are required with operation of the PID and XSD (ECD or DELCD as well as alternative halogen detectors). The FID is a less sensitive detector typically used as a confirmation detector and one used for mapping natural gas components.

If the minimum response test levels are not achievable or throughout the day or project the detector sensitivity falls below these levels, the operator must stop and perform maintenance on the system to enhance the sensitivity of the detectors. Corrective actions could include:

- Changing MIP membrane (see section 9.0)
- Making a fresh chemical stock standard (see section 5.1.1). It does not take long for a volatile chemical standard to lose the original concentration.
- Decreasing trunkline carrier gas flow from 40ml/min to 30 or 20ml/min. This will lower the pressure in the trunkline and at the membrane which will increase system sensitivity. If this is corrective action is taken the operator must update the system trip time which has changed.
- Performing detector maintenance
    - Cleaning or replacing the PID bulb
    - Replacing the XSD probe assembly or reactor core
- Checking and adjusting detector gas flows - especially in the FID.
- Replacing the trunkline (an old trunkline can be a source of contaminant phase buildup. This will reduce detector sensitivity by causing contaminant dispersion in the trunkline which results in reduced response levels as well as delayed trip times).

It is wise for the MIP operator to monitor the detector response heights from the chemical response tests to evaluate membrane performance. With increased membrane footage, detector response will fall off indicating that it is time to change the membrane (see Appendix III). It may be possible to rejuvenate a MIP membrane by scrubbing with a wire brush.

5.2 EC Dipole Test

On the FI6000 and the DI-Acquisition software the EC dipole test screen (Fig. 5.10) will open up after the chemical response test is completed. When ready place the low (brass) side of the EC Dipole test jig (Fig. 5.9) between the EC dipole and body of the probe and start the low level test, hold for 5 sec until the system captures the data (Fig. 5.10). Repeat for the high (stainless steel) EC test. These tests should result in readings of 55mS/m and 290mS/m ± 10%.

Figure 5.9: EC Dipole Test Jig

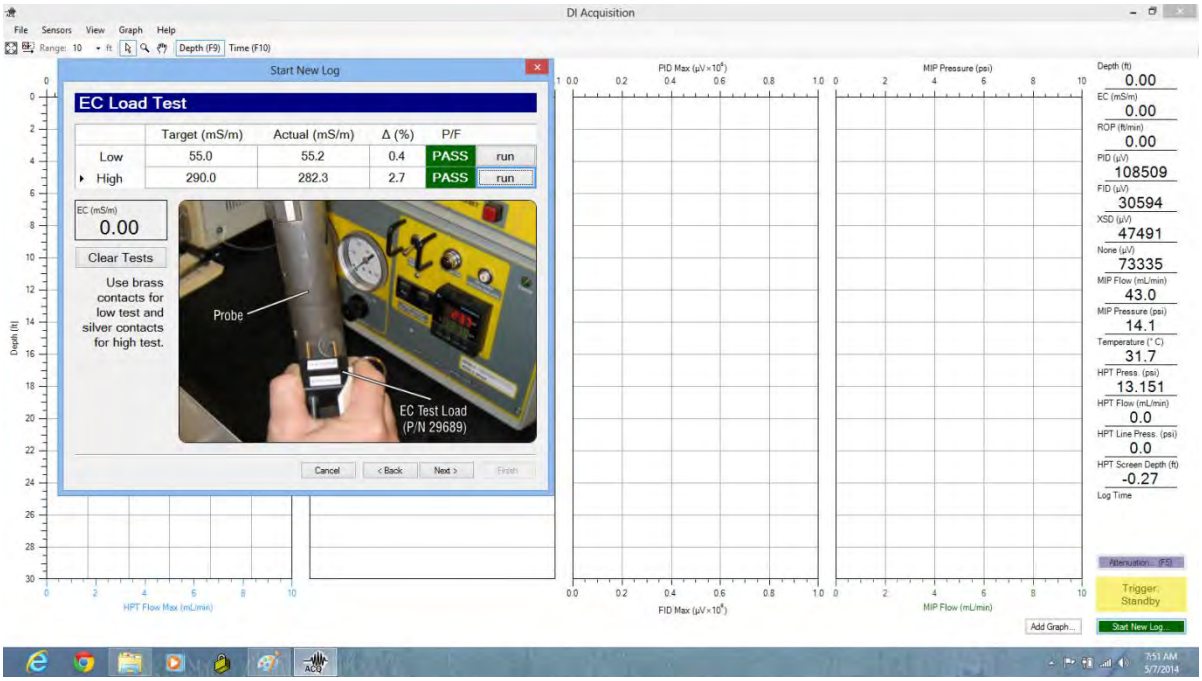


Figure 5.10: EC Dipole Test QA Screen

If the EC readings do not pass, the DI Acquisition (FI6000) software will prompt the user to proceed through a series of troubleshooting tests (Fig. 5.11). These tests will check the EC calibration (Fig. 5.12) to determine if the reason EC Test loads have failed was an issue internal to the FI6000 or if it is external in the trunkline-probe circuit. From here the operator should have an idea where to focus their attention to fix the problem.

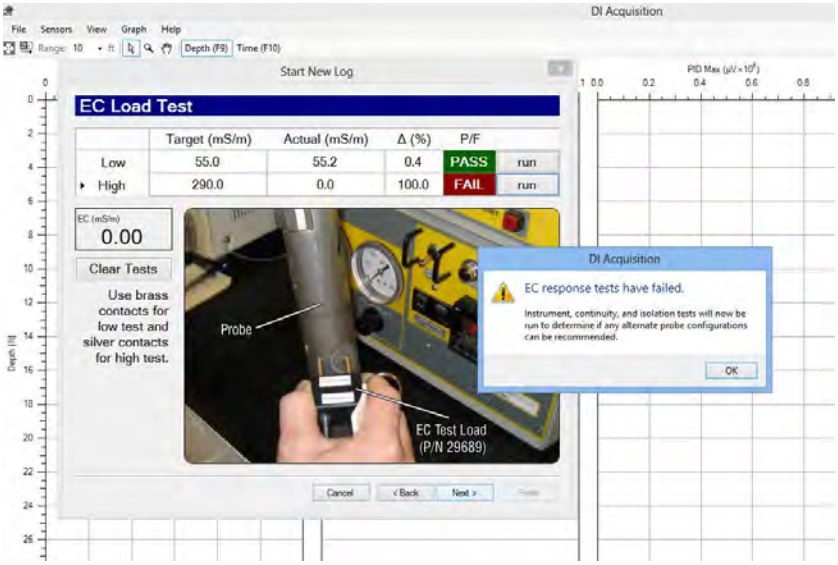


Figure 5.11: Failed EC Dipole Test Error Screen

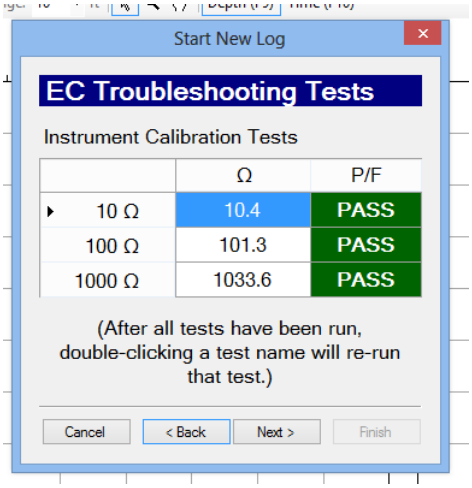


Figure 5.12: EC Calibration Check

5.3 HPT Reference Test – (MiHPT)

Reference testing is done to ensure that the HPT pressure sensor is in working order and to evaluate the condition of the HPT injection screen. The HPT reference test calculates atmospheric pressure which is required to obtain static water level readings and to determine the estimated K values for the log in our post log processing software the DI Viewer.

Reference Test Procedure

1. Connect a clean water source to the HPT controller and turn on the pump.
2. Allow water to flow through the system long enough so that no air remains in the trunkline or probe (air in the system can cause inaccurate flow and pressure measurements).
3. Insert the probe into the HPT reference tube and allow the water to flow out the valve adjusting the flow rate to between 200-300ml/min (Fig. 5.13). Ensure that the reference tube is close to vertical.
4. With a stable pressure reading and the water flowing out of the valve select “capture” - bottom with flow (Fig. 5.14)

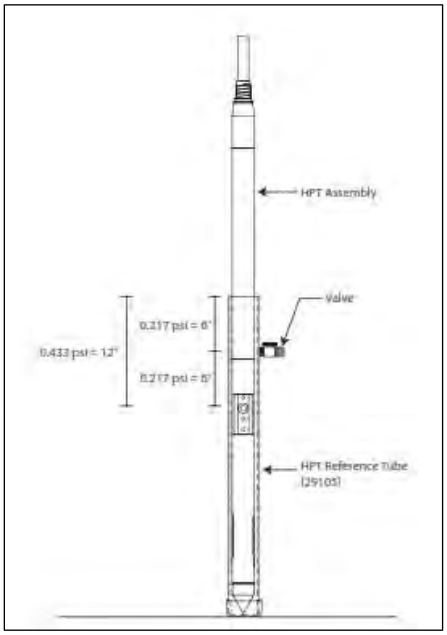


Figure 5.13: HPT Reference Test Setup

5. Close the valve and allow the water to overflow the top of the tube. When the pressure stabilizes select “capture” - top with flow.
6. Shut off the water flow. When the pressure stabilizes select “capture” - top flow = 0.
7. Open the valve and allow the water to drain out. When the pressure stabilizes select “capture” - bottom flow = 0.

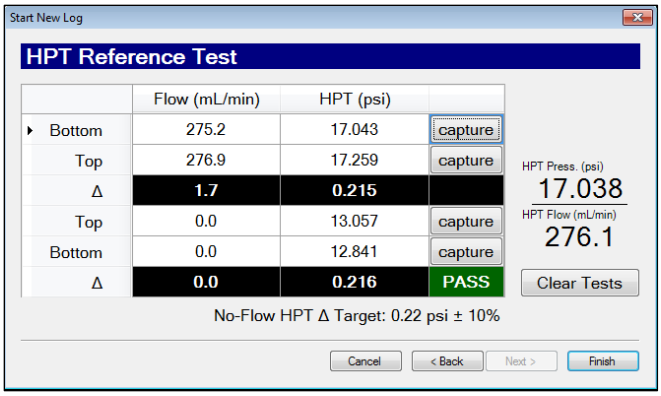


Figure 5.14: HPT Reference Test Screen

The HPT reference test reading flow = 0 is the true test of the condition of the pressure sensor and is the only sensor test to have a pass/fail reading on it. Ideally, the pressure difference between the top and bottom values will be 0.22 psi (1.52kPa). Typical pressure readings of the sensor will be in the 12PSI-15PSI (83kPa-104kPa) range.

6.0 Equipment Preparation for Site Work

The biggest issues to the performance of any specific MIP system is inadequate project preparation and system review, too heavy of a workload which reduces the ability to perform needed maintenance and inexperienced operators how do not fully understand the steps of troubleshooting.

When a MIP system is stored for a period of time between projects, operators must review the equipment and give it a full system checkup which includes checking detector gas flow rates, running response chemical response tests with known chemicals at concentrations at or near required site detection limits. This needs to be performed 1-2 weeks in advance of project work so there is time to obtain required supplies that might be needed for proper operation. A final checkout needs to be performed within 7 days of the project. If the MIP site contaminant of concern is an obscure chemical not normally tested for the operator should run some of that chemical for response tests to confirm it can be detected and to determine reasonable detection limits. The operator should be able to supply the consultant with pre-project performance data of all sensor information to be performed at the site which might include EC, MIP chemical response tests, and HPT reference test information.

If a MIP system is scheduled on a long job or has a number of jobs strung together it is in the best interest of the MIP service company to schedule a maintenance day at least every 3 weeks to allow the operator time to go through system and service the components that need attention. This will help to be able to keep to system performing well for the company and their clients. Pre project performance must still be able to be produced.

New operators will always be needed as the MIP community continues to grow, however it is imperative that operators who are running the MIP systems on their own have been properly trained by experts from their own company or at Geoprobe Systems® headquarters. An inadequately trained operator who faced difficulties onsite and does not understand the system and how to troubleshoot it will quickly bring frustration upon themselves, their company and clients. It is important that each operator is properly trained, is able to spend consistent time with the equipment and the software, and whenever possible operate of the equipment under the guidance of a mentor "MIP specialist."

7.0 MIP Field Operation

1. Power on the generator.
2. Open the gas cylinders that will be used for the MIP system (i.e. nitrogen, hydrogen, air, etc.).
3. Power on the GC and detectors and allow them to warm up (min. 20 minutes) to set temperature.
4. Power on the MIP controller, field instrument and laptop computer.
5. Check the trunkline supply and return flows of the system and MIP pressure. Compare these numbers to previous work.
6. Start the Acquisition software and start a new log.
7. Perform the chemical response test (Section 5.1.2) and record the height of the peak response and the trip time into a field notebook. Refer to Figure 5.4 and Appendix I and III.
8. Complete the EC dipole test (Section 5.3) and finish setting up the log.
9. Record the system parameters in a field notebook at this time (i.e. flow, pressure, trip time, detector baseline voltages).
10. Connect the stringpot cable to the stringpot and the stringpot wire to the weight located on the probe foot and pull keeper pin so the weight will drop to the ground.

NOTE: Do not allow the stringpot cable to snap back into the stringpot housing at a high rate of speed. This will ultimately damage the stringpot transducer.

11. Place the drive cushion onto the probing machine head.
12. Place a slotted drive cap to the MIP drive head.
13. Place the rod wiper on the ground and insert the point of the MIP probe into rod wiper opening.
14. Start the HPT water flow if running MiHPT.

    Note: It is important that there is always water flowing when the probe is moving to avoid soil particles from moving through and plugging up the screen.
15. Align the probe exactly straight and advance the probe to the starting depth: MIP membrane even with the ground surface.
16. Click the trigger button in the lower right hand corner of computer screen. (The Trigger label will flash and the background will change from yellow to green).
17. Standard advancement the probe is at a rate of 1ft/min meaning: advance 1 ft (30 cm) in 15 seconds and then hold at depth for 45 seconds, then advance to the next depth interval (1 foot) over 15 seconds and wait for 45 seconds. Do this until the predetermined log depth or until refusal is attained.

Advancement the MIP probe can be performed using a continuous push method with no stopping intervals which may be desirable in source level contamination. Data collected by this method will result in higher detection limits and is not directly comparable to data collected by the standard advancement method previously discussed.

NOTE: If the there is a loss in MIP pressure or temperature during the logging process, stop and evaluate the problem using the troubleshooting guide located in Appendix II.

NOTE: Refusal is attained when it takes longer than 1 minutes of continuous hammering to advance the probe one foot. This is the maximum time to reach one foot of probe travel.

18. Perform an HPT dissipation test (Section 7.1) in a zone of higher permeability indicated by lower HPT pressure if you are operating the MiHPT probe.
19. When the MIP log is complete, turn the trigger off and slowly return the stringpot cable into the stringpot housing.
20. Turn off the heater switch to the probe during tool string retraction so no as few contaminants as possible are diffused through the membrane and into the trunkline during retraction.
21. Raise the probe foot of the direct push machines foot assembly and place the rod wiper weldment under the foot assembly to keep it in place during rod retraction.
22. Pull the probe rod string using either the Geoprobe® rod grip pull system or a slotted pull cap.
23. When the MIP probe reaches the surface, clean the probe and membrane well with a detergent/water mix and rinse off well.
24. Now turn the probe heat back on to back off the membrane. Make sure the probe membrane and trunkline are clean of contaminants and the detector baselines are stable prior to running a post log response test. View the detector activity in the response test screen.
25. When the baselines are stable run a post log response test. These response test results should be written down in the field notes and compared to the initial test. This system check ensures the data for that log is valid.
26. When using the FI6000, the data will be saved into your designated folder on your laptop in a compact .zip file. Data from the MIP log can now be graphed and printed using the DI-Viewer software (Appendix IV).

7.1 Performing an HPT Dissipation Test

At least one dissipation test must be performed in order to calculate the static water level and estimated K readings from the HPT log. Dissipation tests need to be performed below the water table and are best in zones of high permeability where the injection pressure can dissipate off quickly once the flow is shut off. The following are the steps for running an HPT Dissipation test.

1. Stop in a zone of higher permeability which is indicated by lower HPT inject pressure.
2. Switch the DI Acquisition display view from the depth screen to the time screen by pressing the F10 key (F9 and F10 toggle between the depth and time screen of the acquisition software).

3. The screen will be grayed out which means that the data up to that point has not been saved. Select “Start Dissipation Test” which will turn the screen from gray to a white background indicating that you are now saving the time data.
   Now shut the pump switch off and when the line pressure reaches zero, turn the flow valve off.
4. The HPT Pressure will begin to drop (dissipate the hydrostatic increase) and allow it to stabilize so very little visible drop in pressure is seen. When the pressure has fully dissipated turn the flow valve and the pump switch back on. When the flow and pressure are reestablished select “End Dissipation test.”
5. Select F9 to return to the depth screen and advancing the tool into the ground.

Note: Performing a dissipation test in zones of higher permeability may only take 60 seconds or so but if the HPT pressure was higher to start with it may take a long time up to several hours to dissipate off to equilibrium. This is why targeting the most permeable zone to perform the dissipation tests is most desirable.

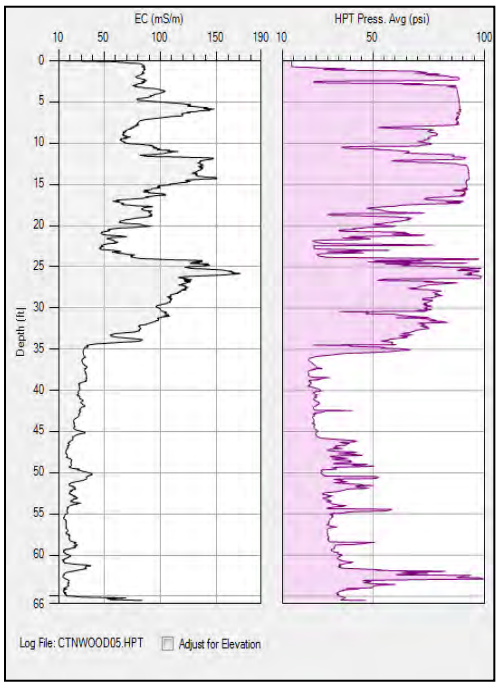


Figure 7.1: EC and HPT Pressure Graphs

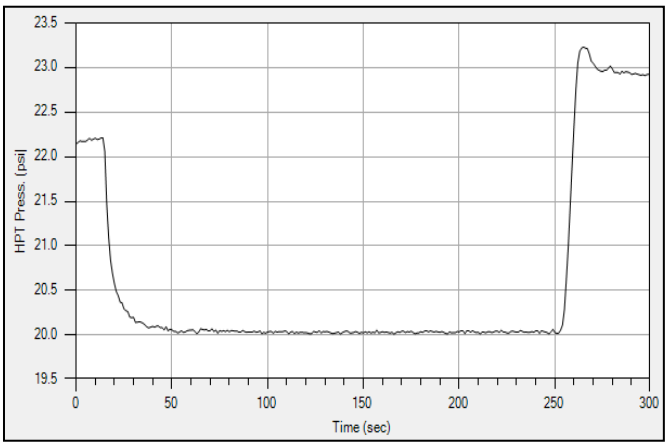


Figure 7.2: HPT Dissipation Test Screen.

The dissipation test shown in Figure 7.2 was performed in the lower pressure zones located at 39.5’ of the log shown in Figure 7.1. With HPT sands and gravels are indicated by lower injection pressure which is primarily seen below 35’ in the above log. The dissipation test in Figure 7.2 shows a higher pressure at the start of the test which falls off which is a result of shutting off the water flow. A good dissipation test will run for a period of time approximately 30-60seconds at a stabilized pressure and then turn the water flow back on during the saved log.

7.2 Detector Gain/Software Attenuation Changes

While mapping volatile contaminants with the MIP system operators commonly encounter highly contaminated/free product zones that can result in the detector signal climbing to the point of saturation or "flat lining." This occurs because the GC or detector system has a limited signal output range. What that range is varies depending upon the GC model or detector controller. Typical signal out limits for are 0-5VDC for SRI and Shimadzu GC models and 0-1VDC for HP/Agilent GCs and the OI XSD. The attenuation settings (software multipliers) for SRI and Shimadzu GCs and the XSD are based on a 10^x multiplication factor. The attenuation settings for detectors operated through an HP GC are based on a 2^x multiplication scale x = HP GC Range with the sum being the corresponding attenuation for the MIP software (Table 7.1).

As the probe is being advanced into higher concentration petroleum hydrocarbon soils the operator, if using an SRI GC, will want/need to adjust the GC gain on the PID and probably the FID from a gain setting of high to medium which takes those detector signals and divides them by a factor of 10 (Table 7.1). This reduction in the signal can be seen in the software both in the digital display as well as on the time graph. After the signal has been reduced the operator will need to select the attenuation tab/F5 in the DI Acquisition software and input a 10x multiplier in for the PID and the FID if both gain switches were changes to the medium setting.

If the operator chooses to go back to the highest sensitivity on those detectors after passing through the high contamination zone they need to first remove the multiplier in the software (F5) and then change the gain setting from medium to high on the GC – removing the signal divider. If either of these is performed in reverse fashion the log will see a very larger false positive peak because the signal is multiplied up without having a signal divider in place. The operator always wants to add the signal divider in first as they go into higher reading soils and remove it last as they come out of them.

Gain/Attenuation Settings on the GC detectors and the DI Acquisition software

HP GC* Range	DI Acq. Attenuation		SRI GC Gain	XSD Gain	DI Acq. Attenuation
0	1		High	High	1
1	2		Medium	Medium	10
2	4		Low	Low	100
3	8				

Table 7.1: GC gain/range settings and associated software multipliers.

*- The detectors on the HP GC can have attenuation settings up to a range of 7 on the GC corresponding to an acquisition software multiplication value of 128.

8.0 Replacing a Membrane on the MIP Probe

A probe membrane is considered in good working condition as long as two requirements are met:

1. Adequate signal response is achieved during the chemical response tests to see the required detection limits.
2. The difference between the supply and return flow has not increased by more than 3mL/min from the original settings. (A digital or bubble flow meter should be kept with the system at all times).

If either one of these requirements are not met, a new membrane must be installed as follows.

1. Turn the heater off and allow the block to cool to less than 50° C on the control panel readout.
2. Clean the entire heating block with water and a clean rag to remove any debris.
3. Dry the block completely before proceeding.
4. Remove the membrane using the membrane wrench (Fig. 8.1). Keep the wrench parallel to the probe while removing the membrane to ensure proper engagement with socket head cap screw.

NOTE: Do <u>not</u> leave the membrane cavity open for extended periods. Debris can become lodged in the gas openings in the plug.

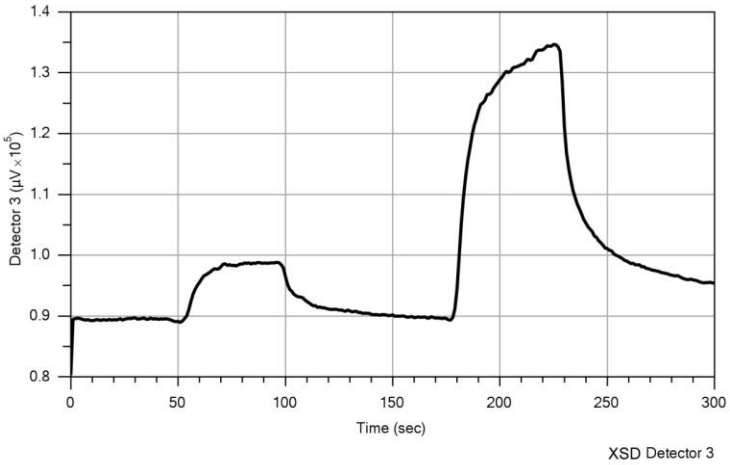
5. Remove and discard the copper washer as shown in Figure 8.2. Each new membrane is accompanied by a new copper washer. **Do not reuse the copper washer**.
6. Clean the inside of the membrane socket with a q-tip and methanol removing dirt and debris that will be present.
7. Insert the new copper washer around the brass plug making sure that it sits flat on the surface of the block.
8. Install the new membrane by threading it into the socket. Thread the membrane into the socket by hand, do not use the membrane wrench until the membrane is nearly all the way threaded. Use the membrane wrench to tighten the membrane to a snug fit. Do not over-tighten.
9. Turn the carrier gas on and leave the heater off. Apply soapy water to the membrane and surrounding area to check for leaks. If bubbles form in the water around the edges of the membrane or in the wrench holes use the membrane wrench to further tighten the membrane.
10. Use a flow meter to check carrier flow. The difference between the supply flow from the MP6505 and the return flow from the trunkline should be less than 3ml/min. Record the values in a field notebook.

Figure 8.1: Unthread the membrane from the probe block.

Figure 8.2: Remove and discard the copper washer.

APPENDIX I

Typical Response Test Data

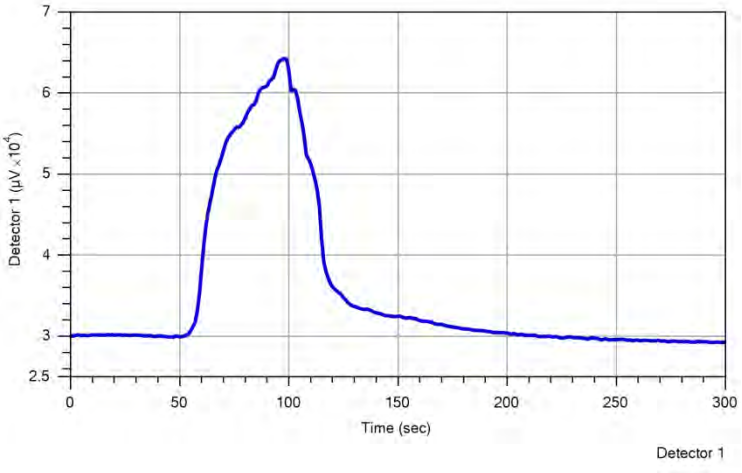


System Parameters:

MP6520 Probe with 121°C setpoint
150' PEEK Trunkline
40ml/min of Nitrogen Carrier Gas
XSD Temperature of 1,100°C

System Response:
1ppm – 9,000µV
5ppm- 45,000µV

Figure 1: Chemical Response Test: TCE 1 & 5ppm on XSD



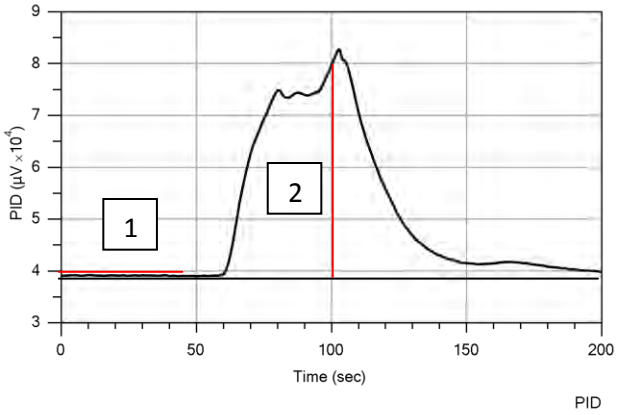
System Parameters:

MP6520 Probe with 121°C setpoint
150' PEEK Trunkline
40ml/min of Nitrogen Carrier Gas
PID Lamp intensity

System Response: 5ppm- 35,000µV

7,000µV/1ppm

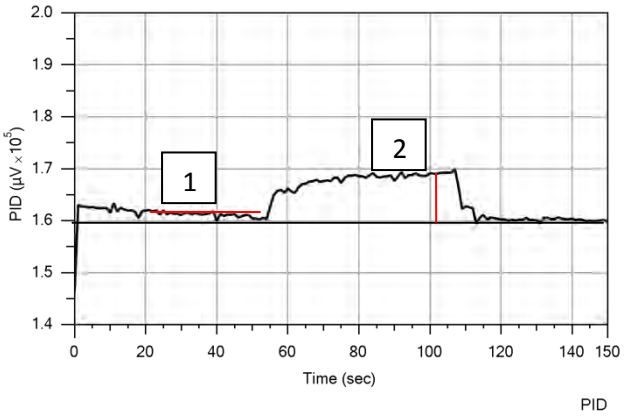
Figure 2: Chemical Response Test: Benzene 5ppm on PID



Response test - PID	5ppm Benzene
Response magnitude (2)	~40,000µV
Response/1ppm	~8,000µV
Baseline noise (1)	<500µV

Parameters: 150'TL/40ml/min flow/12PSI

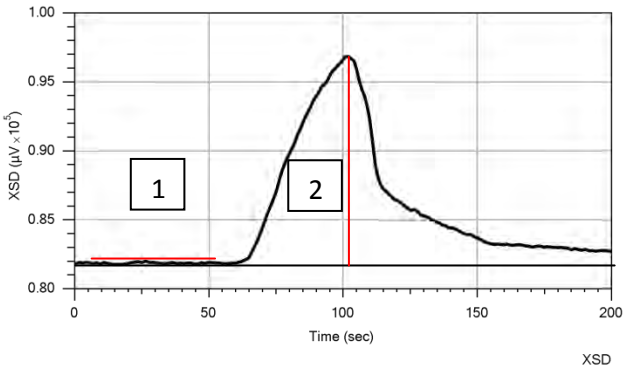
Acceptable response test. Response to baseline noise ratio is >5:1 at 1ppm



Response test - PID	1ppm Benzene
Response magnitude (2)	~8,000µV
Baseline noise (1)	~2,000µV

Parameters: 150'TL/39ml/min flow/12PSI

Not Acceptable response test
Response to baseline noise ratio is <5:1 for 1ppm Benzene
Quick Fix: Lowering carrier flow rate to 25-30ml/min will improve signal response 50% or more.



Response test - XSD	2.5ppm TCE
Response magnitude	15,000µV
Response/1ppm	6,000µV
Baseline noise	<300µV

Parameters: 150'TL/40ml/min flow/11.4PSI
Acceptable response test, Response to baseline noise ratio >5:1 for 1ppm TCE

1. Baseline noise is the amount of variation in baseline signal over a given time.
2. Signal response is the amount of rise in baseline over the stable baseline level.

APPENDIX II

Troubleshooting Guide

Loss of Pressure 1-2 PSI

- If the pressure loss has been gradual, and your MIP controller has a flow sensor check to see if the MIP supply flow has gradually dropped over the course of the log. This can happen due to the control box warming up and will be indicated by a gradual drop of both MIP pressure and flow. To resolve this increase the mass flow controller to bring the supply flow back to its original set point.
- Punctured membrane: Are there any obvious holes in the membrane with bubbles streaming out of them? Replace membrane.
- Membrane leaking out of the face – heavy frothing of bubbles on membrane face but no obvious punctures in membrane. **With the heat off**, place your thumb over the membrane**,** if the pressure goes back up to the gas pressure prior to the boring the pressure and flow loss is due to a leak in at the membrane face. Replace the membrane.
- Swagelok fitting connecting one of the trunkline gas lines to stainless steel gas line of the probe is loose. Check with soapy water, if bubbles build, fix by slowly tighten the gas line 1/16" nut to the probe.
- Examine for cuts, kinks & cracks in the length of the observable gas line. Expect to see bubbling when MEOH or soapy water is placed on it. Cut gas line prior to this and replace nut and ferrule and reconnect onto the probes steel gas line connection.
- Broken gas line somewhere else up the trunkline. Confirmed when trunkline connections are removed from the probe and close coupled. The carrier gas supply and return should be within 2ml/min, if it is >5ml/min first check with soapy water at the connecting nuts and exposed gas line then look for cuts in throughout the trunkline and see if they will show bubbles with soapy water placed on them. If this is seen you will likely need to change the trunkline.

Loss of Pressure >5 PSI

- Large puncture in membrane. Either visible puncture or observable streaming bubble when soapy water or methanol placed on membrane. Replace membrane.
- Loosen the 1/16" Swagelok nut on gas line. Check and carefully tighten.
- Broken gas line in the probe. Compare the supply versus return flow values (should $\leq$ 2/ml/min) of trunkline connected with the probe and with a close coupled trunkline. If close coupled supply/return flow is good but connected to the probe shows a big leak, there is a break is in the probe. This may be seen with soapy water placed on the edges of the heater block or on the top of the probe where the connections come out. If this produces bubbles it confirms a broken internal line or connection point. Replace the probe.

DI Acquisition - Flash Warnings:

The DI acquisition system, operated with the FI6000 field instrument, will flash a large warning screen – MIP pressure out of Range - to the operator if the probe pressure (PSI) changes over 1 PSI from the initial starting MIP pressure of the log. This alerts the operator that something in the system has changed and the operator can take the necessary precautions for a punctured membrane, broken gasline or a plug in the system.

Increase in Pressure (clearing a blockage)

- After setting the mass flow, an increase of more than 3 PSI over the original set pressure indicates a potential blockage, especially if you can verify that the pressure first dropped a 2-5 PSI prior to rising toward 20PSI.
  - Shut off the Nitrogen carrier gas flow ASAP. Do this by turning off the black regulator knob on the MIP controller or removing the carrier gas supply line from the breakout panel or the back of the MIP controller.
  - Remove the tools from the ground.
  - Look for a hole in the membrane and water or dirt got into the up-hole gas line just behind the membrane.
  - Remove connection tube and membrane.
  - Remove the trunkline gas lines from the top of the probe. Take note of which one had the gas flow coming out because this is the line that will be plugged.
  - Look for any obvious particles in either holes behind the membrane or in the gas line at the top of the probe. If any are evident attempt to remove them.
  - Take the return gas line at the surface and connect it to the supply gas connection on the breakout panel or on the back of the MIP controller.
  - Place the probe end of this line into a jar of methanol to see if the line is clear which is evident by streaming bubbles. If there are no bubbles, increase the flow to try to expel the blockage. If this does not work you may need to cut back the trunkline.
  - To clear out the probe take a 5 ml plastic syringe (or a 3 foot section of Teflon/PEEK gas line will work) filled with methanol and attempt to inject through the plugged gas line at the top of the probe. If it clears it will shoot the methanol in an arcing stream out one of the ports in the plug that sits behind the membrane.
  - The probe must be dried of the methanol which can be accelerated by heating the probe. Don't reconnect the trunkline to the detectors until you are sure the blockage is clear and the methanol is out of the system.
  - If the blockage cannot be cleared a new probe or trunkline will need to be connected.

Blinking Temperature Light

- If the temperature light on the MP6505 begins blinking in an unreadable number, it means that there is an open thermocouple in the system.
  - To complete the log in progress, replace the thermocouple for the trunkline with a thermocouple wire and twist-tie the wires together. This will fool the system to thinking there is continuity of the thermocouple wire and allow you to finish a log. The probe will continually heat set up this way and if left on when out of the ground it will overheat. When the log is complete remove the tricked thermocouple and remove tools from the ground.

- When you have the probe out of the ground, replace the thermocouple as follows.
  - Remove the connection tube from the probe.
  - Check the crimp connections of the thermocouple wires from the trunkline to the probe.
    - If one of the crimp connections has broken then strip back the wire on both sides of the thermocouple – probe and trunkline ends and reconnect in a new crimp connection and see if the probe temperature comes back.
    - If the thermocouple connection is good, the thermocouple wire in the probe has likely broken. Cut off the crimp connections of the thermocouple wires between the probe and the trunkline Check the resistance between the red and yellow thermocouple wires coming out of the probe. A resistance reading of approximately 40ohms indicates that the thermocouple is good reconnect. If they are open (O.L.) or mega ohms then the leads are broken on the thermocouple. Replace the thermocouple.
- To check the trunkline thermocouple wires, measure each wire from top to bottom. The resistances will be different between the two colored wires but should be somewhere approximately 50 ohms – 150ohms for the length of the trunkline. The resistances will also increase with an increase in trunkline length.
  - If they are open (no resistance) then there is a break in the trunkline. Replace the trunkline.

Blinking temperature readout or
Spiking in the Pressure and/or Temperature Data

- If spikes show up in the temperature or pressure data especially when related to hammer strikes it is likely an intermittent break in the thermocouple connection. Spiking of the temperature may reach single point readings of 250^{o}C in the data but may not be visible when watching the temperature display on the MIP controller.
  - When you check the resistance between the two thermocouple wires they may check out at approximately 40 ohms, however there likely is an intermittent break in the wire.
  - Replacing the thermocouple should eliminate the pressure and temperature data spikes.

Probe Not Reaching Temperature

- If the heater light is on but the temperature seems low (<100^{o}C with a set point of 120^{o}C) a heater may have broken in the probe.
  - Check the resistance of the heater wires.
    - If a heater is broken the resistance will be over 40 ohms. The probe needs to be replaced.
    - Two good heaters will read approximately 22 ohms on the MP6520, MP8520 and MK6530.
      - Check to see if the thermocouple has pulled of few inches out of the probe.
        - If the thermocouple duct has broken and pulled back away from the probe, the probe will need to be replaced and rebuilt.
        - A thermocouple can unscrew and vibrated loose out of the thermocouple duct connection if it is not secured with shrink tubing or electrical tape. Reseated back into the leur-lock connection and secure. When the thermocouple pulls away from the probe it measures the probe temperature in the wrong location.

Flash Warning:

The DI acquisition system, operated with the FI6000 field instrument, will flash a large warning screen – Temperature out of Range - to the operator if the temperature goes outside of a set range from the setpoint temperature of 121^{o}C. This alerts the operator that something in the system has failed and the operator can take the necessary precautions for a broken probe heater or thermocouple problem.

System explanations and warnings

MIP Flow

MIP flow is the carrier gas flow set by the MIP controller. This flow is supplying carrier gas to the trunkline and probe and is typically set to approximately 42ml/min. This parameter may be monitored by the DI-Acquisition system if the operator has the necessary components in their MIP Controller. The return flow, or Flow-R, is the flow coming back to the GC up the return gas line. Flow-S and Flow-R should be within 3-4ml/min and are usually much closer.

MIP Pressure

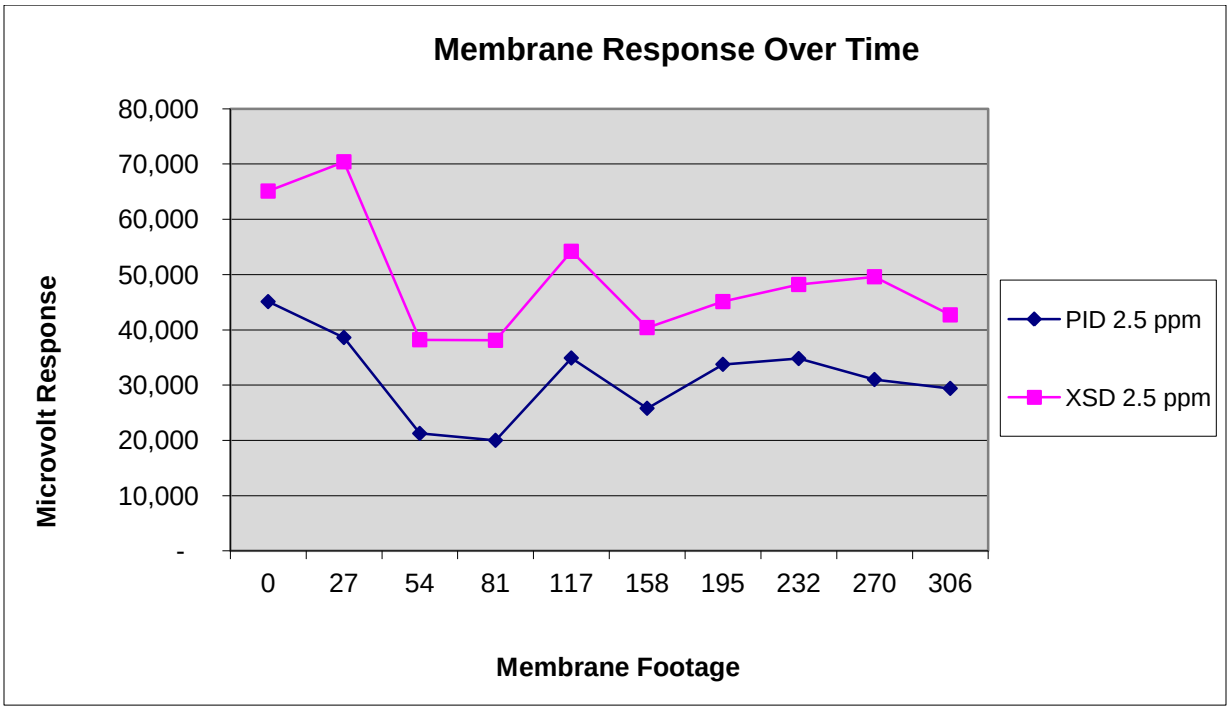
The MIP pressure is the back pressure of the carrier gas as it moves through the trunkline and probe. This is monitored digitally on the DI-Acquisition screen as well as by an analog pressure gauge on the front of the MIP controller. The MIP pressure is directly related to the MIP return flow (Flow-R) and the length of the trunkline. If the MIP pressure falls, the return flow has also dropped, if the MIP flow (Flow-S) has remained the same then there is likely a punctured membrane of problem with the gas lines.

APPENDIX III

Membrane Performance Control Charts

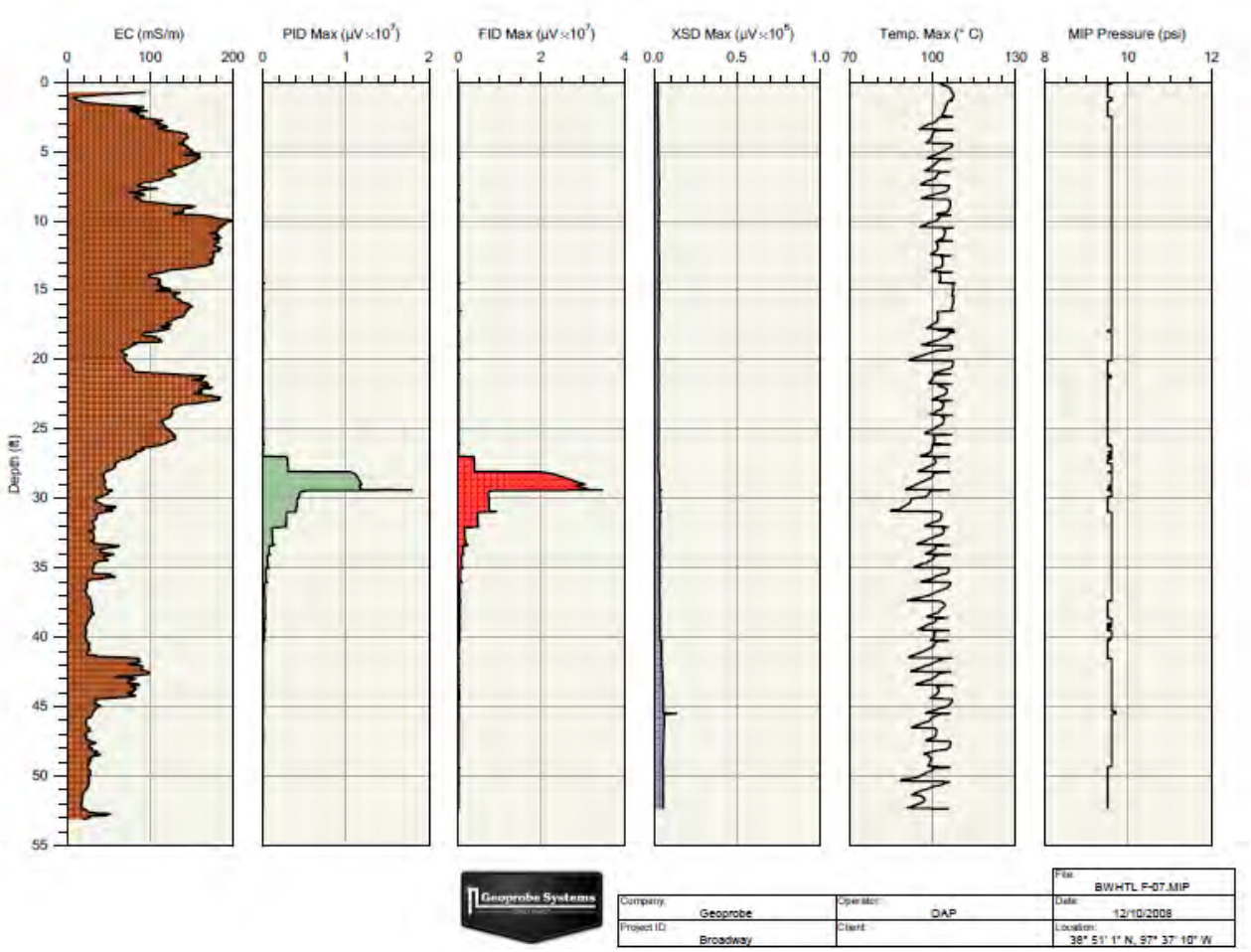
Response Tests using TCE

Pre/Post Log Response Test	Log ID:	PID Response 2.5ppm	XSD Response 2.5ppm	Log Footage	Membrane Footage
Pre-Log	MIP01	45,100	65,100	27	0
Pre-Log	MIP02	38,600	70,400	27	27
Pre-Log	MIP03	21,250	38,200	27	54
Pre-Log	MIP04	20,000	38,100	36	81
Pre-Log	MIP05	34,900	54,200	41	117
Pre-Log	MIP06	25,800	40,400	37	158
Pre-Log	MIP07	33.750	45,100	37	195
Pre-Log	MIP08	34,800	48,200	37	232
Pre-Log	MIP09	31,000	49,600	36	270
Post-Log	MIP09	29,400	42,700		306



APPENDIX IV

Sample Logs and Interpretation



Here is a MIP log with the graphs left to right: electrical conductivity, detectors (PID, FID and XSD), probe temperature and trunkline carrier gas pressure.

The above log shows contamination from 27ft to 33ft bgs. The main detector response is on the PID and FID with minimal response on the XSD (Halogen Specific Detector). This indicates that the main contaminant would not contain halogenated (Cl-, Br-, Fl-) atoms, but would likely be hydrocarbon based. The contaminants are present in the lower electrical conductivity formations which typically indicate courser grained formations of higher permeability. The temperature deflections of the MIP block heater are indicative of the probe heat cycling and the trunkline carrier gas maintains a constant stable pressure which indicates no leak or plug issues occurred with the gas line or membrane during the log.

Detector Interpretation

Standard MIP systems are able to identify compound families and determine general compound classes. The identification of individual compounds is not possible. Standard MIP systems have a continuous carrier gas flow that is brought to the detectors from the down-hole probe. To be able to effectively speciate (determine specific contaminant chemicals) the operator would need a highly modified system in place. The carrier gas stream would need to be run through a mass spectrometer or trapped and run a secondary GC onsite.

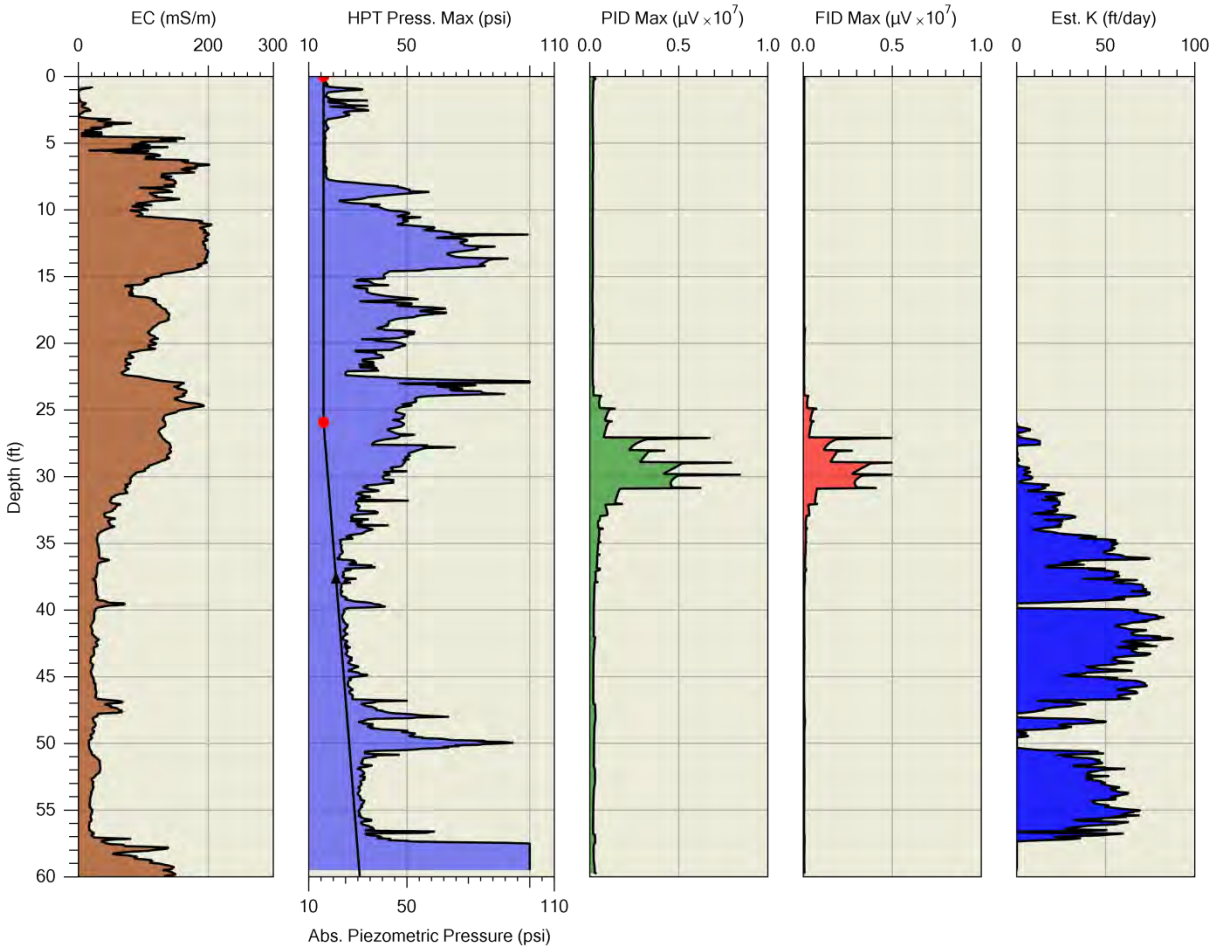
Typical standard MIP configurations use 3 gas phase detectors: a photo-ionization detector (PID), flame-ionization (FID) and a halogen specific detector (XSD). The PID responds to compounds which have an ionization potential $\leq$ electron voltage of the PID bulb. These compounds include both chlorinated and non-chlorinated hydrocarbons. A typical PID bulb has a 10.6eV lamp. The FID will respond when organic compounds (anything containing carbon) are present in the carrier gas stream in high enough concentration burn up in the flame which increases the flames ionization voltage. The XSD responds only to halogenated compounds which are made up of chlorinated (most typical halogen environmental contaminant), brominated and fluorinated compounds. Based upon which detector or detector series a contaminant responds on, we can determine if the contaminants are halogenated or petroleum based.

Petroleum hydrocarbons will respond on the PID and FID but not on the XSD. Fresh gasoline primarily contains aromatic hydrocarbons such as benzene, toluene, ethyl benzene and xylenes, which respond strongly on a photo-ionization detector (PID) and not so well on the FID. As gasoline breaks down or weathers the molecular structure changes from primarily aromatic to mainly straight chain hydrocarbons (single bonded hydrocarbons). Straight chain hydrocarbons typically do not show up on the PID do having a higher ionization potential but will respond on a flame ionization detector (FID). Weathered petroleum will still have a decent signal on the PID but may show a stronger FID signal.

Chlorinated compounds such as trichloroethylene and perchloroethylene are detected by the XSD and PID and respond in a similar profile. This is typical of the common double bonded chlorinated compounds seen in the subsurface which have an ionization potential that the PID can see. Chlorinated compounds without multiple bonds such as chloroform, methylene chloride and 1,1,1,-trichloroethane have an ionization potential higher than the PID electron voltage which results in a solid response on the XSD but will not show up on the PID.

The only sure way of determining contaminant concentration from MIP responses is to take confirmation soil and/or groundwater samples for laboratory analysis. After obtaining the results the actual concentrations can be compared to the MIP detector responses and concentrations may be estimated across the site.

MiHPT Log Example– Combined MIP & HPT



The addition of the HPT sensor to the MIP detectors and EC has provided valuable information to the subsurface lithogy encountered by the MIP operator. The above log shows graphs left to right: electrical conductivity, HPT injection pressure with the absolute piezometric pressure profile on the secondary axis, detectors (PID and FID) and estimated hydraulic conductivity (K).

The above log shows contamination from 24ft to 31ft bgs both on the PID & FID at similar magnitudes which is likely from petroleum hydrocarbons but without showing the XSD we cannot tell for sure that there are no halogenated (Cl-, Br-, Fl-) compounds present. The contaminants are present in the higher electrical conductivity and HPT pressure formations which indicate finer grained formations of lower permeability. The second graph with the Absolute Peizometric profile graph has a triangle on the increasing line at approximately 37 feet which indicates that an HPT dissipation test was performed at that depth. By taking the hydrostatic pressure at that interval and subtracting off the weight of water (0.445psi/ft) until the atmospheric pressure (calculated in the pre log HPT reference test) we can see the static water table is approximately 26 feet indicated by the red dot. Estimated hydraulic conductivity (K) is shown as the final graph which is a relationship between HPT injection pressure and HPT flow.

APPENDIX V

GC1000 Configuration and Operating Parameters

Figure 1: GC1000: SRI 310GC with XSD Controller

GC1000 Configuration

SRI310 GC with PID, FID & OI Analytical XSD
(all standalone detectors)

Flows:

TL Carrier (N_2): 40ml/min
Detector split 60:40 – 24ml/min-XSD
16ml/min-FID

Nafion Dryer (installed in GC Oven)
80ml/min (2x carrier flow rate)

A built in air compressor is split underneath the GC between the XSD & FID. The XSD & FID air supply is controlled through the GC air pressure screw control on front of GC and with different air line sizes and lengths to provide 250ml/min to the FID and 30 ml/min to the XSD.

Detectors front of GC to back: XSD, FID & PID

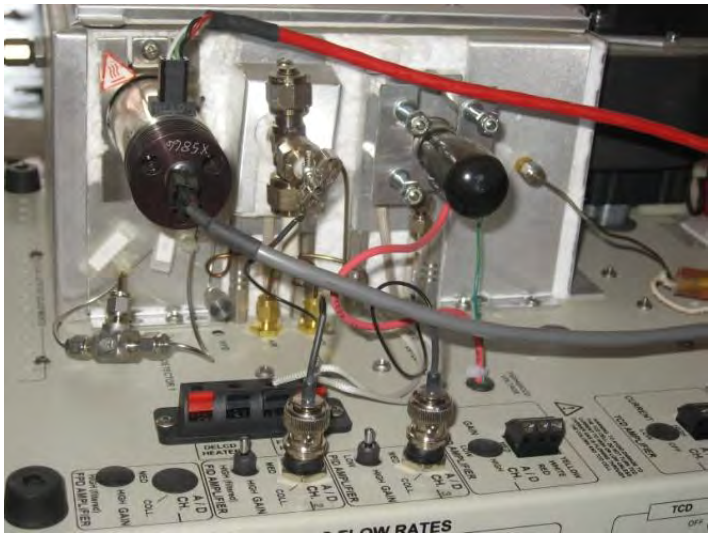


Figure 2: GC Detectors – left to right - XSD, FID, PID

SRI 310 GC Detector 1 position – XSD
(not controlled by GC)

SRI 310 GC Detector 2 position – FID

SRI 310 GC Detector 3 position – PID

Nafion dryer installed inside GC oven

GC Oven set to 85°C – 130°C max temp.

Flow comes into the GC oven via a 1/16" bulkhead fitting located in the 4th detector position furthest back (upper right inside oven) behind the PID detector. The trunkline will connect to this bulkhead and a 1/16" stainless steel line transports flow into the Nafion dryer. Silco steel takes this to the PID lamp which is inserted up to the lamp and backed off a 1/16" and tightened. A 1/16" stainless steel line brings it back into the GC oven where it is split between the FID and XSD and sent to them via a silco-steel line to the XSD and a stainless steel line to the FID.

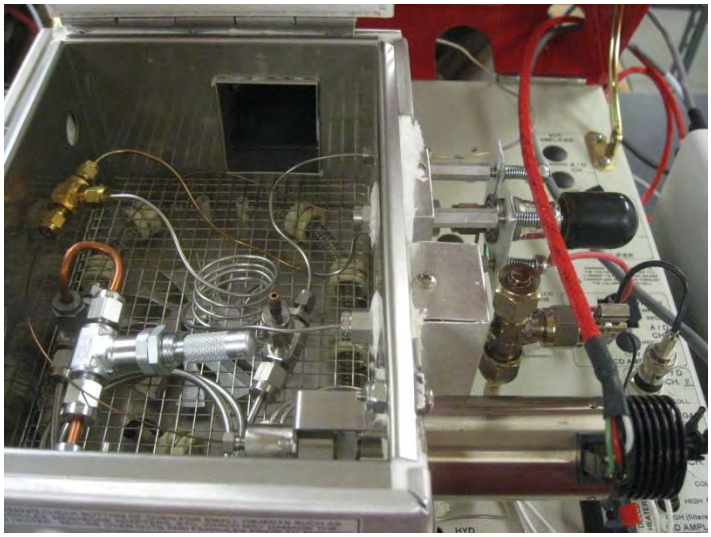


Figure 3: GC Oven Configuration

Detector Operating Parameters:

PID:

- MIP Carrier Flow (N_2) – 100% - 40ml/min
- Carrier return back into oven split between XSD & FID
- Detector Temperature setting – 150°C
- PID current 70 (0.70ma)

FID:

- Carrier N_2 MIP effluent – 40% - 16ml/min
- Hydrogen – 25ml/min
- AIR – 250ml/min
- Detector Temperature setting – 250°C
- FID igniter set at -600 (6.0V)

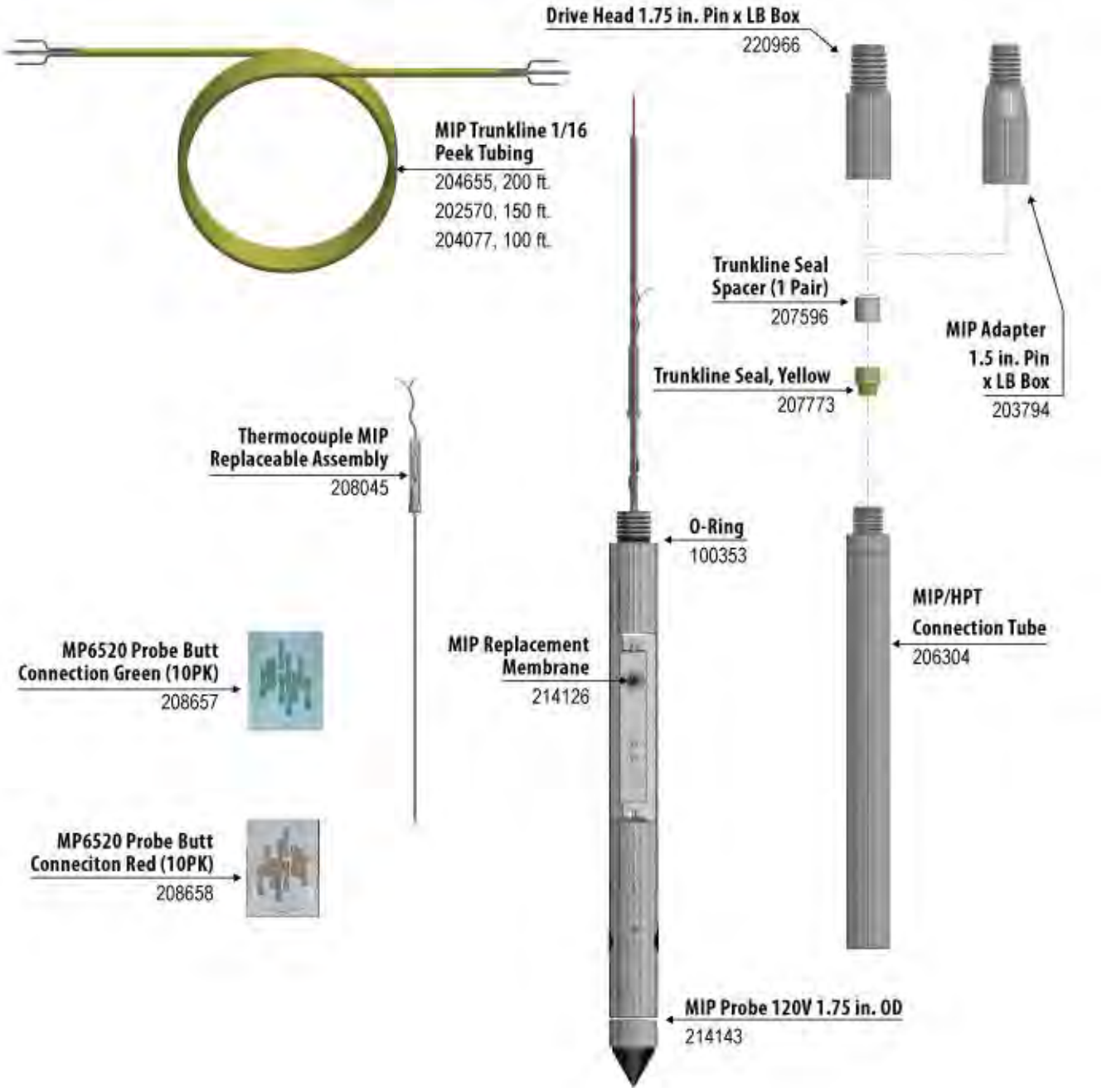
XSD:

- Carrier N_2 MIP effluent – 60% - 24ml/min
- Air – 30ml/min (split 50:50 wall & jet input of XSD)
- Detector Temperature setting – 1,100°C

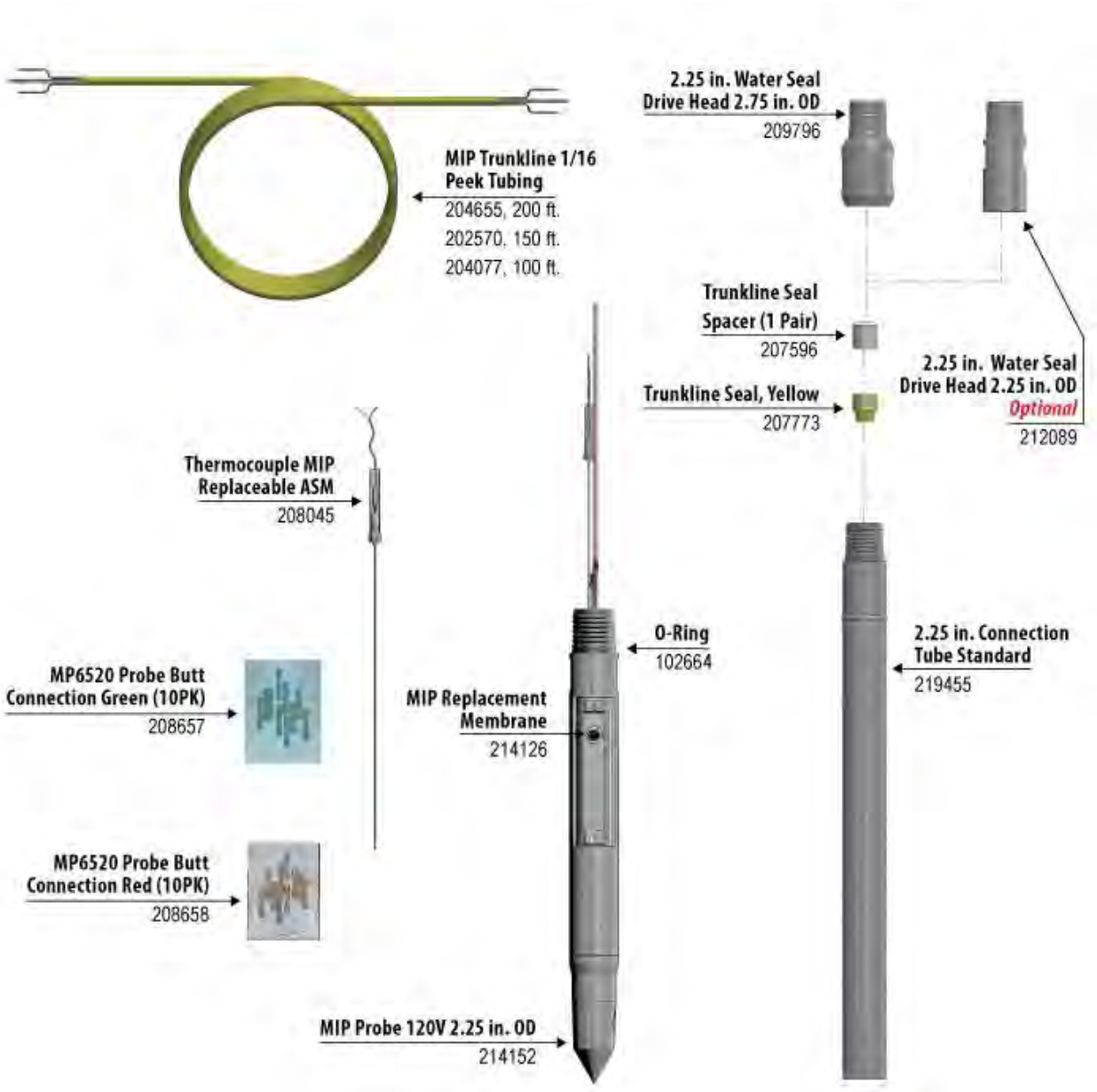
APPENDIX VI

MIP Tool Configurations

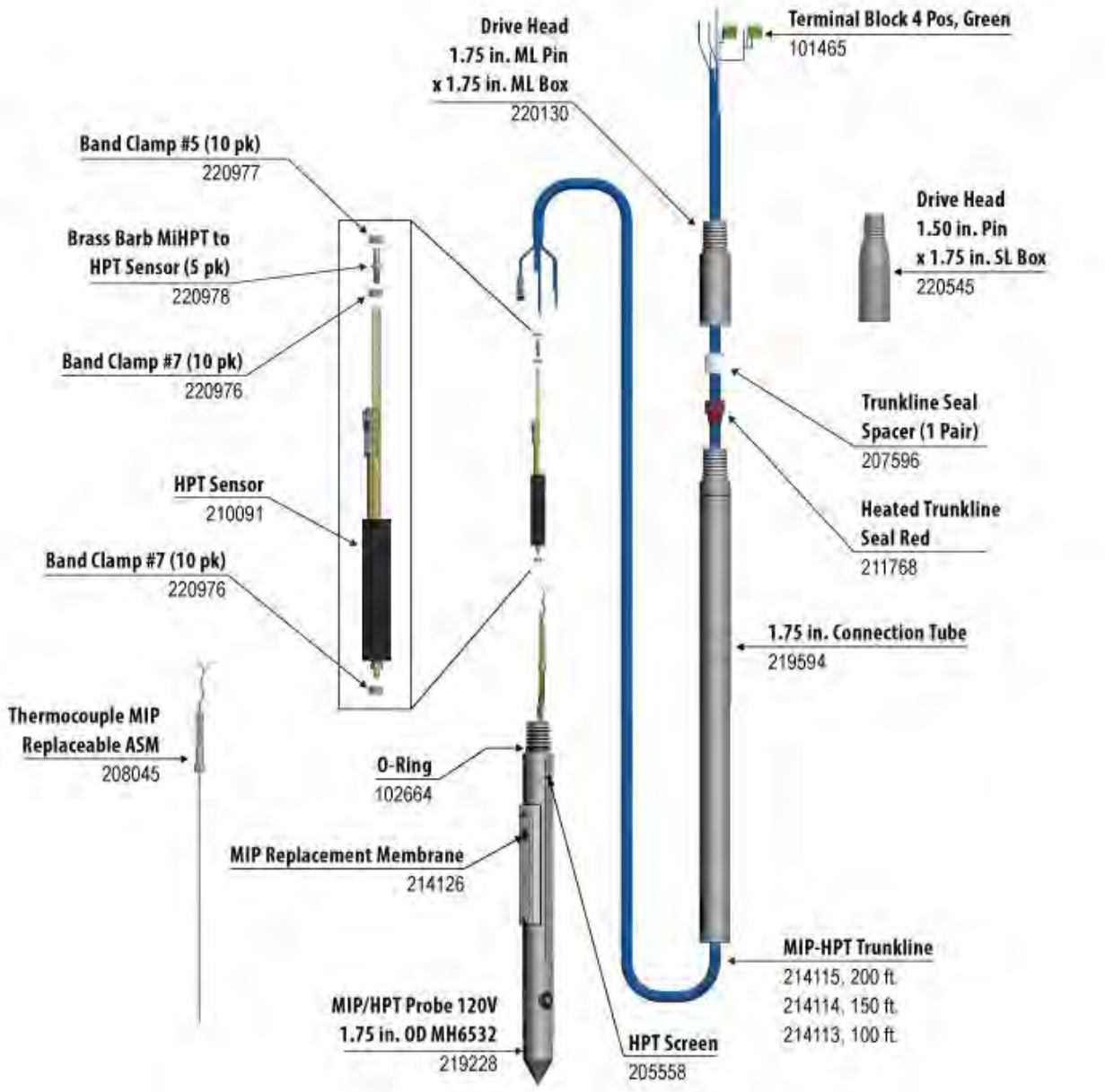
MIP - MP6520 (1.5 in. / 1.75 in. system)



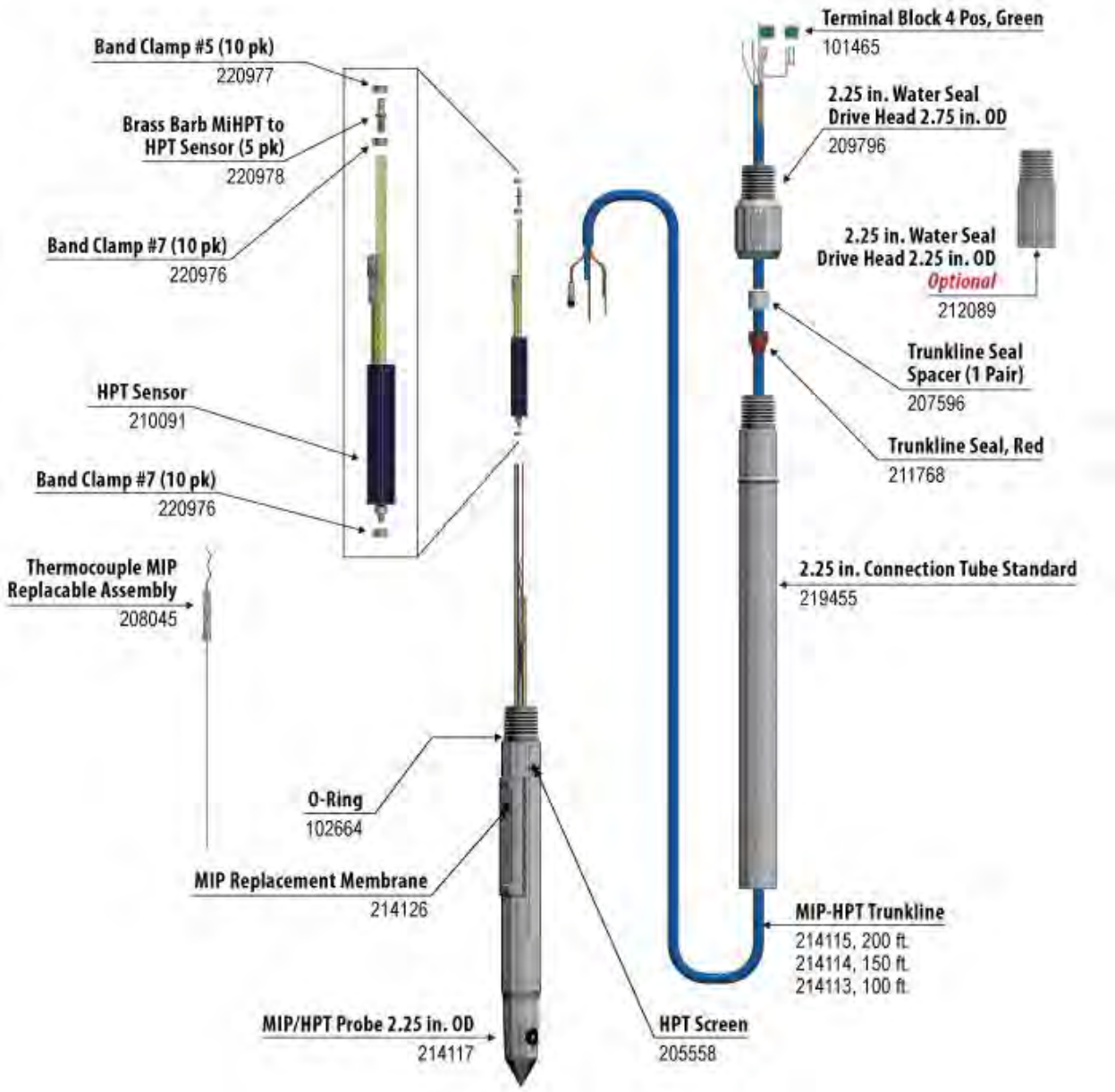
MIP – MP8520 (2.25 in. System)



MiHPT – MH6532 (1.5 in. / 1.75 in. system)



MiHPT – MH8530 (2.25 in. System)



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Attachment 2 Field Forms

TRINITY
ANALYSIS & DEVELOPMENT CORP.
Environmental & Engineering Services

CONTRACTOR'S DAILY QUALITY CONTROL REPORT

PROJECT NAME: _____ Date: _____
Location: _____ Weather: _____

PERSONNEL: Name	Company	EQUIPMENT: Description	License Number	FIELD INSTALLATIONS: ID Nos.	Drilled from (ft):	Drilled to (ft):

Brief Description of Work Performed?

Environmental or Geotechnical Samples Collected:

Health and Safety Levels:

Changes from Work Plan:

Remarks:

Signature: _____

HTRW DRILLING LOG

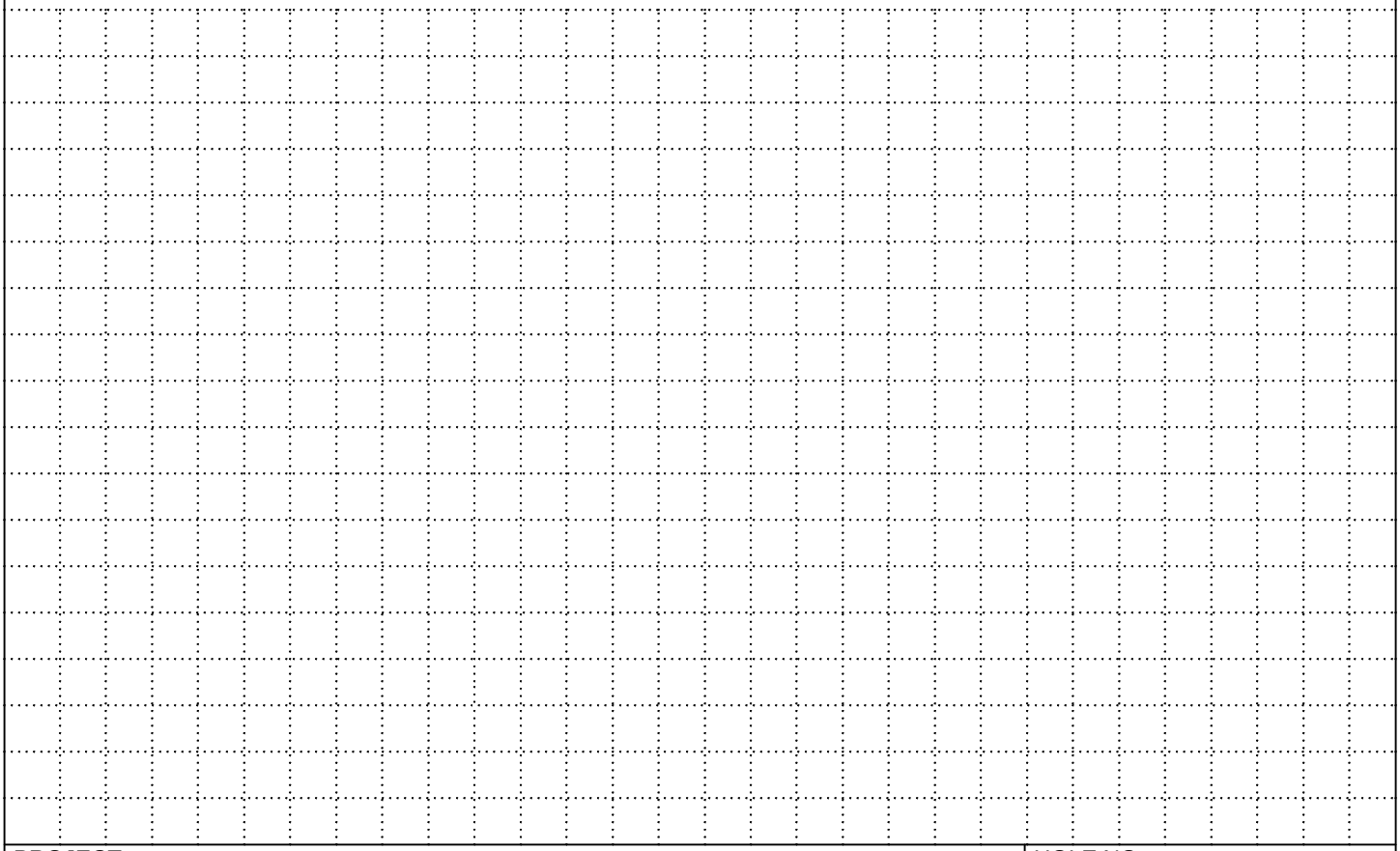
DISTRICT		HOLE NUMBER
1. COMPANY NAME	2. DRILLING CONTRACTOR	SHEET OF SHEETS
3. PROJECT	4. LOCATION	
5. NAME OF DRILLER	6. MANUFACTURER'S DESIGNATION OF DRILL	
7. SIZES AND TYPES OF DRILLING AND SAMPLING EQUIPMENT	8. HOLE LOCATION	
	9. SURFACE ELEVATION	
	10. DATE STARTED / 11. DATE COMPLETED	
12. OVERBURDEN THICKNESS	15. DEPTH GROUNDWATER ENCOUNTERED	
13. DEPTH DRILLED INTO ROCK	16. DEPTH TO WATER AND ELAPSED TIME AFTER DRILLING COMPLETED	
14. TOTAL DEPTH OF HOLE	17. OTHER WATER LEVEL MEASUREMENTS (SPECIFY)	

18. GEOTECHNICAL SAMPLES	DISTURBED	UNDISTURBED	19. TOTAL NUMBER OF CORE BOXES

20. SAMPLES FOR CHEMICAL ANALYSIS	VOC	METALS	OTHER (SPECIFY)	OTHER (SPECIFY)	OTHER (SPECIFY)	21. TOTAL CORE RECOVERY %

22. DISPOSITION OF HOLE	BACKFILLED	MONITORING WELL	OTHER (SPECIFY)	23. SIGNATURE OF INSPECTOR

LOCATION SKETCH/COMMENTS SCALE:



PROJECT	HOLE NO

ENG FORM 5056-R, AUG 94 (Proponent: CECW-EG)

HTRW DRILLING LOG (CONTINUATION SHEET)

PROJECT	INSPECTOR	HOLE NUMBER SHEET SHEETS OF

ELEV. (a)	DEPTH (b)	DESCRIPTION OF MATERIALS (c)	FIELD SCREENING RESULTS (d)	GEOTECH SAMPLE OR CORE BOX NO. (e)	ANALYTICAL SAMPLE NO. (f)	BLOW COUNT (g)	REMARKS (h)

PROJECT	HOLE NO

ENG FORM 5056A-R, AUG 94 (Proponent: CECW-EG)

TRINITY
ANALYSIS & DEVELOPMENT CORP.
Environmental & Engineering Services

FIELD DATA SHEET

Site Name:	Sample Date:	Sample Time:	Sample Number:
Sampled By:	Signature(s):	Sampling Method:	Sampling Location:
Client:	Contract Number:	Delivery Order:	Chain of Custody Number:

Solid Sample

Solid Sample Type:

- ☐ Surface Soil
- ☐ Subsurface Soil
- ☐ Sediment
- ☐ Waste
- ☐ Other _____

Sample Collection:

- ☐ Grab
- ☐ Composite
- ☐ Multi-increment
- ☐ Other _____

Sample Description (classification, color, plasticity, moisture content, consistency)

Aqueous Sample

Aqueous Sample Type:

- ☐ Surface Water
- ☐ Groundwater
  - ☐ Monitoring Well
  - ☐ Domestic Well
  - ☐ Other _____
- ☐ Seep
- ☐ Sump
- ☐ Waste
- ☐ Other _____

Well Information

Well Casing Size:
Total Well Depth
Static Water Level:
One Purge Volume:
Start Purge:
End Purge:
Total Purge Time:
Total Purge Volume:
Purge Method:

Analysis

☐ Volatiles ☐ Semivolatiles ☐ Ions ☐ RCRA Metals ☐ TAL Metals ☑ Select Metals (list) _____

☐ Pesticides ☐ Herbicides ☐ PCB ☐ Cyanide ☐ Explosives ☐ Other (list): _____

Purge Data

Time (hrs)	Temperature (ºC)	pH (SU)	Conductance (mS/cm)	D.O. (mg/L)	ORP (mv)	Turbidity (NTU)

Comments:

Weather Conditions: cloudy rainy sunny snowy	Temperature:	Barometer:

Rev. 4/2010

CHAIN OF CUSTODY

Page _____ of _____

Company:
Project Contact:
Telephone:
Project Name:
Project #:
Location:
Sampled By:

CT LABORATORIES

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www.ctlaboratories.com

Lab Use Only
Place Header Sticker Here:

Program:
QSM RCRA SDWA NPDES
Solid Waste Other _____

PO #

Report To:
EMAIL:
Company:
Address:

Invoice To:*
EMAIL:
Company:
Address:

**Client Special Instructions*

*Party listed is responsible for payment of invoice as per CT Laboratories' terms and conditions

Matrix:
GW – groundwater **SW** - surface water **WW** - wastewater **DW** - drinking water
S - soil/sediment **SL** - sludge **A** - air **M** - misc/waste

Turnaround Time
Normal RUSH*
Date Needed:
_____
Rush analysis requires prior CT Laboratories' approval
Surcharges:
24 hr 200%
2-3 days 100%
4-9 days 50%

Collection Date	Collection Time	Matrix	Grab/ Comp	Sample ID Description	Filtered? Y/N	ANALYSES REQUESTED	Total # Containers	Designated MS/MSD	CT Lab ID # (Lab use only)
						Fill in Spaces with Bottles per Test			

Relinquished By:	Date/Time	Received By:	Date/Time
Received by:	Date/Time	Received for Laboratory by:	Date/Time

Lab Use Only
Ice Present Yes No
Temperature _____
Cooler # _____

CUSTODY SEALS

Date _____

Signature _____

Attachment 3 Analytical Standard Operating Procedures

CT LABORATORIES

delivering more than data from your environmental analyses

STANDARD OPERATING PROCEDURE

VO 004 Analysis of Volatile Organic Compounds by GC/MS (8260C)

Review Date: 3/09/15

03/09/2015

Technical Review by: Date

04/01/15

Approved by: Quality Assurance Date

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1. SCOPE OF APPLICABILITY

1.1. This method is used to quantify Volatile Organic Compounds (VOCs) with boiling points below 200° Celsius (°C) in water and soils. See Table 1 for typical target analyte list (TAL). This method is designed to follow procedures and QC requirements found in EPA SW-846 method's EPA 624, 5030B, 5035, 8000C and 8260C in order to determine quantities of volatile organic compounds found in a variety of different sample.

1.2. The scan mode is utilized by the instrument's software to identify and quantitate analysis results. When collecting data in the full scan mode, a target range of mass fragments is determined and put into the instrument's method. An example of a typical broad range of mass fragments to monitor would be *m/z* 35 to *m/z* 300. The determination of what range to use is largely dictated by what one anticipates being in the sample while being cognizant of the solvent and other possible interferences. A MS should not be set to look for mass fragments too low or else one may detect air (found as *m/z* 28 due to nitrogen), carbon dioxide (*m/z* 44) or other possible interferences. Additionally if one is to use a large scan range then sensitivity of the instrument is decreased due to performing fewer scans per second since each scan will have to detect a wide range of mass fragments. Full scan is useful in determining unknown compounds in a sample. It provides more information than Selected Ion Monitoring (SIM) when it comes to confirming or resolving compounds in a sample. During instrument method development it may be common to first analyze test solutions in full scan mode to determine the retention time and the mass fragment fingerprint before moving to a SIM instrument method.

1.3. Volatile organic compounds are quantitated from a variety of matrices. This method is applicable to nearly all types of samples regardless of water content, including ground water, surface water, wastewater, soils, sediments, and TCLP/SPLP extracts; as well as, other matrices noted in SW-846 method 8260C.

1.4. Examples of other compounds which have been analyzed by this method include: iodomethane, 2,3-dichloro-1-propene, 1-chlorohexane, acrolein, acrylonitrile, 1,1,2-trichloro-1,2,2-trifluoroethane, ethyl ether, hexane, ethyl acetate, 1-chlorohexane, 2-Chloroethylvinyl ether, methyl acetate, methyl methacrylate, cyclohexane, and cyclohexanone. Ethanol, 2-propanol, tert-butylalcohol, 1,4-dioxane may also be analyzed using this method but are poor responders. To achieve lower detection limits for these types of compounds, the SIM mode can be utilized. In selected ion monitoring certain ion fragments are entered into the instrument method and only those mass fragments are detected by the mass spectrometer. The advantages of SIM are that the detection limit is lower since the instrument is only looking at a small number of fragments (e.g. three fragments) during each scan. More scans can take place

each second. Since only a few mass fragments of interest are being monitored, matrix interferences are typically lower. To additionally confirm the likelihood of a potentially positive result, it is relatively important to be sure that the ion ratios of the various mass fragments are comparable to a known reference standard.

1.5. SW-846 method 8260C notes a number of other compounds amenable to this test.

2. SUMMARY OF METHOD

2.1. A Purge & Trap system (including autosampler), a Gas Chromatograph (GC), and a Mass Spectrometer (MS) are utilized for the detection of VOCs. The autosampler introduces the sample to the purge and trap concentrator. The concentrator then removes the volatile constituents by purging the sample with an inert gas (helium or nitrogen). The constituents are then collected onto an adsorption trap. The trap is then rapidly heated and the volatilized compounds are introduced to the GC. The GC is temperature programmed to facilitate separation of the individual organic compounds. Finally the separated compounds enter the MS (which is interfaced with the GC) for quantitative and qualitative analyses.

2.2. Utilizing computer software, identification of target analytes is accomplished by comparing the mass spectra of the sample constituent with that of commercially purchased standards. Quantitation is achieved by comparing the response of a quantitation ion relative to an internal standard using a five point (minimum) calibration curve.

3. DEFINITIONS

3.1. For definitions on all terms applicable to this method, see Section 25.1 of the Quality Assurance Manual (QAM).

3.2. For a list of common acronyms and abbreviations, see QAM.

4. HEALTH AND SAFETY

4.1. Gloves and protective clothing shall be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure must utilize appropriate laboratory safety systems.

4.2. The toxicity and carcinogenicity of the chemicals used in this method are not precisely defined. Each chemical and sample shall be treated as a potential health hazard, so care must be taken to prevent undue or extensive exposure.

5. INTERFERENCES

5.1. Volatile materials in the laboratory and impurities in the purging gas and sorbent trap can cause significant amounts of background contamination. Improper tubing such as certain plastics and rubber shall not be used. The analysis of IBs and MBs will indicate as to whether or not this type of contamination is present. Since subtraction on background contamination is not allowed, care must be taken to eliminate this type of contamination.

5.2. Carry over contamination is a problem when a highly contaminated sample is followed by a clean sample. Rinsing the autosampler and concentrator and adequate baking of the trap can greatly reduce contamination from carry over.

5.3. Some samples contain a lot of water soluble materials, suspended solids, compounds with high boiling points, or target analytes with very high concentrations which may contaminate some or all of the analytical system. Removing components of the system for cleaning or cleaning of the entire system may be required to eliminate the interferences.

5.4. Compounds with poor purging efficiencies may remain in the purge system, particularly with 25 ml purges. Ensuring adequate rinsing and increased line temperatures will help reduce this problem.

5.5. All chromatography gas/purge lines shall be stainless steel or copper to prevent permeation from possible background contaminants (i.e. Methylene chloride). Background levels of Methylene chloride are possible so care needs to be taken to reduce this possibility. Analyst clothing previously exposed to Methylene chloride must not be worn and isolating the instruments from possible air born contamination is essential in reducing Methylene chloride background contamination.

5.6. A trip blank normally accompanies sample in shipment and storage as a check on possible contamination from volatile organics by diffusion through the septum seal in sample vials/containers.

5.7. Mass spectrometer sensitivity, column degradation, and contamination can also contribute to background interferences. A proper maintenance procedure on instrumentation is essential to continually producing quality data. Maintenance manuals are provided with each piece of equipment and are essential for proper instrument care. The presence of semi-volatile hydrocarbons need also be taken into consideration, so appropriate post analysis bake out times need to be incorporated.

5.8. Cross-contamination can be a possibility when samples containing high concentrations of target analytes are stored in the same location as other

samples. To prevent cross-contamination, samples suspected of containing high concentrations of volatiles organics should be isolated from other volatile organics samples. Storage Blanks are analyzed bi-weekly to determine whether cross-contamination has occurred.

6. EQUIPMENT AND SUPPLIES

6.1. 40 ml screw cap "VOA" vials-borosilicate glass with a Teflon faced silicone septum (C&G or equivalent)

6.2. 2 oz., 4 oz., or 60- ml Teflon lined screw top sample jars (C&G or equivalent).

6.3. 5 g or 25 g samplers for low level soils (Encore).

6.4. Top loading balance sensitive to 0.01 g (Mettler-Toledo, BD202).

6.5. pH paper to confirm water sample preservation(Color pHast, EM Reagents).

6.6. Stainless steel spatulas.

6.7. 10, 25, 50, 100, 500, and 1000 ul gas tight syringes for sample dilutions and standard preparation (Hamilton or equivalent).

6.8. 5.0, 10.0, 25.0, 50.0 ml syringes with luer-lok tips for methanol preserved soil sample preparation and sample dilutions (Hamilton/SGE or equivalents).

6.9. 10, 50 100, 200, 1000, and 2000 ml Volumetric flask for sample dilutions and standard preparation (Class A, Pyrex/Kimble or equivalents).

6.10. Auto pipetter – 2.5 to 25.0 ml – for dispensing methanol (Dispensette).

6.11. Sonicator used for methanol-preserved soil sample extraction (Fisher, FS-28 or equivalent).

6.12. Auto sampler used for sample introduction to the Purge and Trap (Archon, EST-Centurion or equivalents).

6.13. 3 ml standards vial (Mininert or equivalent).

6.14. Purge and Trap concentrator (EST-Encon Evolution or equivalent).

6.14.1. The glass purging tubes are of 5 ml or 25mL size. The all-glass purging device shall be designed to accept 5 or 25 ml samples with a water column at least 5 cm deep. The smaller (5 ml) purging device is

recommended if the GC/MS system has adequate sensitivity to obtain the method detection limits required for a specific project or program.

6.14.2. The traps currently used are Supelco Type K or EST-EV1. As required by SW-846 methods, the trap must be at least 25 cm long and have an inside diameter of at least 0.105 inches. Starting from the inlet, the trap contains 1.0 cm of methyl silicone coated packing and the following amounts of adsorbents: 33% of 2,6-diphenylene oxide polymer, 33% of silica gel, and 33% of coconut charcoal.

6.15. Gas Chromatograph/Mass Spectrometer Data Systems (GC/MS).

6.15.1. Hewlett Packard Gas Chromatographs (5890 & 6890).

6.15.1.1. Columns, Supelco (SPB-624), Agilent (DB-624UI), or Zebron (ZB-624).

6.15.1.2. 30 Meter x 0.25 mm ID, 1.4 um film thickness or equivalents.

6.15.2. Hewlett Packard 5972 & 5973 Mass Spectrometers.

6.15.3. Hewlett Packard Chemstation Data Management System (version G1701AA v. A.03.02 for the 5972's) and MSD Chemstation (version D.01.02.16.15 for the 5973) with Enviroquant and Prolab data processing software.

7. REAGENTS AND STANDARDS

7.1. Purge and trap grade methanol: (Fisher, Purge & Trap grade or equivalent), stored in laboratory warehouse.

7.2. Reagent grade water, organic free (Milipore, 18 mega ohm quality).

7.3. Certified Calibration Standards:

7.3.1. (VOC Mix--2000 ug/ml, Ultra Scientific--#DWM-588; Addition mixes--1000/10,000 ug/ml, SPEX Certiprep--#'s VO-CTWI-4 & VO-CTWI-5 or equivalents), stored in VOC Standards Freezer in Volatiles laboratory at $\leq$ -10 °C.

7.3.2. A 100 ug/mL[1] Continuing Calibration Verification (CCV/Calib.) working standard is prepared by adding 150 ul of the VOC mix and 300 ul of

Addition mix VO-CTWI-4 and 300 ul of Additions Mix VO-CTWI-5 to 2250 ul of methanol into a 3 ml Mininert vial.

7.3.3. Calibration standards used for SIM mode calibrations can be prepared by further dilution of the working standards (7.3.2) or by purchasing individual compound standards (e.q. 1000 ug/mL 1,4-dioxane, SPEX Certiprep--#S175 or equivalent). For t-butyl alcohol and 1,2-dioxane the working CCV/Calib. standard concentration is 100 ug/ml.

7.4. Certified Calibration Check Standards:

7.4.1. (VOC Mix--2000 ug/ml, Accustandard--#M-502-10X; Addition mixes--1000/10,000 ug/ml, SPEX Certiprep--#'s VO-CTWI-4 & VO-CTWI-5 or equivalents), stored in VOC Standards Freezer in Volatiles laboratory at ≤ -10 °C.

7.4.2. A 100 ug/mL[1] Initial Calibration Verification (ICV/Spiking) working standard is prepared by adding 150 ul of the VOC mix and 300 ul of Addition mix VO-CTWI-4 and 300 ul of Additions Mix VO-CTWI-5 to 2250 ul of methanol into a 3 ml Mininert vial. The ICV standard is prepared from standards of a different manufacturer or different lot than the standards used for calibration.

7.4.3. ICV standards used for SIM mode calibrations can be prepared by further dilution of the working standards (7.4.2) or by purchasing individual standards (e.q. 1000 ug/mL 1,4-dioxane, SPEX Certiprep--#S175 or equivalent). For t-butyl alcohol and 1,2-dioxane the working ICV/Spiking standard concentration is 100 ug/ml.

7.5. Certified Internal Standards (ISTD) and Surrogate Standards (SSTD):

7.5.1. ISTD/SSTD Mix (2500µg/mL): Ultra Scientific catalog # STM-540 or equivalent, stored in the Volatiles Standards Freeze at ≤ 10°C.

7.5.2. SSTD [2] 1,2-Dichlorobenzene-d_4 (2000µg/mL): Ultra Scientific catalog # STS-210 or equivalent, stored in the Volatiles Standards Freezer at ≤ 10°C.

7.5.3. A 20µg/mL ISTD/SSTD Working Standard is prepared by adding 200µL of ISTD/STD Mix and 250µL of SSTD 1,2-Dichlorobenzene-d_4 to 25mL of MeOH.

7.6. Certified Internal Standards (alternative to section 7.5 and used only with the Archon autosamplers):

7.6.1. (ISTD Mix--2500 ug/ml, Restek--#30241 or equivalent), stored in VOC Standards Freezer in Volatiles laboratory at ≤ -10 °C.

7.6.2. An 80 ug/ml ISTD working standard is prepared by adding 320 ul of the ISTD mix to a 10 ml volumetric flask and brought to volume with methanol.

7.7. Certified Surrogate Standards (alternative to section 7.5 and used only with the Archon autosamplers):

7.8.

7.8.1. (SSTD Mix—2500 ug/ml, Restek--#30240; 1,2-DCA-d_4 --2000 ug/ml, Ultra Scientific--#STS210, or equivalents[2]), stored in VOC Standards Freezer in Volatiles laboratory at ≤ -10 °C.

7.8.2. A 100 ug/ml SSTD working standard is prepared by adding 120 ul of the SSTD mix and 150 ul of 1, 2-DCA-d_4 to 2730 ul of methanol in a 3 ml mininert vial.

7.8.3. An 80 ug/ml ISTD/SSTD working standard is prepared by adding 320 ul of the ISTD Mix (sec. 10.5), 320 ul of the SSTD Mix (sec. 10.6), and 400 ul of 1,2-DCA-d_4 (sec. 10.6) to a 10 ml volumetric flask and brought to volume with methanol.

7.8.4. A 16 ug/ml ISTD/SSTD working standard is prepared by adding 2 ml of the 80 ug/ml ISTD/SSTD (sec. 10.6.2) to a 10 ml volumetric flask and brought to volume with methanol.

7.9. Certified Tuning Standard:

7.9.1. 4- bromofluorobenzene {BFB} (Ultra Scientific—2000 ug/ml, #STS-110N or equivalent), stored in VOC Standards Freezer in Volatiles laboratory at ≤ -10 °C.

7.9.2. A 50 ug/ml working standard is prepared by adding 75 ul of the certified standard to 2925 ul of methanol in a 3 ml mininert vial.

7.10. Sodium bisulfate (JT Baker--#3534-01 or equivalent), stored in cabinet in Volatiles laboratory.

7.11. All certified stock standards use the expiration date provided by the manufacturer/supplier.

7.11.1. The working standards (not including gases) expire one month after preparation. These standards include the BFB, ISTD, SSTD, and/or ISTD/SSTD.

7.11.2. The working standards (which include gases) expire one week after preparation. These standards include the ICV and CCV. When standards used for calibration are prepared from freshly open stock standard vials, the expiration of working standards used from that point on can be extended if the integrity of those standards can be confirmed and documented. For example, if a CCV/ICV standard continues to produce acceptable results after one week from preparation, it can be assumed still valid.

[1] Due to lower response or purging efficiencies, a number of compounds are purchased and prepared at concentrations greater than 100 ug/ml. Those compounds and concentrations are noted on the calibration curve.

[2] This surrogate compound is needed for Method 524.2 and is not used for this method/SOP.

8. Sample Handling and Preservation.

8.1. Water samples are stored at 0-6°C. The sample storage area must be free of organic solvent vapors and direct or intense light. Samples are stored in the Volatiles lab in a double door refrigerator (separate from analytical standards).

8.1.1. Analyze properly preserved samples (pH <2) samples within 14 days of collection. Samples not analyzed within this period must be discarded and recollected. If samples are not preserved then they must be noted (or qualified) as improperly preserved if not analyzed within 7 days.

8.1.2. Samples analyzed for Acrolein and Acrylonitrile are to be preserved at a pH of 4-5 and analyzed within 14 days (3 days if unpreserved).

8.1.3. If reactive compounds such as 2-Chloroethyl vinyl ether are target compounds than no preservatives are added and the sample needs to be analyzed as soon as possible.

8.1.4. Samples containing residual chlorine require alternative preservation (ascorbic acid or sodium thiosulfate) to reduce the chlorine. These sample shall be reduced to a pH of <2 (using HCL or $NaHSO_4$) to meet the 14 day is the hold time.

8.2. Soil samples are stored at 0-6° C. The sample storage area must be free of organic solvent vapors and direct or intense light. Samples are stored in a double door refrigerator located in the laboratory warehouse.

8.2.1. Samples received for low level analysis in "Encore" samplers must be preserved within 48 hours from time of collection. To preserve a sample, weigh it into a 40 ml VOA vial, record the weight, and then add 0.2 grams of sodium bisulfate per 1.0 gram of sample. Finally add 5.0 ml of DI H2O and a stir bar. Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and recollected.

8.2.2. Samples received for low level analysis in "Terra Core" sampler vials are already preserved with bisulfate at 0.1 g per 1.0 gram of sample. Samples that are received for low level analysis in DI water are placed in a freezer at ≤-10° C. The pre-weighed vial weight (tare weight which includes the weight of vial + 5 ml of preservative/DI water & a stir bar) is subtracted from the total weight of the vial to determine sample weight. Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and recollected.

8.2.3. Samples received in filled 2 oz. or 4 oz. jars can be weighed and prepared for low level analysis as described in section 11.2.1. or they can be weighed into a VOA vial and preserved at a 1:1 ratio with methanol for medium/high- level analysis. Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and recollected.

8.2.4. Samples collected and preserved with methanol in the field in pre-weighed 60-ml jars are weighed as is. The pre-weighed jar weight, as well as the methanol weight (19.8 grams for 25 ml of methanol) is subtracted from the total weight of the jar to determine sample weight. For Wisconsin LUST samples if the weight to volume ratio is more than 1:1 then methanol is added using the auto-pipetter to correct the ratio to 1:1. Unless instructed by the client to do otherwise, the maximum acceptable weight for volume correction is 35 grams. If samples are being analyzed for the Wisconsin LUST program then the hold time is 21 days from collection. Otherwise hold time is 14 days.

8.2.5. Samples collected and preserved in the field using "Terra Core" sample vials are weighed as is. The pre-weighed jar weight (tare weight which includes weight of the vial + MeOH) is subtracted from the total weight of the vial to determine sample weight. Unless instructed to do so samples are not adjusted for volume to weight differences. Analyze sample within 14 days from collection.

8.2.6. All soil samples are weighed on the top loading balance which is connected to a computer so that all weights can be automatically entered onto an Excel spread sheet. The Excel spreadsheet is set up to record the weights as well as calculate the methanol to weight differences. The spreadsheets are saved so the data can be transferred electronically to the LIMS system. See forms FVO4-(2-7) for examples of the sample weight spreadsheets.

8.2.7. Each prepared methanol soil sample is then placed on a shaker table for 2 minutes and then sonicated for 20 minutes prior to preparation for analysis.

8.3. Most samples received are accompanied with a Trip Blank (TB). In most cases the TBs are prepared by the lab and are sent along with the vials used for sample collection. The intent of the TB is to accompany the sample vials through all collection, preservation, shipping, and storage procedures. The infusion of outside contamination in the TB is not common, but can be an indicator of incorrect preparation/sampling procedures or inadequate sample storage.

9. PROCEDURE

9.1. Prior to sample analysis a GC/MS tune and calibration check must be analyzed. Verify the MS tune and initial calibration at the beginning of each 12-hour work shift during which analyses are performed.

9.1.1. Introduce into the GC (by direct injection) 25 to 50 ng of BFB and acquire a mass spectrum that includes data for m/z 35-260. If the spectrum does not meet all criteria, the MS must be retuned and adjusted to meet all criteria before proceeding with the continuing calibration check.

9.1.2. The calibration curve integrity for each analyte must be confirmed with the use of a CCV standard once every 12 hours of analysis time. The CCV standard is prepared at concentrations near the midpoint of the calibration curves (10/100 ug/L for water-5 ml purge, 4.0/40 ug/L for water-25 ml purge, 0.010/0.10 mg/kg for low level soils, and 0.50/5.0 mg/kg for MeOH preserved soils). The QSM recommends the CCV's to be varied throughout an Analytical run. Typically the concentrations used are 10, 20, and 30 ppb for Low Level soil and water-5 ml purged; 0.5, 1.0 1.5 mg/kg for MeOH preserved soil; and 1.0, 2.0, and 4.0 ppb for water-25 ml purged (preparation procedures are the same as listed 14.1.3, using the appropriate amount of the 100/1000 ug/ml CCV Std).

9.1.3. QSM 5.0 states a CCV must be analyzed at the beginning of the sequence, every 12 hours, and at the end of each analytical batch. The criteria for the ending CCV is all targets analytes within 50%. If a compound in the ending CCV fails, then 2 additional CCV's may be analyzed within a 60 minute timeframe. If both CCV's have acceptable recoveries, then the data can be reported without qualification.

9.1.4. The CCV is placed on the autosampler in the same manner as the samples (sec. 14.2.3). Preparation of CCV's is as follows:

9.1.4.1. **Water (5 ml purge)** -- Spike 50 ml of DI water (volumetric flask) with 5.0 ul of the 100/1000 ug/ml CCV/Calib. standard, invert three times and transfer to a VOA vial for analysis.

9.1.4.2. **Water (25 ml purge)** -- Spike 50 ml of DI water (volumetric flask) with 2.0 ul of the 100/1000 ug/ml CCV/Calib. standard, invert three times and transfer into a VOA vial for analysis.

9.1.4.3. **Low-level Soils** -- Spike 50 ml of DI water(volumetric flask) with 5.0 ul of the 100/1000 ug/ml CCV/Calib. standard, invert three times and transfer 5.0 ml into a VOA vial (containing and a stir bar) for analysis. As an alternative, prepare a 10.0/100 ug/ml working standard, then add 5.0 ul of this to 5.0 ml of DI water and transfer into a VOA vial (containing and a stir bar) for analysis.

9.1.4.4. **Med/high-level soils --** Spike 49 ml of DI water (volumetric flask) with 1.0 ml of MeOH and 5.0 ul of the 100/1000 ug/ml CCV/Calib. standard, invert three times and transfer into a VOA vial for analysis.

9.1.5. Each of the most common target compounds in the CCV should meet the minimum RFs as noted in Table 4. This is the same check that is applied during the initial calibration (sec. 13.4). If the minimum RFs are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins.

9.1.6. All target compounds of interest must be evaluated using a 20% variability criterion. Use percent deviation when performing the ARF model calibration. Use percent drift when calibrating using a regression fit model. If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large number of compounds that are

analyzed by this method, it is expected that some compounds will fail to meet the criterion. In cases where compounds fail, they can still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations where the failed compound is present, the concentrations must be reported as estimated values.

9.1.7. The internal standard responses and retention times in the CCV standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards changes by a more than a factor of two (-50% to +100%), when compared to the average from the calibration, then the mass spectrometer must be inspected for malfunctions and corrections must be made. Reanalysis of CCV's and associated samples while the system was malfunctioning is necessary.

9.1.8. Samples can be directly injected after the successful analysis of the initial calibration curve, ICV, BFB, and CCV. There can be up to 20 samples in an analytical batch. A MS/MSD and LCS must be analyzed with every analytical batch. Recoveries shall be compared to laboratory generated QC limits or client specified limits for all surrogate, MS/MSD and LCS injections.

9.2. Sample Introduction and Purging.

9.2.1. BFB tuning criteria and daily GC/MS calibration criteria must be met before analyzing samples. Currently 18-25 (depending on the temperature program used) purged samples including QC can be analyzed within 12 hours of the BFB injection. The Archon or the Centurion autosampler can be programmed to accommodate the number of samples needed per analytical shift.

9.2.2. After the continuing calibration is verified, the system must be proven to be free of contamination by analyzing a MB. The MB shall not contain detects above the detection limits for any given compound. Some programs allow detects up to but not exceeding one half the MRL. If the MB contains detects above the detection limits or RL's, then corrective actions must be performed to ensure the system is free from contamination; all affected samples shall also reanalyzed. The MBs are also placed on the autosampler in the same manner as the samples. For QSM, 5.0 common contaminants must not be detected above the LOQ.

9.2.3. Analysis of samples begins by allowing the sample to come to ambient temperature prior to analysis. The VOA vials containing the water samples are placed on the autosampler where a 5.0 to 25.0 ml aliquot is withdrawn from the vial and added into the appropriate purge vessel. The same procedure is followed for methanol preserved soils (1.0 ml of soil extract/49.0 ml DI H_2O is prepared and added into a 40 ml VOA vial prior to adding the samples to the autosampler). Low level soils are prepared by adding the VOA vial containing a magnetic stir bar and ≈5 g sample/5.0 ml DI H_2O to the autosampler (2-5 grams of sample is required for low-level analysis). The autosampler then adds an additional 5.0-mL of H_2O containing the ISTD/SSTD mixture. The sample is heated to 40°C and purged in the VOA vial while being stirred, and the volatiles are collected onto the trap.

9.2.3.1. The ISTD/SSTD is added automatically by the Archon or the Centurion autosampler as the sample is transferred from the 40 ml sample vial to the sparge tube; the exception is for low level soils as noted above.

9.2.3.2. The sample is purged for 11 minutes at 32°C for waters and MeOH-preserved soils and 40°C for low-level soils using helium or nitrogen with a flow of 35-40 ml/min.

9.2.3.3. During the 11-minute purge time, the purge able volatile organics are adsorbed onto the Supelco Carbosieve K trap.

9.2.3.4. During desorption the trapped materials are rapidly heated while back-flushing the trap with helium or nitrogen at 35-40 ml/min. for 1 minute at 260°C and introduced in the GC/MS. After the valve to the GC is closed the trap is then baked and back flushed with helium for ≈8 minutes at 265°C.

9.2.3.5. The GC is temperature programmed at 32°C for 2.5 minutes, then ramped to 165°C at 10°C/min, and finally ramped to 220°C at 15°C/min. The column flow is set at 1 ml/min. constant flow using helium as the carrier gas.
The transfer line to the MS is maintained at 250°C and the ion source is maintained at ≈260°C while under constant vacuum. The GC injector is set at 200°C.

Note: Samples suspected of containing high levels of contamination or samples with known historical data may need to be diluted prior to analysis. Multiple

dilutions may be needed to cover the entire working range of the current calibration

9.2.4. For each sample batch a MS, MSD, and LCS is prepared and analyzed. The concentrations for water spikes are 10.0/100 ug/L for 5 ml purge and 4.0/40.0 ug/L for 25 ml purge. The spiked concentrations for soil samples are ≈0.010/0.10 mg/kg for low level and ≈0.50/5.0 mg/kg for MeOH preserved depending on sample weights and percent solids. One exception is for the analysis of samples with low sample volume. These samples may be analyzed with a LCS and a LCSD upon client request. All spikes are transferred into 40 ml VOA vials and added to the autosampler. The spike concentrations may vary depending on program/project specific criteria, but the preparation volumes are constant and only the spiking amount changes. 14.2.4.1 lists examples of spike preparation based on the concentrations above.

9.2.4.1. The preparation of the matrix spikes is performed as follows:

9.2.4.1.1. **Water (5 ml purge)**--Spike 40 ml of sample with 4.0 ul of the 100/1000 ug/ml CCV standard, invert three times and transfer to a VOA vial for analysis. As an alternative the sample VOA vial may be spiked with 4.2 ul of the 100/1000 ug/ml CCV standard. When adequate sample amounts are not provided, one 40 ml aliquot of sample is spiked and split into two separate VOA vials containing 15 ml glass inserts.

9.2.4.1.2. **Water (25 ml purge)**-- Spike 50 ml of sample with 2.0 ul of the 100/1000 ug/ml CCV standard, invert three times and transfer into a VOA vial for analysis. Alternatively, A MS/MSD can be prepared by spike ~1.6 ul of the 100/1000 ug/ml CCV standard directly into the sample vial. The sample is then inverted three times and then placed on the autosampler for analysis.

9.2.4.1.3. **Low-level Soils**--Spike 50 ml of DI water(volumetric flask) with 5.0 ul of the 100/100 ug/ml CCV standard, invert three times and transfer 5.0 ml into a VOA vial containing ≈5 g of sample and a stir bar for analysis. As an alternative, prepare a 10.0/100 ug/ml working standard, then add 5.0 ul of this to 5.0 ml of DI

water and transfer into a VOA vial containing ≈5 g of sample and a stir bar for analysis.

9.2.4.1.4. **Med/high-level soils--**Spike ≈10 g of sample contained in a VOA vial with 50 ul of the 100/1000 ug/ml CCV standard. Add 9.95 ml of methanol to the spiked sample and sonicate for 20 minutes. Add 1.0 ml of methanol extract to 49.0 ml DI water in a 50 ml syringe and then transfer into a VOA vial for analysis. For samples that are MeOH preserved in the field, take 1.0 mL of sample into 49 mL of DI water and add 5.0 ul of the 100/1000 ug/ml CCV standard, invert three times and transfer to a VOA vial for analysis.

9.2.4.2. The preparation of a LCS is performed as follows:

9.2.4.2.1. **Water (5 ml purge)**--Spike 50 ml of DI water (volumetric flask) with 5.0 ul of the 100/1000 ug/ml CCV standard, invert three times and transfer to a VOA vial for analysis. The LCS and the CCV may be run as a single analysis.

9.2.4.2.2. **Water (25 ml purge)**-- Spike 50 ml of DI water (volumetric flask) with 2.0 ul of the 100/1000 ug/ml CCV standard, invert three times and transfer into a VOA vial for analysis. The LCS and the CCV may be run as a single analysis.

9.2.4.2.3. **Low-level Soils**--Spike 50 ml of DI water(volumetric flask) with 5.0 ul of the 100/1000 ug/ml CCV standard, invert three times and transfer 5.0 ml into a VOA vial containing 5 g of control and a stir bar for analysis. As an alternative, prepare a 10.0/100 ug/ml working standard, then add 5.0 ul of this to 5.0 ml of DI water and transfer into a VOA vial containing 5 g of control sand and a stir bar for analysis.

9.2.4.2.4. **Med/high-level soils--**Spike 10 g of control sand contained in a VOA vial with 50 ul of the 100/1000 ug/ml CCV standard. Add 9.95 ml of methanol to the spiked sand and sonicate for 20 minutes. Add 1.0 ml of methanol extract to 49.0 ml DI water in a

50 ml syringe and then transfer into a VOA vial for analysis.

9.2.5. The data is collected by the Chemstation software using the RFs (or linear/second order regressions when necessary), and results are calculated using the internal standard method of quantitation. Response factors for each detected compound are compared with that obtained in calibration, and based on those comparisons, results are generated. Software manuals define the procedures for creating and understanding a specific method (Understanding Your Chemstation, Hewlett Packard, G2070-90100, October, 1994, Environmental Forms Software, Hewlett Packard, G1032-90021, November, 1992, and Productivity Enhancement Software for HP Chemstation, Prolab Resources Inc., XMS01A-002, Rev. G, 2001).

10. CALCULATIONS, DATA ANALYSIS AND REDUCTION.

10.1. The Chemstation software (using response factors) calculates the initial concentration (or raw result) of target compounds as follows:

10.1.1. **Liquids**

$$\text{Initial Concentration (ug/L)} = \frac{A_x \times I_{is}}{A_{is} \times RF}$$

Where:
A_X = Area of characteristic ion for compound being measured in the sample.
I_{IS} = Amount of internal standard injected (ug/L). Typical concentrations used are 20.0 ug/L for 5.0 ml purge, and 4.0 ug/L for 25.0 ml purge
A_{IS}= Area of characteristic ion for the internal standard.
RF = Response factor for compound being measured.

10.1.2. **Solids**

$$\text{Initial Concentration (ug/L)} = \frac{A_x \times I_{is}}{A_{is} \times RF}$$

Where:
A_X = Area of characteristic ion for compound being measured in the sample.
I_{IS} = Amount of internal standard injected (ug/L). Typical concentrations used are 20.0 ug/L for 5.0 ml purge, and 4.0 ug/L for 25.0 ml purge
A_{IS}= Area of characteristic ion for the internal standard.

RF = Response factor for compound being measured.

10.2. The Chemstation software (using linear regression) calculates the initial concentration of target compound as follows:

Response Ratio = slope * amount ratio + intercept
Where: Response Ration = response of target compound/response of associated ISTD.
Amount Ratio = target compound concentration/associated ISTD concentration.

Example: Tr / ISr = m * Tc /ISc + b

Where: Tr = response of target compound
ISr = Internal Standard Response
M = slope of the curve (for the target compound)
ISc = Internal Standard Concentration
Tc = Target compound concentration
B = y-intercept of the curve (for the target compound)

Solve for "Tc"

10.3. The initial concentration results are then transferred to the laboratory's LIMS system where the final concentrations are calculated.

10.3.1. The final concentration for water samples is calculated as follows:

Final Concentration (ug/L) = Initial concentration x Dilution Factor

10.3.2. The final concentrations for low-level and med/high-level soils are calculated by the following equation:

Final concentration (mg/kg) =

$$\frac{\text{Initial concentration x Sample Volume x Dilution factor}}{\text{Sample weight x \% solids}}$$

Where:

Sample volume = 5.0 mL for low –level soils, or volume of MeOH used for med/high-level soils preservation.
Sample weight = grams of sample in VOA vial for low level soils, or total grams of sample preserved for med/high-level soils.
% Solids = fraction equivalent (e.g. 97.1% = 0.971)

10.4. The spike percent recoveries (%R) and relative percent differences (RPD) are calculated in LIMS as follows:

10.4.1. Liquids – Concentration of spike added:

$$\text{ug/L} = \frac{\text{mL of spike added x concentration of spiking standard}}{\text{mL of sample (or DI } H_2O\text{) spiked}} \times 100$$

10.4.2. Solids – Concentration of spike added:

$$\text{mg/kg} = \frac{\text{mL of spike added x concentration of spiking standard}}{\text{grams of sample (or control sand) spiked}}$$

10.4.3. Final Calculations:

$$\text{MS/MSD \%R} = \frac{\text{Concentration of spike obtained - concentration of sample obtained}}{\text{Concentration of spike added}} \times 100$$

$$\text{MS/MSD RPD} = \frac{\text{[Concentration MS – Concentration MSD]}}{(\{\text{Concentration MS + concentration MSD}\}/2)} \times 100$$

$$\text{*LCS \% R} = \frac{\text{Concentration of Spike obtained}}{\text{Concentration of Spike added}} \times 100$$

Notes:

--Concentrations (conc.) of samples, MS/MSD, and LCS spikes are obtained directly from calibration curve.
--Soil spike concentrations and recoveries are calculated on a dry weight basis.
-- [ ] Signifies absolute values
--* Equation can also be used to calculate surrogate recoveries

11. Calibration and Standardization

11.1. To facilitate appropriate separation and provide adequate sensitivity, the entire operating system must be correctly set up and maintained before calibration and analyses can occur. Proper settings and programming of the GC/MS volatile system greatly increase the likelihood that calibrations will be acceptable. Generating and reproducing results will also be affected favorably in a well-maintained system.

11.1.1. The following tables provide instrument settings for the daily use of the Archon/Encon or Centurion/ Encon Purge and Trap Systems. Any modifications are noted in the specific instrument's maintenance log:

PARAMETER SETTINGS FOR ENCON EVOLUTION	
Trap Ready Temp.	≈35° C
Mort Ready Temp.	≈39° C
Purge Flow	40 ml/minute
Purge Time	11.00 minutes
Dry Purge Time	2.00 minutes
Desorb Preheat	255° C
Desorb Temp.	1.00 minutes at 260 ° C
Trap Bake Temp.	265° C
MoRT Bake Temp.	235 ° C
Bake Flow Rate	45 ml/minute
Gas	Helium or Nitrogen
Sample Purge Temp.	32 ° C
Sample Bake Temp.	75-90 ° C
Valve and Line Temp.	150 ° C

11.1.2. An example of the GC temperature program for the SPB/DB/ZB-624 columns used for the analysis of samples is as follows:

Start temp °C	End temp °C	Rate °C/minute	Time minutes
32	32	0.0	2.5
32	165	10.0	0.00
165	220	15.0	1.00

11.1.3. The injector is a split/split less injector operated in split mode ranging from 1:10 to 1:60. The injector temperature is 200 °C.

11.1.4. The MS detector parameters are subject to change to achieve optimum chromatography. See instrument maintenance logbook for recent changes regarding source maintenance, as well as filament and multiplier replacements. Current tune values and EM voltage settings are documented and can be found in the appropriate instrument's tuning logbook.

11.1.5. 4-Bromofluorobenzene (BFB) Standard:

11.1.5.1. A standard solution containing 50 ug/ml is used for the daily tune check. The BFB is directly injected onto the column in 25 to 50 ng injections (0.5 to 1.0 ul).

11.1.5.2. The GC/MS system tune must be verified at the beginning of any calibration or a sequence run and verified every 12 hours thereafter. The tuning compound is BFB which is injected directly onto the GC column The software is set up to check the tune by using the mean of three scans across the apex. Background subtraction is performed using a single scan no more than 20 scans prior to the elution of BFB. Manual scans can be checked by taking an average of scans across the BFB peak. The tuning acceptance criteria are listed below (m/z range 35-260):

Mass (m/z)	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	<2% of mass 174.
174	>50% of mass 95.
175	5 to 9% of mass 174.
176	>95% but <101% of mass 174.
177	5 to 9% of mass 176.

11.1.6. The preparation of working standards is routinely performed each week unless integrity is shown to be intact. All standards are assigned a unique identification number and preparations are documented in a Standards Logbook.

11.1.6.1. Calibration Standards - Calibration standards are prepared at a minimum of five concentration levels (in most instances, eight levels are currently used) and are prepared from the working standard dilutions of stock standards. One of the concentration levels shall be at a concentration near, but above, the detection limit and at or below the reporting limit. The remaining concentration levels shall correspond to the expected range of concentrations found in real samples and shall contain each analyte for detection by this method. For low-level soil calibrations, sodium bisulfate is added at a 0.2 g/1.0 g sample to the water to match sample matrix /acidity if the samples were collected and preserved with sodium bisulfate. Med/high-level soil calibrations have MeOH added at a 0.1 ml/5.0 ml H2O to match sample matrix/preservation. All final concentrations are brought to volume with DI water. The following tables outline the preparation calibration curves for water and soil samples (all calibration standards are transferred into 40 ml VOA vials for placement on the auto sampler):

1) Waters Curve (5.0 ml Purge)

Concentration (ug/L)	Amount added of the 100 ug/ml CCV/Calib. Std. (in ul)	Final Volume (ml) (Volumetric flask)
0.5	1.0	200
2.0	2.0	100
5.0	5.0	100
10.0	10.0	100
20.0	20.0	100
30.0	30.0	100
40.0	40.0	100
80.0	80.0	100

2) Waters Curve (25.0 ml Purge)

Concentration (ug/L)	Amount added of the 100 ug/ml CCV/Calib. Std. (in ul)	Final Volume (ml) (Volumetric flask)
0.1	0.5	500
0.5	1.0	200
1.0	1.0	100
2.0	2.0	100
4.0	4.0	100
6.0	6.0	100
8.0	8.0	100
16.0	16.0	100

3) Low Level Soils Curve

Concentration (mg/kg)	Amount added of the 100 ug/ml CCV/Calib. Std. (in ul)	Grams of sodium bisulfate added (if needed)	Final Volume (ml) (Volumetric flask)
0.001	1.0	1.0	100
0.002	2.0	1.0	100
0.005	5.0	1.0	100
0.010	10.0	1.0	100
0.020	20.0	1.0	100
0.030	30.0	1.0	100
0.040	40.0	1.0	100
0.080	80.0	1.0	100

4) Medium/High Soils Curve

Concentration (mg/kg)	Amount added of the 100 ug/ml CCV/Calib. Std. (in ul)	ul of MeOH added	Final Volume (ml) (Volumetric flask)
0.050	1.0	999	100
0.100	2.0	998	100
0.250	5.0	995	100
0.500	10.0	990	100
1.000	20.0	980	100
1.500	30.0	970	100
2.000	40.0	960	100
4.000	80.0	920	100

5) Waters Curve (5.0 ml Purge-SIM)

Concentration (ug/L)	Amount added of the 100 ug/ml CCV/Calib. Std. (in ul)	Final Volume (ml) (Volumetric flask)
1.0	1.0	100
2.0	2.0	100
5.0	5.0	100
10.0	10.0	100
20.0	20.0	100
40.0	40.0	100
80.0	80.0	100

6) Medium/High Soils Curve (SIM)

Concentration (mg/kg)	Amount added of the 100 ug/ml CCV/Calib. Std. (in ul)	ul of MeOH added	Final Volume (ml) (Volumetric flask)
0.050	1.0	999	100
0.100	2.0	998	100
0.250	5.0	995	100
0.500	10.0	990	100
1.000	20.0	980	100
2.000	40.0	960	100
4.000	80.0	920	100

7) Low Level Soils Curve (SIM)

Concentration (mg/kg)	Amount added of the 100 ug/ml CCV/Calib. Std. (in ul)	Grams of sodium bisulfate added (if needed)	Final Volume (ml) (Volumetric flask)
0.001	1.0	1.0	100
0.002	2.0	1.0	100
0.005	5.0	1.0	100
0.010	10.0	1.0	100
0.020	20.0	1.0	100
0.040	40.0	1.0	100
0.080	80.0	1.0	100

11.1.6.2. Internal Standards - The internal standards used are Chlorobenzene-d_5, 1,4-Difluorobenzene, 1,2-Dichloroethane-d_4 and Fluorobenzene (sec. 7.6.1). Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS.

11.1.6.3. Surrogate Standards - The surrogate standards used are Toluene-d_8, 1,2-Dichloroethane-d_4, 4-Bromofluorobenzene,

and Dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. The 100 ug/ml working surrogate standard (sec. 7.7.2) may be used for calibration added at the same concentrations as the target compounds (see above).

11.1.6.4. ISTD/SSTD Combined Standard - A combination of internal standard and surrogate standard at 80ug/ml (sec. 7.7.3), or 20 ug/ml for Centurion auto-sampler (sec. 7.5.3) , is automatically added by the autosampler to all calibration levels, samples, blanks, CCV's and spikes used for any given sequence (actual volume for the archon autosamplers is determined by an ISTD/SSTD study and is documented in the maintenance logbooks for each instrument). Limits are generated internally or project/program limits are used.

11.1.6.5. Calibration curves are prepared fresh from newly made working standards to ensure accurate concentrations are maintained.

11.1.6.6. Secondary dilution standards (when necessary) - secondary dilution standards containing the compounds of interest (usually at 10.0/100 ug/ml) for SIM, low level, and MDL analyses may be prepared in methanol and stored with minimal headspace and shall be checked frequently for degradation. They are to be stored for one week only.

11.1.6.7. Preparation of standards is documented in the Volatile standards logbook. Each standard solution is documented with the standard name, concentration, preparation date, expiration date and a unique number given to that standard for future traceability.

11.2. The curve is generated using the relative response factor (RRF or RF). The data system tabulates the area response of the characteristic ions against the concentration of each compound and each internal standard. Calculate RFs for each compound relative to one of the internal standards. The internal standard selected for the calculation for the RF for a compound is the internal standard that has a retention time closest to the compound being measured.

11.2.1. The RF is calculated by the data system as follows:

$$RF = \frac{A_S \times C_{IS}}{A_{IS} \times C_S}$$

Where:

A_s = Area of the characteristic ion for the compound being measured in the calibration standard.
A_{IS} = Area of the characteristic ion for the specific internal standard.
C_{IS} = Concentration of the specific internal standard.
C_s = Concentration of the compound being measured in the calibration standard.

11.2.2. The average response factor (ARF) for all calibration levels is used when determining sample concentration and is calculated (along with the standard deviation) to evaluate the linearity of the curve (SW-846 Method 8000C. Sec. 11.5.1).

11.3. When ARFs are not acceptable, results are sometimes calculated using linear (1^{st} order) regression curves and/or quadratic (2^{nd} order) curves. Internal standard quantitation is also used when generating linear and non-linear calibrations. All equations and acceptance criteria follow the examples in SW-846, Method 8000C (sec. 11.5.2 and sec. 11.5.3).

11.4. If the RSD of the RFs is less than 20%, then the RF is assumed to be constant over the calibration range, and the average response factor may be used for quantitation. If the RSD of any analyte or surrogate mean RF exceeds 20% than linear regression or second order curves may be used for quantitation.

11.4.1. Linear Calibration: If the RSD of the calibration factor is greater than 20% over the calibration range, then the linearity through the origin cannot be assumed. If this is the case, the analyst can employ a regression equation that does not pass through the origin. This approach can also be employed based on the past experience of the instrument response.

11.4.2. The use of origin (0,0) as a calibration point is not allowed. However, most data systems and many commercial software packages will allow the analyst to “force” the regression through zero. This is not the same as including the origin as a fictitious point in the calibration. It can be appropriate to force the regression through zero for some calibrations (SW-846 Method 8000C sec. 11.5.2.1). The use of linear regression cannot be used as a rationale for reporting results below the calibration range.

11.4.3. The method of linear regression analysis has a potential for a bias to the lower portion of a calibration curve. If linear regression is used,

then the lowest point in the calibration curve is calculated using the new curve. The recalculated concentration of the low calibration point should be within +/- 30% of the standard's true concentration.

11.4.4. Non-Linear Calibration: In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model can be employed. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range. The model chosen shall have no more than four parameters, i.e., if the model is polynomial, it can be no more than third order.

11.4.4.1. The statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approaches described above. Linear regression employs five calibration standards for the linear model; a quadratic model requires a minimum of six calibration standards.

11.4.4.2. Under ideal conditions, with a "perfect" fit of the model to the data, the coefficient of the determination (COD) will equal 1.0. In order to be an acceptable non-linear calibration, the COD must be greater than or equal to 0.99 Weighting in a calibration model can significantly improve the ability of the least squares regression to fit the data calibrations (SW-846 Method 8000C sec. 11.5.3).

11.5. Each of the most common target compounds ARFs in the ICV and the CCV should meet the minimum RF as noted in Table 4.

11.5.1. A number of compounds (primarily the ketones) do not respond well at normal concentrations, especially for low level analyses, resulting in RF's below the minimum requirement. These compounds are purchased at concentrations 10x the normal concentration to ensure adequate responses for working calibrations. Other poor responding compounds are commonly requested to be analyzed by this procedure and are purchased at concentrations that best ensure adequate responses to achieve successful calibrations.

11.5.2. If the minimum response factors are not met, the system should be evaluated, and corrective action should be taken before sample analysis begins. Examples of possible occurrences are as follows:

	Comment
Chloromethane	This compound is the most likely compound to be lost if the purge flow is too fast.
Bromoform	This compound is one of the compounds most likely to purge poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.
1,1,2,2-Tetrachloroethane	The response of this compound is degraded by contaminated transfer lines in purge and trap systems and/or active sites in trapping materials.
1,1-Dichloroethane	The response of this compound is also degraded by contaminated transfer lines in purge and trap systems and/or active sites in trapping materials.

11.6. All calibrations are confirmed by the analysis of a "second source" ICV standard before daily checks and analyses are performed. The RSD limit for all target compounds is ±20%, unless specified differently by any other applicable program or project's criteria (QSM: ±25% RSD for all analytes). If these criteria are not met and a reanalysis of the ICV confirms the nonconformities, then corrective actions must be taken and the instrument recalibrated. Any outliers suggest a problem and poor performers shall be addressed. The concentrations of the ICV are near the midpoint of the curve (10/100 ug/L for water-5 ml purge, 4.0/40 ug/L for water-25 ml purge, 0.010/0.10 mg/kg for low level soils, and 0.50/5.0 mg/kg for MeOH preserved soils). The preparation of ICV's is as follows:

Water (5 ml purge)--Spike 50 ml of DI water (volumetric flask) with 5.0 ul of the 100/1000 ug/ml ICV/Spiking standard, invert three times and transfer to a VOA vial for analysis.

Water (25 ml purge) -- Spike 50 ml of DI water (volumetric flask) with 2.0 ul of the 100/1000 ug/ml ICV/Spiking standard, invert three times and transfer into a VOA vial for analysis.

Low-level Soils--Spike 50 ml of DI water(volumetric flask) with 5.0 ul of the 100/1000 ug/ml ICV/Spiking standard, invert three times and transfer 5.0 ml into a VOA vial (containing and a stir bar) for analysis. As an alternative, prepare a 10.0/100 ug/ml working

standard, then add 5.0 ul of this to 5.0 ml of DI water and transfer into a VOA (containing a stir bar) for analysis.

Med/high-level soils-- Spike 49 ml of DI water (volumetric flask) with 1.0 ml of MeOH and 5.0 ul of the 100/1000 ug/ml ICV/Spiking standard, invert three times and transfer into a VOA vial for analysis.

11.7. An Initial Calibration Blank (ICB) is analyzed to confirm that the instrument is free from contamination. Any detects in the ICB shall be less than the method detection limit and/or less than ½ the program/project limits. Any detects above MDL or program limits must be addressed before sample analyses begin. To prepare an ICB fill a 40 ml VOA vial preserved with 5% HCL with DI water.

11.8. Demonstration and documentation of an acceptable initial calibration is required before any samples are analyzed. Refer to EPA SW-846, Method 8000B, Section 7, for a detailed discussion of calibration procedures.

12. QUALITY CONTROL

12.1. Method Performance

12.1.1. Certified standard solutions, properly maintained instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external performance test samples provided by the appropriately accredited agencies. Internal blind spikes are also utilized to check analyst performance.

12.1.2. Initial demonstration of capability (IDC) is another technique used to ensure acceptable method performance.

12.1.2.1. An analyst must demonstrate initial precision and accuracy through the analysis of 4-5 laboratory control spikes for each matrix and sample type. After analysis, the analyst calculates the average recovery (x) in μg/L and the relative standard deviation (RSD) of the recoveries for each analyte. In the absence of specific criteria found in the SW-846 methods or project specific limits, the default criteria of 70-130% recovery and 20 % RSD are used until internal limits are generated (Method 8000C, sec. 9.4.9)

12.1.3. Examples of the preparation of IDCs are as follows:

Water (5 ml purge)--Spike 50 ml of DI water (volumetric flask) with 5.0 ul of the 100/1000 ug/ml CCV/Calib. standard, invert three times and transfer to a VOA vial for analysis.

Water (25 ml purge) -- Spike 50 ml of DI water (volumetric flask) with 2.0 ul of the 100/1000 ug/ml CCV/Calib. standard, invert three times and transfer into a VOA vial for analysis.

Low-level Soils--Spike 50 ml of DI water (volumetric flask) with 5.0 ul of the 100/1000 ug/ml CCV/Calib. standard, invert three times and transfer 5.0 ml into a VOA vial containing 5 g of control and a stir bar for analysis. As an alternative, prepare a 10.0/100 ug/ml working standard, then add 5.0 ul of this to 5.0 ml of DI water and transfer into a VOA vial containing 5 g of control sand and a stir bar for analysis.

Med/high-level soils--Spike 10 g of control sand contained in a VOA vial with 50.0 ul of the 100/1000 ug/ml CCV/Calib. standard. Add 9.95 ml of methanol to the spiked sand and sonicate for 20 minutes. Add 1.0 ml of methanol extract to 49.0 ml DI water in a 50 ml syringe and then transfer into a VOA vial for analysis.

12.1.4. Many projects require the analysis of MRL standards and MDL check samples as another means of checking method performance. The MRLs are analyzed at the beginning and end of each 12 hour shift and are typically prepared at concentrations equal to the lowest standard on the calibration curve. Recovery limits are program specific but are usually set at 70-130%. The MDL check sample is usually spiked at approximately 2x the method detection limit. The MDL check sample is analyzed quarterly (as a minimum) to confirm instrument sensitivity (e.g. to verify that the method detection limits are still achievable). The MDL check samples are taken through all preparation and extraction steps used for actual samples (e.g. spiking/preserving control sand for soil samples). In most instances, a method detection limit check sample is analyzed at the end of each sequence requiring an MRL standard. The recovery criteria for MDL check samples are the ability to detect all compounds. If any given compound is not detected, the MDL check is spiked at a higher level and analyzed again. Detection limits for those compounds not detected on the initial MDL check analysis need to be raised to match the MDL check analysis at which they were detected.

12.1.5. Creating and monitoring control charts is also important for maintaining and improving method performance. Currently all SSTD, MS, MSD, and LCS recoveries are monitored with the use of the LIMS system. The

data collected is used to recognize trends in recovery performance, as well as for generating new in-house QC limits. Default accuracy limits of 70-130 % recovery and a precision limit 20 % RSD are used until enough data points are generated to provide usable internal limits. Other programs such as the WI UST program uses default accuracy and precision limits for surrogates and spikes of 80-120/20 %. Client and/or project specific limits are also used frequently in sample analyses. The Quality Control Requirements chart (Table 2.) also lists recovery limits specific to the method/project/program.

12.1.6. Performance Testing (PT's) must be done on all compounds on the list. If a compound is not available from a PT provider, the LCS studies must be performed and documented (at least 4 reps) twice a year to demonstrate proficiency.

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12.2. This SOP is designed to follow a variety of different projects and programs requirements. Table 2. is designed to illustrate the control steps and provisions required to adequately producing acceptable data.

12.3. Contract Specific Sample Analysis: For certain samples, limits are specified by the QAPP (Quality Assurance Project Plan) associated with a given project. For these samples follow the limits specified in the QAPP for that project.

12.4. Contract Specific Sample Analysis: For certain samples, limits are specified by the QAPP (Quality Assurance Project Plan) associated with a given project. For these samples follow the limits specified in the QAPP for that project.

13. DATA ASSESSMENT/ACCEPTANCE CRITERIA FOR QC MEASURES

13.1. If the initial analysis of a sample or a dilution of the sample has a concentration of a particular analyte that exceeds the calibration range, the sample must be reanalyzed at a dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a blank water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analyses can not resume until a blank can be analyzed that is free of interferences.

13.2. After the analysis of water samples, the pH shall be taken to verify proper field preservation. pH strips are used to verify the pH which is then documented in the bench sheet logbook.

13.3. Qualitative Analysis:

13.3.1. The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum (ion scans) after background correction with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated (by the laboratory) using the conditions of this method. The mass spectral library is updated with each new calibration and is continually updated with the mass spectra from CCV's.

13.3.2. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity or any ions over 30% relative intensity if fewer than three such ions occur in the reference spectrum. Table 3 lists compounds along with the Primary Ion (Quantitation ion) used for calculating results, and the Secondary Ions (Qualitative ions) used for qualitatively matching sample spectrums with reference spectrums for positive identifications. Compounds shall be identified as present when the criteria below are met.

13.3.2.1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

13.3.2.2. The relative retention time (RRT) of the sample component is within +/- 0.06 RRT units of the RRT of the standard component.

13.3.2.3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

13.3.2.4. Structural isomers that produce very similar mass spectra shall be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

13.3.2.5. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra

containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra are important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes co elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co eluting compound.

13.3.3. For samples containing compounds that are not a part of the normal target list, a library search may be required for the purpose of tentative identification. Tentative identified compounds (TICs) are needed only when requested or required by a particular project or program. Data system library search routines shall not use normalization routines that would misrepresent the library of unknown spectra when compared to each other. Use the following a guidance for reporting TICs.

13.3.3.1. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) shall be present in the sample spectrum.

13.3.3.2. The relative intensities of the major ions agree within ± 20%.

13.3.3.3. Molecular ions present in the reference spectrum shall be present in the sample spectrum.

13.3.3.4. Ions present in the sample spectrum but not in the reference spectrum shall be checked for possible background contamination. They shall also be reviewed for possible co elution with another compound.

13.3.3.5. Ions present in the reference spectrum but not in the sample spectrum shall be check against the possibility of subtraction from the sample spectrum due to background contamination or co-eluting peaks. Some data reduction programs can create these discrepancies.

13.4. Ions present in the reference spectrum but not in the sample spectrum shall be check against the possibility of subtraction from the sample spectrum due to

background contamination or co-eluting peaks. Some data reduction programs can create these discrepancies.

13.5. When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. Once the reference spectrums are compared and the sample spectrums and identifications have been made, the sample data can be reported. Assessments of all spiked and calibration control samples and standards shall also be finalized before reporting the data.

13.5.1. When the analyst has finished processing the analytical batch, the results are electronically transferred to the LIMS system where weight to volume corrections, dilution factors and percent solids adjustments are made. Once the final results have been verified, a checklist (FVO4-01) is filled out and signed confirming that all the data has been thorough scrutinized. At this point the data is turned over to another qualified analyst for final validation. The second analyst confirms the results and electronically marks them validated and signs his or her portion of the checklist. Finally, the validated results are made available to the client services personnel in order for the data to be given to the client or appropriate agencies.

13.5.2. A PDF copy of the data is then electronically filed and archived. The package includes the checklist, the sequence run log, a copy of the bench sheets, the LIMS run log, verification of tuning and system performance data, and verification of calibration data. For each sample, the chromatogram, quantitation and library spectra (ion scans) for all detected target compounds are also included. Each data file header shall contain the sample ID # and the date and time acquired.

14. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

14.1. See QAM Appendix 9.

15. CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

15.1. See QAM Appendix 9.

16. DATA RECORDS MANAGEMENT

16.1. Records are stored for a minimum of 5 years in accordance with the Quality Manual.

16.2. See SOP QA 003 for specifics on document control.

17. WASTE MANAGEMENT

17.1. See QAM Appendix 9.

18. REFERENCES

18.1. Determinative Chromatographic Separations, USEPA SW-846 Methods 8000C, Rev. 3, March, 2003

18.2. Volatiles Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), USEPA SW-846 Method 8260C, Rev. 3, August, 2006.

18.3. Purge and Trap for Aqueous Samples, USEPA SW-846 Methods 5030B, Rev. 2, December, 1996.

18.4. Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, USEPA SW-846 Methods 5035 (Inc. App. A), Rev. 0, December, 1996.

18.5. Wisconsin DNR, Lust Guidance, July, 1993.

18.6. USEPA, Method 603, Acrolein and Acrylonitrile, July, 1982.

18.7. CT Laboratories Quality Manual, current revision.

18.8. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010.

18.9. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013 or most recent revision.

18.10. National Environmental Laboratory Accreditation Conference (NELAC), 2003 NELAC Standard Chapters 1 to 6, EPA/600/R-04/003, June 5, 2003 or most recent version.

18.11. ISO. 2005. General requirements for the competence of testing and calibration laboratories. ISO17025.

18.12. Appendix A to part 136, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewaters, EPA method 624-Purgeable.

19. Attachments.

Table 1

Analyte List

Analyte	Analyte
Acetone Benzene Bromobenzene Bromochloromethane Bromodichloromethane Bromoform Bromomethane 2-Butanone n-Butylbenzene sec-Butylbenzene tert-Butylbenzene Carbon disulfide Carbon tetrachloride Chlorobenzene Chloroethane 2-Chloroethylvinyl ether Chloroform Chloromethane 2-Chlorotoluene 4-Chlorotoluene Dibromochloromethane 1,2-Dibromo-3-chloropropane 1,2-Dibromoethane Dibromomethane 1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene Dichlorodifluoromethane 1,1-Dichloroethane 1,2-Dichloroethane 1,1-Dichloroethene cis-1,2-Dichloroethene trans-1,2-Dichloroethene 1,2-Dichloropropane 1,3-Dichloropropane	2,2-Dichloropropane 1,1-Dichloropropene cis-1,3-Dichlropropene trans-1,3-Dichloropropene Diisopropyl ether Ethylbenzene Hexachlorobutadiene 2-Hexanone Isopropylbenzene p-Isopropyltoluene Methylene chloride 4-Methyl-2-pentanone Methyl tert butyl ether Naphthalene n-Propylbenzene Styrene 1,1,1,2-Tetrachloroethane 1,1,2,2-Tetrachloroethane Tetrachloroethene Tetrahydrofuran Toluene 1,2,3-Trichlorobenzene 1,2,4-Trichlorobenzene 1,1,1-Trichloroethane 1,1,2-Trichloroethane Trichloroethene Trichlorofluoromethane 1,2,3-Trichloropropane 1,2,4-Trimethylbenzene 1,3,5-Trimethylbenzene Vinyl chloride Vinyl acetate o-Xylene m/p-Xylene 112Trichloro122trifluoroethane

Table 2
Volatile Organic Compounds by GC/MS
Summary of Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Tune Check (BFB)	Beginning of Every 12 hours shift.	Ensure correct mass assignment. BFB % Relative abundance criteria as specified in method 8260 or use program/project specific criteria.	Retune. Do not proceed with analysis until tune meets criteria.
Initial Calibration (ICAL)	Each time the instrument is set up and when compounds in the continuing calibration verification (CCV) do not meet criteria. Established initially at minimum five concentration levels (six concentration levels if a second order {quadratic} curve is used) - low standard at or below project required reporting limit (PRRL), near but above method detection limits (MDL). Heated purge for low-level soils.	1. Average relative response factors (RRFs) for compounds on Table 4. 2. % RSD for RRFs for all target compounds ≤20%. IF RF % RSD >20% use linear curve, r =.995, r2 = .990. 4. LCG, NELAC, DoD-QSM, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents	Correct system and recalibrate. Criteria must be met before sample analysis may begin. Any samples reported from data not meeting these criteria must be qualified (Z).
Initial Calibration Verification standards (ICV)	After each initial calibration. Shall be at or near the mid-point of calibration range for all target compounds, and is prepared from second source standards. Typically use 10/100 ppb for H2O and Low Level Soils, 0.5/5.0 mg/kg for MeOH preserved soils. Two ICV's are required for 2Nd order quadratic curves (one below and one above the inflection point). Heated purges for low-level soils.	1. RRF for compounds on Table 4. 2. %RSD <20% Deviation for RRFs, <20 % Drift for linear and nonlinear curves 3. LCG, NELAC, DoD-QSM, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents (ex: ± 25% D. for QSM projects).	Correct system and recalibrate. Criteria must be met before sample analysis may begin. IF % relative standard deviation (RSD) >20%, then system must be inspected and problem corrected before sample analysis. If >20% RSD then confirm the integrity of the second source standard by reanalysis, and/or determine if it's a sporadic problem involving compounds that are typically poor performers. ACOE allows no tolerances for % D. Problem compounds need to be addressed on a project to project basis.

Continuing Calibration Verification standards(CCV)	Beginning of Every 12 hour shift, after the BFB injection. Shall be at or near the mid-point of calibration range for all target compounds, and is prepared from standards used for calibration (Typically use 10/100 ppb for H2O and Low Level soils, 0.5/5.0 mg/kg for MeOH preserved soils). Varied CCV levels are required for QSM when multiple CCV's are necessary on a run (Typically use 10, 20, and 30 ppb for H2O and Low Level soils, 0.5, 1.0, and 1.5 mg/kg for MeOH preserved soils). Heated purges for low-level soils.	1. Average relative response factors (RRFs) for compounds on Table 4. 2. %RSD <20% Deviation for RRFs, <20 % Drift for linear curve and nonlinear curves 3. NELAC, DoD-QSM, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents.	Correct system and recalibrate. Criteria must be met before sample analysis may begin. IF % RSD >20%, then system must be inspected and problem corrected before sample analyses. If >20% RSD correct problem if determinable then reanalyze, and/or determine if it's a sporadic problem involving compounds that are typically poor performers. In any case sample results reported that have %D failures must be qualified (Z). ACOE allows no tolerance for % D. Problem compounds need to be addressed on a project to project basis QSM 5.0 - Immediately analyze two additional consecutive CCVs. If both pass (for those compounds that initially failed), 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.
Internal Standards (ISTD)	Added to all blanks, standards, and samples.	1. Peak area within -50% to +100% of area in associated CCV standard. 2. Retention time (RT) within 10 sec of RT for associated CCV standard. 3. NELAC, DoD-QSM, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents.	Inspect instrument for malfunctions; correct identified malfunctions, then reanalyze samples. If no instrument malfunction identified proceed as follows: * Reanalyze sample. * If reanalysis is outside limits the data shall be qualified (S). Follow specified criteria as stated in Shell or other documentation.

Method Blank (MB)	1 / 20 samples per matrix or at program/project specific frequencies. The MB is used to document contamination resulting in the analytical process and shall be carried through the complete sample preparation and analytical procedure.	1. Concentration of analytes of concern shall be less than the highest of either : *Method Detection Limit *Five percent of the regulatory limit for that analyte or, *Five percent of the measured concentration in the sample. 2. DoD-QSM: ≤ ½ RL 3. Follow criteria according to specific program or project. 4. QSM5.0 – Know Lab contaminants = No detection above the LOQ	Reanalyze to determine if instrument or laboratory background contamination was the cause. If the method blank is still non-compliant, re-prepare and reanalyze blank and samples.* For ACOE/QSM data if less than ½ RL no action required.* *If reanalysis of blank still contains contamination above specified limits, affected data shall be flagged (B).
Laboratory Control Sample (LCS)	1 / 20 samples or at contact/ program specific frequencies. Must undergo all sample preparation procedures. Prepared from a second source and contain target compounds with concentrations at or near the mid-point of the calibration range.	1. % Recoveries (and RPDs, if applicable) within in-house generated limits. Default 70- 130% (20% RPD). 2. Use DoD-QSM, program/project specific, or client contract limits when applicable.	If LCS recoveries are within control limits or within SMF frequency and limits then no action is required. If the LCS exceeds control limits, as well as SMF criteria the reanalyze the LCS to confirm proper preparation procedure. If still exceeding limits then reanalyze associated samples with a new LCS.. If sample data is reported with LCS failures then that data must be qualified (Q). Exception: If the LCS recoveries are high with no associated positives then no further action is taken.

Matrix Spike/Matrix Spike Duplicate	One per set of 20 samples per matrix. Must undergo all sample preparation procedures. Must be spiked with target compounds with concentrations at or near the mid-point of the calibration range.	1. % Recoveries and RPDs within in-house generated limits. Default 70-130 / 20%RPD. 2. Use DoD-QSM, program/project specific, or client contract limits when applicable. 3. QSM 5.0 – RPD within 20% 4. QSM 4.2 – RPD within 30%	If LCS is acceptable, then report probable matrix interference. Qualify non-detects if the recoveries are low (M), and detects if the recoveries are low and the sample amount + the true spiked amount shall be within calibration range. Qualify detects if recoveries are high and the detects + the true spiked amount are within calibration range. If recoveries are high and there are no detects in the parent sample then that data does not require flagging. If spiked amount + sample amount for any given compound exceeds calibration range than the spike is considered invalid for that compound. Qualify data for RPD failures (Y) when there is a detect for the failing compounds (non-detected compounds are not qualified). Exception: If a compound is already qualified for a LCS failure then no RPD qualifier is applied.
Qualitative/Quantitative Issues	1. If detection level of any compound in a sample exceeds the detection level of that compound in the highest level standard, the sample must be diluted to approximately mid-level of the calibration range and reanalyzed. 2. If the concentration of the target analyte (that exceeded the calibration range) is present in the sample following the high level sample and is greater than the RL but ≤5x RL, then that sample must be reanalyzed to determine if carryover occurred.	1. The instrument level of all compounds must be within the calibration range for all samples. 2. The sample analyzed immediately after a high-level sample must display concentrations of the high level target compounds less than the RL or greater than 5x RL.	Dilute the sample to bring the level of the highest concentration of target compounds within the calibration range. If any data is reported with any results over range then those results shall be flagged (X). A sample displaying concentrations of target compounds between the RL and 5x the RL that was analyzed immediately after a high-level sample must be reanalyzed. If the results do not agree within the RL, report only the second analysis.

Surrogate	1. Typically use single point calibrations. 2. Added to all blanks, samples, and QC samples, as a part of the internal standard-surrogate spiking mixture.	1. All % Recoveries within in-house generated limits. Default 70-130%. 2. Use DoD-QSM, program/project specific, or client contract limits when applicable.	If recoveries are not within limits: Check to be sure that there are no errors in calculations, surrogate solutions, or internal standards. Also, check instrument performance. If no problem is found, re-prepare and reanalyze the sample. If the reanalysis is within limits, report only the reanalysis. If the reanalysis is still out of limits the sample shall be qualified (S). Due to matrix affect, no reanalysis is required if the MS and/or MSD are outside limits.
Sample Duplicate (Dup) – when required	1. Program/contract specific. 2. When limited sample is available a sample duplicate may be used in lieu of a MSD.	1. RPD < or = 10% (between sample and sample duplicate) for QSM projects. 2. RPD's within in-house limits. Default ± 20%. 3. DoD-QSM, NELAC, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents.	If RPDs are not within limits: Check to be sure that there are no errors in calculations. Also, check instrument performance and correct if necessary. If corrected or no problem is found, re-prepare and reanalyze the sample. If the reanalysis is within limits, report only the reanalysis. If the reanalysis is still out of limits the sample shall be qualified (Y).
Method Reporting Limit (MRL) Spike – when required	1. Program/contract specific. 2. Typically bracketing samples for every 12 hour analysis window.	1. % Recoveries within in-house generated limits. Default 70-130 %Rec. 2. Program or project/contract specific limits shall be followed as stated in their documents..	If there is a failure investigate problem. If system is in control run an MDL check sample to verify detection limits.
Continuing Calibration Verification Final(CCVF) (QSM 5.0 only)	For QSM 5.0: at the end of the analytical sequence for a batch of 20 or fewer samples. Shall be at or near the mid-point of calibration range for all target compounds, and is prepared from standards used for calibration (Typically use 10/100 ppb for H2O and Low Level soils, 0.5/5.0 mg/kg for MeOH preserved soils).	1. %RSD <50% Deviation for RRFs, <50 % Drift for linear curve and nonlinear curves	QSM 5.0 - Immediately analyze two additional consecutive CCVFs. If both pass (for those compounds that initially failed), 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 CCVF. (it is allowable, if needed, the two reanalysis of the ending CCVF can extend beyond the 12 hour analysis window.

Table 3
Characteristic ions

Analyte	Primary Ion	Secondary Ion	Analyte	Primary Ion	Secondary Ion
Acetone	43	58	2,2-Dichloropropane	77	97,79
Benzene	78	51,77	1,1-Dichloropropene	110	77,75
Bromobenzene	156	77,158	cis-1,3-Dichlropropene	75	110
Bromochloromethane	128	49,130	trans-1,3-Dichloropropene	75	77,110
Bromodichloromethane	83	85,129	Diisopropyl ether	45	87,43
Bromoform	173	175,171	Ethylbenzene	91	106
Bromomethane	94	96	Hexachlorobutadiene	225	223,227
2-Butanone	43	72,57	2-Hexanone	43	58,57
n-Butylbenzene	91	92,134	Isopropylbenzene	105	120
sec-Butylbenzene	105	134	p-Isopropyltoluene	119	134,91
tert-Butylbenzene	119	91,134	Methylene chloride	84	86,49
Carbon disulfide	76	78	4-Methyl-2-pentanone	43	58,57
Carbon tetrachloride	119	121	Methyl tert butyl ether	73	57,43
Chlorobenzene	112	77,114	Naphthalene	128	51,129
Chloroethane	64	66	n-Propylbenzene	91	120
2-Chloroethylvinyl ether	63	65,106	Styrene	104	78
Chloroform	83	85	1,1,1,2-Tetrachloroethane	131	133,119
Chloromethane	50	52	1,1,2,2-Tetrachloroethane	83	85
2-Chlorotoluene	91	126	Tetrachloroethene	166	168,129
4-Chlorotoluene	91	126	Tetrahydrofuran	42	72,71
Dibromochloromethane	129	127,131	Toluene	92	91
1,2-Dibromo-3-chloropropane	157	155	1,2,3-Trichlorobenzene	180	182,145
1,2-Dibromoethane	107	109	1,2,4-Trichlorobenzene	180	182,145
Dibromomethane	93	95,174	1,1,1-Trichloroethane	97	99,61
1,2-Dichlorobenzene	146	111,148	1,1,2-Trichloroethane	83	97,85,99
1,3-Dichlorobenzene	146	111,148	Trichloroethene	95	130,132
1,4-Dichlorobenzene	146	111,148	Trichlorofluoromethane	101	103,105
Dichlorodifluoromethane	85	87	1,2,3-Trichloropropane	75	110
1,1-Dichloroethane	63	65,83	1,2,4-Trimethylbenzene	105	120
1,2-Dichloroethane	62	98,64	1,3,5-Trimethylbenzene	105	120
1,1-Dichloroethene	96	61,63	Vinyl chloride	62	64
cis-1,2-Dichloroethene	96	61,98	Vinyl acetate	43	86
trans-1,2-Dichloroethene	96	61,98	o-Xylene	106	91
1,2-Dichloropropane	63	76,112	m/p-Xylene	106	91
1,3-Dichloropropane	76	78			
			SSTD		
ISTD			Dibromofluoromethane	113	111,192
Fluorobenzene	96	77	1,2-Dichloroethane-d_4	102	104
Chlorobenzene-d_5	117		Toluene-d_8	98	100
1,4-Dichlorobenzene-d_4	152		4-Bromofluorobenzene	95	174,176

***Refer to Method 8260C for characteristic ions not listed here**

FV04-01 (Example)
8260C Checklist

FVO4-01 Data Review Checklist

FORM #: FVO4-01
Rev. #: 1.1
Effective Date: 4/01/15

INDEPENDENT DATA REVIEW CHECKLIST		Method: GCMS (EPA SW-846 8260C)					
Analysis Date	LIMs Run #(s):	Analyst/Data Interpreter	Independent Reviewer	Date of Review	Approved? Yes / No		Instrument
					Yes		VMS
Instructions: Complete one checklist per analytical sequence. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section and may require the initiation of a Nonconformance Report.							

Requirement:	Acceptance criteria	Analyst Yes	Analyst Review No	Independent Review Yes	Independent Review No	Comments:
I. BFB Tune Check						
Was a BFB tune check analyzed with acceptable results?	Relative abundance criteria met?	Yes		Yes		If no, Do not proceed with analyses.
II. Initial Calibration Verification (ICV)						
Was initial calibration performed using a minimum of five standard concentration levels (minimum of 6 levels for quadratic curves)?	Lowest standard at or below RL?	Yes		Yes		If no, recalibrate with required # of levels
Were the Average Relative Response Factors (ARRF) acceptable?	ARRF≥ specified limits (see SOP)	Yes		Yes		If no, analyses stopped, recalibrate.
Was a second-source ICV analyzed?	Required before sample analyses.	Yes		Yes		If no, analyze prior to sample analyses.
Were all target compound %Deviation or %Drift acceptable?	%D < 20% or project/program specific					If no, reanalyzed ICV to address failures
Was Initial Calibration Blank (ICB) analyzed?	Required before sample analyses.	Yes		Yes		If no, analyzed ICB before sample analyses.
Were the ICB results for all target analytes less than the limit of detection (LOD).	<LOD or < project/program specific limits					If no, analyze another blank to address detects.
III. Continuing Calibration Verification (CCV)						
Was an acceptable BFB tune check run at the beginning of every twelve hour shift?	Relative abundance criteria met?	Yes		Yes		If no, reanalyze BFB until acceptable; re-tuning the instrument may be necessary.
Was a CCV analyzed after every 12 hour tune check?	Required before sample analyses.	Yes		Yes		If no, analyzed CCV after acceptable tune check.
Were all target compound %Deviation or %Drift acceptable?	%D < 20% or project/program specific					If no, reanalysis or recalibration may be required.
If necessary, were the results for outlying compounds qualified?						If yes, results qualified "Z"

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IV. Blanks						
Was a Method Blank (MB) analyzed prior to analysis of samples?	1 per 20 samples or project/program specific.	Yes		Yes		If no, analyze a MB with each sample batch.
Were the MB results for all target analytes less than the limit of detection (LOD)?	*All target analytes <LOD or < project/program specific limits (<1/2 RL for DoD-QSM)					If no, reanalyze MB on all affected data if possible or see SOP for more detailed criteria
If analytes were detected in the MB with no associated positives in the samples, no further action is needed. If the analyte detects in the MB were greater than the acceptance criteria and there were detects in the samples, was the data qualified.?						If yes, affected results qualified "B"
V. Laboratory Control Spike (LCS)						
Was a LCS analyzed at the required frequency?	1 per 20 samples or project/program specific.	Yes		Yes		If no, analyze a LCS with each sample batch.
Were the LCS recoveries for all analytes within acceptance criteria?	Default 70-130%, or see internally generated limits, or project/program specified limits.					If no, Reanalyze LCS and all affected data if possible or data requires qualification.
If applicable, were associated sample detects (and non-detects for low recoveries) qualified?						If yes, affected results qualified "Q"
VI. Matrix Spike/Matrix Spike Duplicate (MS/MSD)						
Was a Matrix Spike (MS) and a Matrix Spike Duplicate (MSD) analyzed at the required frequency?	1 per 20 samples or project/program specific.	Yes		Yes		If no, analyze a MS/MSD pair with each sample batch.
Were the MS/MSD recoveries for all analytes within acceptance criteria?	Default 70-130%, or see internally generated limits, or project/program specified limits.					If no, qualify detects (with an "M" flag) in the parent sample, also qualify non-detects if MS/MSD recoveries were low.
Is the relative percent difference (RPD) for each analyte between the MS and MSD acceptable?	generated limits, or client specific limits.					If no, affected results qualified "Y"
VII. Sample Analyses						
Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?		Yes		Yes		If no, instrument maintenance may be required to correct problems.
Were surrogate recoveries for all samples and QC within acceptance criteria?	Default 70-130%, or see internally generated program limits, or client specified limits.					If no, samples with high surrogate recoveries and no associated analyte detects were not reanalyzed. Low surrogate recoveries require reanalysis.
If possible, were the affected samples reanalyzed?		Yes				If no, see below **
**Were reported sample results with failing surrogate recoveries qualified?		Yes				If yes, affected results qualified "S"
Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed? If not qualify (X).	Target upper middle range of calibration curve.	Yes		Yes		
Did all samples meet hold time and preservation criteria as defined by method/program?	H2O sample: pH < 2 = 14 days, pH >2 – 7 days. Soil samples: 14 days (other criteria may apply)					If no, see below: If analyzed past hold, qualify "H" If improperly preserved qualify "T"
Were all samples and QC injected within 12 hours of BFB tune check?		Yes		Yes		If no, affected samples reanalyzed
Were internal standard recoveries acceptable relevant to associated ICAL?	Response = -50 to +200%; Ret. time = +/- 30 sec.					If no, affected samples reanalyzed

VIII. Records and Reporting						
Is sequence file / injection log present in the data package?		Yes		Yes		If no, include sequence run with raw data.
Were all data, calculations, and values verified in LIMS upon completion of data capture?		Yes		Yes		If no, recapture the data and verify data prior to validation.
Were manual integrations addressed properly and were the audit trails turned on (where applicable)?	Manual integration must be initialed, dated, and reason given, along with before & after chromatograms. Audit trail must be on (if available).	Yes		Yes		IF No, address manual integration and/or turn on audit trail feature and document reason why it may have been turned off.
Are reported results whose amounts exceeded the acceptance criteria flagged with an appropriate qualifier and, if needed, were any non-matrix related nonconformities documented in the NCR spreadsheet?						If No, include proper qualification(s) in LIMS and enter nonconformities into the NCR spreadsheet before data review/validation.

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Non-applicable Yes/No cells are left blank

Comments: _____

FVO4-02 (Example)
VOC Soils Preserved Prep Bench Sheet (Non-Lust)

FVO4-02

VOC SOILS PRESERVED - PREP BENCHSHEET

Prep Batch #:	
Method:	
Analyst:	
Date:	
Start Time:	

NON-LUST

	Sample ID	Vial Weight (g)	Tare Weight (g)	Sample Weight (g)		MeOH Added (mL)
1				-0.25		
2				-0.25		
3				-0.25		
4				-0.25		
5				-0.25		
6				-0.25		
7				-0.25		
8				-0.25		
9				-0.25		
10				-0.25		
11				-0.25		
12				-0.25		
13				-0.25		
14				-0.25		
15				-0.25		
16				-0.25		
17				-0.25		
18				-0.25		
19				-0.25		
20				-0.25		
21				-0.25		

Samples received unpreserved are preserved at a 1:1 ratio, weight to volume with methanol
Balance: Mettler Toledo, BD202
LCS & MB prepared @ 1:1 ratio MeOH/Silica Sand
MeOH used (Supplier/Lot#):
Sample Weight = Vial Weight - Tare Weight - 0.25g (label weight)

Stop Time:	

VocSoilPresNonLust_FVO3-02 05/07/201415:07

FVO4-03 (Example)
VOC Soils Preserved Prep Bench Sheet (Lust)

VOC SOILS PRESERVED - PREP BENCHSHEET

Prep Batch #:	
Method:	
Analyst:	
Date:	
Start Time:	

LUST

(volume adjustment)

	Sample ID	Total Weight (g)	Jar Weight (g)	Sample Weight (g)	MeOH Added (ml)	MeOH Total Volume (mL)
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						

Total Wt = Jar Wt + Sample Wt + MeOH Wt (25mL = 19.8g) + CTL Label (0.25g)
Jar Wt = Tared Wt of jar
Sample Wt = Total Wt - Jar Wt - 19.8 - 0.25
MeOH Added = Additional MeOH added if Sample Wt is greater than 25g
MeOH Total Volume = Total amount of MeOH added to sample
Balance: Mettler Toledo, VBD202
LCS & MB prepared @ 1:1 ratio MeOH/Silica Sand
MeOH used for LCS, MB, and volume adjustment (Supplier/Lot#):

Stop Time:	

VocSoilPresLust_FVO3-03 05/07/201415:13

FVO4-04 (Example)
VOC Soils Preserved Prep Bench Sheet (Non-Lust) B

VOC SOILS PRESERVED - PREP BENCHSHEET

Prep Batch #:	
Method:	
Analyst:	
Date:	
Start Time:	

NON-LUST

	Sample ID			Sample Weight (g)		MeOH Added (mL)
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						

Samples received unpreserved are preserved at a 1:1 ratio, weight to volume with methanol
Balance: Mettler Toledo, BD202
LCS & MB prepared @ 1:1 ratio MeOH/Silica Sand
MeOH used (Supplier/Lot#):
Sample Weight = Vial Weight - Tare Weight

Stop Time:	

VocSoilPresNonLustB_FVO3,4-04 05/07/201415:22

FVO4-05 (Example)
VOC Soils Low Level Prep Bench Sheet (Non-Lust)

VOC SOILS LOW LEVEL - PREP BENCHSHEET

Prep Batch #:	
Method:	
Analyst:	
Date:	
Start Time:	

NON-LUST

	Sample ID	Sample Weight B (g)	Sample Weight C (g)	Sample Weight A (g)		DI H20 (mL)
1						5.0
2						5.0
3						5.0
4						5.0
5						5.0
6						5.0
7						5.0
8						5.0
9						5.0
10						5.0
11						5.0
12						5.0
13						5.0
14						5.0
15						5.0
16						5.0
17						5.0
18						5.0
19						5.0
20						5.0

Voc LLSoil Temp_FVO3-05 05/07/201415:20

FVO4-06 (Example)
VOC Soils Preserved Prep Bench Sheet (5035)

VOC SOILS PRESERVED - PREP BENCHSHEET

Prep Batch #:	
Method:	
Analyst:	
Date:	
Start Time:	

5035

	Sample ID	Total Weight (g)	Jar Weight (g)	Sample Weight (g)		MeOH Total Volume (mL)
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						

Total Wt = Jar Wt + Sample Wt + MeOH Wt (25mL = 19.8g) + CTL Label (0.25g)
Jar Wt = Tared Wt of jar
Sample Wt = Total Wt - Jar Wt - 19.8 - 0.25
MeOH Total Volume = Total amount of MeOH added to sample
Balance: Mettler Toledo, VBD202
LCS & MB prepared @ 1:1 ratio MeOH/Silica Sand
MeOH used for LCS & MB (Supplier/Lot#):

Stop Time:	

VocSoilPres25mL_FVO3-06 05/07/201415:25

FVO4-07 (Example)
VOC Soils Preserved Prep Bench Sheet (Lust) 5 mL

VOC SOILS PRESERVED - PREP BENCHSHEET

Prep Batch #:	
Method:	
Analyst:	
Date:	
Start Time:	

LUST

	Sample ID	Total Weight (g)	Jar Weight (g)	Sample Weight (g)	MeOH Added (ml) (volume adjustment)	MeOH Total Volume (mL)
1				-0.25	-5.3	-0.3
2				-0.25	-5.3	-0.3
3				-0.25	-5.3	-0.3
4				-0.25	-5.3	-0.3
5				-0.25	-5.3	-0.3
6				-0.25	-5.3	-0.3
7				-0.25	-5.3	-0.3
8				-0.25	-5.3	-0.3
9				-0.25	-5.3	-0.3
10				-0.25	-5.3	-0.3
11				-0.25	-5.3	-0.3
12				-0.25	-5.3	-0.3
13				-0.25	-5.3	-0.3
14				-0.25	-5.3	-0.3
15				-0.25	-5.3	-0.3
16				-0.25	-5.3	-0.3
17				-0.25	-5.3	-0.3
18				-0.25	-5.3	-0.3
19				-0.25	-5.3	-0.3
20				-0.25	-5.3	-0.3
21				-0.25	-5.3	-0.3

Total Wt = Jar Wt + Sample Wt + MeOH Wt (25mL = 19.8g) + CTL Label (0.25g)
Jar Wt = Tared Wt of jar
Sample Wt = Total Wt - Jar Wt - 0.25
MeOH Added = Additional MeOH added if Sample Wt is greater than 5g
MeOH Total Volume = Total amount of MeOH added to sample
Balance: Mettler Toledo, VBD202
LCS & MB prepared @ 1:1 ratio MeOH/Silica Sand
MeOH used for LCS, MB, and volume adjustment (Supplier/Lot#):

Stop Time:	

Table 4
Recommended Minimum relative response factor criteria for Initial and Continuing Calibration Verification

Volatile Compounds	Minimum Response Factor (RF)
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone[1]	0.100
Carbon disulfide	0.100
Methyl Acetate	0.100
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone[1]	0.100
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichlorpropene	0.200
trans-1,3-Dichlorpropene	0.100
4-Methyl-2-pentanone[1]	0.100
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone[1]	0.100
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100

Volatile Compounds	Minimum Response Factor(RF)
Chlorobenzene	0.500
Ethylbenzene	0.100
Meta-/para-Xylene	0.100
Ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorbenzene	0.200

[1] Due to low response at standard levels, these compounds are run at a concentration ten times the normal.

20.0 Description of Changes

Revision Number	Description of Changes	Date
00	Document changed to incorporated administrative requirements of ISO 17025 and QSM 5.0. Descriptions of changes have not been tracked in previous versions of this document.	03/12/2014
01	Changed SOP to new format, updated for QSM 5.0	01/28/2015
02	Added SIM mode analyses to Scope & Application	03/09/2015

CT LABORATORIES

delivering more than data from your environmental analyses

STANDARD OPERATING PROCEDURE

PR 002 TCLP and SPLP Extraction, Volatile Fraction (ZHE)

Review Date: 03/31/2016

03/31/2016

Technical Review by: Date

03/31/2016

Approved by: Quality Assurance Date

1.0 Identification of Test Method

The Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP) are designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes. The following procedure will be used for performing the volatile organic ZHE procedure.

2.0 Applicable Matrix or Matrices

This procedure is used for the extraction of purgeable VOCs for a variety of liquid and solid matrices including soils, sludge, and waste samples.

3.0 Detection Limits

Method Detection Limits (MDLs) are compound, instrument, and matrix dependent. MDL analyses are performed annually for the instruments and matrices applicable to this procedure. The reporting limits (RLs) used are based on whether or not samples have been diluted prior to analyses. Default reporting limits are used for the standard TCLP/SPLP list used for the volatile organic analysis (see Scope and Application, figure 1).

4.0 Scope and Application

This procedure is applicable to a wide range of volatile organic compounds though typically only the standard TCLP/SPLP list is required (see figure 1). The reporting limits used reflect the Maximum Contaminant Level (MCL) allowed for TCLP samples. After performing the TCLP or SPLP extractions, the samples are analyzed by GCMS following the procedures outline in SOP 5280B (*Analysis of Volatile Organic Compounds by GC/MS*).

Figure 1.Standard TCLP/SPLP Compound List

Compound	Reporting limit (mg/L)
vinyl chloride	0.2
1,1-dichloroethene	0.7
1,2-dichloroethane	0.5
2-butanone	200
chloroform	6
carbon tetrachloride	0.5
benzene	0.5
trichloroethene	0.5
tetrachloroethene	0.7
chlorobenzene	100

5.0 Method Summary

5.1 TCLP

5.1.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP extract.

5.1.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. If the sample is a waste or wastewater, the extraction fluid employed is a pH 4.93 solution. A special extractor vessel (ZHE) is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

5.1.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

5.2 SPLP

5.2.1 For liquid samples (i.e., those containing less than 0.5 % dry solid material), the sample, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the SPLP extract.

5.2.2 For samples containing greater than 0.5 % solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of reagent DI water equal to 20 times the weight of the solid phase. A special extractor vessel (ZHE) is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

5.2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

6.0 Definitions

6.1 For definitions on all terms applicable to this method, see Appendix 10 of the Quality Assurance Manual (QAM).

6.2 For a list of common acronyms and abbreviations, see QAM Appendix 7.

7.0 Interferences

7.1 Carry over contamination is a problem when a highly contaminated sample is followed by a clean sample. Thorough cleaning and rinsing the ZHE device will eliminate the potential for carry over.

7.2 Other potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

8.0 Safety

8.1 Gloves and protective clothing should be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems.

8.2 The toxicity and carcinogenicity of the chemicals used in this method are not precisely defined. Each chemical and sample shall be treated as a potential health hazard, so care must be taken to prevent undue or extensive exposure.

8.3 For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psig or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced.

8.4 The ZHE should be checked periodically for leaks. If the device contains a built-in pressure gauge, pressurize the device to 50 psig, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psig, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device.

8.5 All personnel performing this analysis shall be instructed in the use of personal protective equipment prior to beginning analysis.

9.0 Equipment and Supplies

9.1 Rotator apparatus capable of turning at 30 +/- 2 rpm (Lars Land or equivalent).

9.2 ZHE extraction devices (Lars Land or equivalent).

9.3 Pressure filtration apparatus (Millipore or equivalent).

9.4 Glass fiber filters 0.6-0.8 micron 11.0 cm. (Environmental Express, Cat. # FG75110MM or equivalent).

9.5 Glass fiber filters 0.6-0.8 micron: 15.0 cm. (Environmental Express, Cat. # FG75150MM or equivalent).

9.6 Fluid pump (FMI Lab Pump, Model QSY or equivalent).

9.7 Gas pressure/vacuum pump (Gast, Model DOA-P104-AA or equivalent).

9.8 250 mL and 500 mL beakers (Pyrex or equivalent).

9.9 Transfer line, 1/8”ID x 1/4”OD (Nalgene, 280 Pur-ester tubing)

9.10 Tedlar Bag

9.11 1000 ml Graduated cylinders, Class A (Kimble or equivalent).

9.12 100mL glass gas tight syringe (Hamilton 7000 series or equivalent).

9.13 40 mL VOA vials (C&G or equivalent).

9.14 Analytical balance (Ohaus, Voyager Pro or equivalent).

9.15 Top-loading balance (Mettler-Toledo, Model BD-202 or equivalent).

9.16 TCLP/SPLP prep log (see Tables 2 & 3).

Note: The interior surface of the ZHE (9.2) and the pressure filtration apparatus (9.3) should be smooth and free of scratches. Clean using only a very soft bristled brush if necessary. In addition, the screen on which the filter is placed should be clear of debris. If any of the holes are clogged they can be cleaned by sonicating for 15 minutes.

10.0 Regents and Materials

10.1 Reagent grade DI water, organic free (Millipore, 18 mega ohm quality).

10.2 Glacial Acetic Acid (CH3CH2OOH), ACS Grade (Fisher, Cat.# A38S-212 or equivalent).

10.3 Sodium Hydroxide (NaOH), pellets

10.3.1 Fisher, Cat.# S318-3 or equivalent.

10.3.2 10 N NaOH solution. Into a 1 liter volumetric flask, add 500 mL of DI water. Dissolve 400g of NaOH pellets (caution: mixture will become very hot). When cool, dilute to volume with D.I. H2O and mix well.

10.4 Extraction Fluids

10.4.1 TCLP extraction fluid #1: (To prepare a 20 liter quantity): Fill a 20 L carboy with 19 L of DI water. Add 114 mL CH3CH2OOH and 128.6 mL 10N NaOH. Dilute to 20 L With D.I. H_2O and mix by stirring. When correctly prepared, the pH of this fluid will be 4.93 +/- 0.05.

Note: This extraction fluid should be monitored frequently for impurities. The pH should be checked prior to use to ensure that the fluid is made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

10.4.2 SPLP extraction fluid: Reagent DI water.

11.0 Sample Preservation and Storage

11.1 Preservatives shall not be added to samples before extraction.

11.2 Care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 0-6ºC. Samples shall be opened only immediately prior to extraction. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses.

11.3 Volatiles have 14 days from the date of collection to be extracted.

11.4 Volatiles have 14 days from the date of extraction to be analyzed.

12.0 Quality Control

12.1 This SOP is designed to follow a variety of different projects and programs requirements. Table 1 is designed to illustrate the control steps and provisions required to adequately producing acceptable data.

12.2 Contract Specific Sample Analysis: For certain samples, limits are specified by the QAPP (Quality Assurance Project Plan) associated with a given project. For these samples follow the limits specified in the QAPP for that project.

12.3 Program Specific Limits: Samples analyzed under the guidance of certain programs; such as the Department of Defense Quality Systems Manual (DoD/QSM) or Louisville Chemistry Guidance (LCG), require their own specified limits. For these samples follow the limits specified in the manuals for that program.

13.0 Calibration and Standardization

See SOP 8260B (*Analysis of Volatile Organic Compounds by GC/MS*) for the appropriate analyses calibration.

14.0 Procedure

14.1 Determine sample % solids:

14.1.1 For solid samples which contain no free liquids, proceed to sec. 14.3.

14.1.2 For samples which are liquid, contain free liquids, or are multi-phasic, filtration or liquid/solid separation is required as follows:

14.1.2.1 Preweigh a GFF filter and record the weight.

14.1.2.2 Preweigh a receiving beaker and record the weight.

14.1.2.3 Preweigh a transfer beaker and record the weight.

14.1.2.4 Assemble the pressure filtration device with the GFF filter, and place the receiving beaker underneath.

14.1.2.5 Weigh out a subsample of the waste (100g. minimum) and record the weight. An additional minimum 25g will be needed for the extraction. For low volume samples, consult your supervisor.

14.1.2.6 Transfer the waste to the filtration device and secure the top.

14.1.2.7 Re-weigh the empty transfer beaker and record the weight.

14.1.2.8 Slowly apply air pressure to the filtration device in 10 psi increments up to 50 psi. or until air passes through the filter. Hold at each increment for 2 minutes before proceeding to the next higher increment.

Note: Some wastes, such as oily wastes and some paint wastes will contain material that appears to be a liquid. Even after applying pressure to 50 psi, this material may not filter. In this case, for the non-volatile extraction, the material in the filter holder is defined as the solid phase and is carried through the extraction procedure as a solid. However, since the volatile procedure requires utilizing the ZHE device to filter the sample following rotation, the ZHE filter may become plugged and filtration of the extract from the ZHE device may not be possible. Typically the % solids is predetermined in the preparation procedure for the non-volatile constituents.

14.1.2.9 Weigh the receiving beaker and record the weight.

14.1.2.10 The material in the filter holder is defined as the solid phase of the waste, and the material in the receiving beaker is defined as the liquid phase.

14.1.2.11 Determine and record the weight of the liquid phase.

14.1.1.12 Determine the weight of the solid phase by subtracting the weight of the liquid phase from the total weight of the waste.

14.1.1.13 Calculate the % solids as follows:

$$\% \text{ solids} = \frac{\text{weight of solid phase}}{\text{total weight of waste}} \times 100$$

14.2 Evaluation of % solids:

14.2.1 If the % solids are <0.5%, a fresh portion of the waste will be filtered through the ZHE device and collected into a Tedlar bag or 100 mL syringe. This filtrate will be defined as the TCLP extract. Proceed to sec.13.3, then 13.4.4

14.2.2 If the % solids are significantly >0.5%, proceed to section 14.3, then 14.4.1. or 14.4.2.

14.2.3 If the % solids are ≥0.5% or are very close, and it is noticed that the solid material is entrained in the filter, dry the filter at 80-120C until two successive weighings agree within +/- 1%. Determine the % dry solids. If the % dry solids are <0.5%, follow 14.2.1. If the % dry solids are >0.5%, see note below.

Note: There must be a significant level of % solids such that a minimum of 5-10g of solids can be generated for the extraction. This minimum amount of solids will yield 100-200 mL of extract.

14.3 ZHE device preparation:

14.3.1 Assemble the ZHE device as follows:

14.3.1.1 Place two O-rings on the piston.

14.3.1.2 Place an O-ring in the ZHE base.

14.3.1.3 Wet the O-rings of the piston and place the piston inside the ZHE body. Depress the piston into the ZHE body only far enough to allow room for the sample.

14.3.1.4 With the piston installed, place the ZHE body into the base.

14.3.1.5 Place an O-ring on the top of the ZHE body.

14.3.1.6 Place an 11.0 cm. GFF filter between the two filter screens and set aside.

14.3.1.7 The ZHE device is now ready to receive a sample.

14.4 Adding sample to the ZHE device:

14.4.1 For samples that are 100% total solids:

14.4.1.1 Weigh out 25.0g. of sample into a beaker or other suitable container and record the weight. If particle size reduction if required, chill the sample and reduction equipment to 4^0C to reduce the loss of volatile compounds. Proceed to chop, crush, or grind the sample to a minimum 1cm^2 size. Minimize exposure to the atmosphere.

14.4.1.2 Transfer the waste material to the ZHE device and secure the top.

14.4.1.3 Attach the air line from the pressure pump to the lower valve. Open the upper and lower valves.

14.4.1.4 Slowly pressurize the ZHE device to 50 psi to force the piston to the top of the ZHE body, thereby removing any headspace. Remove the air line.

14.4.1.5 Determine the amount of extraction fluid to add follows: amount of fluid added = 20 x's the weight of the sample

14.4.1.6 Proceed to sec. 14.5

14.4.2 For samples >5% but <100% total solids:

14.4.2.1 Determine the amount of sample to add to the ZHE as follows:

$$\text{Weight of sample to use} = \frac{25}{\%\ \text{total solids (sec. 14.1)}} \times 100$$

14.4.2.2 Proceed to sec. 14.4.3, using the amount of sample determined above.

14.4.3 For samples that are between 0.5% - 5% total solids:

14.4.3.1 Weigh a 500 mL beaker and record the weight.

14.4.3.2 Into the beaker, weigh out 500 g of sample and record the weight.

14.4.3.3 Transfer the waste to the ZHE device and secure the top.

14.4.3.4 Connect the air line to the lower valve. Open upper and lower valves.

14.4.3.5 Slowly pressurize the ZHE to expel any air. Close the top valve when liquid appears.

14.4.3.6 Pre-weigh a Tedlar bag and record the weight.

14.4.3.7 Slowly pressurize the ZHE to expel the liquid into the bag. Do not exceed 50 psi.

14.4.3.8 Close the valves. Remove the bag, weigh the bag and its contents, and record the weight.

14.4.3.9 Determine the amount of extraction fluid to add follows:

$$\text{weight of extraction fluid} = \frac{20 \times \text{weight of sample waste} \times \%\ \text{solids}}{100}$$

11.4.3.10 Proceed to sec. 14.5

14.4.4 For samples that are <0.5% total solids:

14.4.4.1 The liquid portion of the waste, after filtration is defined as the TCLP extract. The ZHE device will be used to filter the sample.

14.4.4.2 Add an appropriate amount of sample to the ZHE device to complete the requested analyses.

14.4.4.3 Secure the top; connect the air line to the lower valve. Open upper and lower valves.

14.4.4.4 Slowly pressurize the ZHE to expel any air. Close the top valve when liquid appears.

14.4.4.5 Connect a 100 mL syringe or evacuated Tedlar bag to the top valve. Slowly pressurize the ZHE to force the liquid into the syringe or bag. Alternatively a transfer line can be connected to the outlet valve on the ZHE and the sample extract can be transferred directly into the VOA vial. If collecting the liquid with a syringe, carefully transfer, (after discarding the first 5 mL) with headspace, to 40 mL VOA vials. If using a bag, allow the liquid to flow into the bag until a sufficient quantity has been collected for analysis. Store the extract at 0-6^{0}C until analysis.

14.5 Adding extraction fluid to the ZHE device:

14.5.1 Transfer via a graduated cylinder the appropriate amount and type of extraction fluid to a 500 mL beaker.

14.5.2 Using the fluid pump, place the intake line into the beaker of extraction fluid. Turn on the pump, and allow the fluid to enter the pump. Stop the pump when the fluid appears at the end of the outlet line.

14.5.3 Connect the outlet line to the ZHE top valve. Turn on the pump, and allow the pump to charge the ZHE with the entire contents of the beaker. Stop the pump before any air reaches the ZHE. Close the top and bottom valves, and remove the inlet line. Manually rotate the ZHE device in an end over end fashion 2 or 3 times.

Note: While the ZHE is filling, check for fluid leaking out of the bottom valve. If this happens, stop the pump and use a different ZHE device with a fresh sample.

14.5.4 Attach the air line to the bottom valve. Open the top valve, and while holding a paper towel at the valve, slowly pressurize the ZHE to expel any remaining air. Close the valve at the first sign of fluid at the valve outlet.

14.5.5 With the air line still attached, re-pressurize the ZHE device to 10 psi. Check for leaks. The ZHE is now ready for the 18 +/- 2 hr. rotation. Proceed to sec. 14.6.

14.6 Extraction:

14.6.1 Secure the samples in the rotation apparatus.

14.6.2 Rotation time is 18 +/- 2 hours at 30 +/- 2 rpm. A room temperature of 23 +/- 2^0C shall be maintained during the extraction period. Begin rotating. Record the time, rotation rate, and room temperature in the Extraction Summary Logbook (Table 3).

14.6.3 Following the rotation period, record the end time of rotation and the room temperature. Proceed to sec 14.7 for filtering the extract.

14.7 Filtration following extraction:

14.7.1 Following the rotation period record the date, time, and temperature. Check the ZHE pressure gauge to ensure that the device did not leak. If pressure was not maintained, the sample must be re-extracted with a new portion of waste sample (Document in prep logbook).

14.7.2 If pressure has been maintained, the material in the ZHE device is separated into liquid and solid phases.

14.7.3 For samples with no initial liquid phase**:**

14.7.3.1 Attach the 100 mL gastight syringe to the ZHE outlet valve. Open the valve and carefully withdraw the extract into the syringe. Do not allow air bubbles into the syringe. Pressurizing the ZHE to 10 psi may help in withdrawing the extract. Alternatively, a transfer line can be connected to the outlet valve on the ZHE and the sample extract can be transferred directly into the VOA vial.

14.7.3.2 Discard the first 5 mL out of the syringe, and then transfer the remaining extract to 40 mL VOA vials (without headspace). Store at 0-6^0C until analysis.

14.7.4 For samples containing an initial liquid phase**:**

If an initial liquid phase was collected, determine if the ZHE liquid extract will mix with the initial liquid phase:

14.7.4.1 Using a transfer pipette and a small beaker, add a few drops of the liquid phase to a small quantity of DI water. Observe to see if the two liquids are miscible, or if they separate into layers.

14.7.4.2 If the two phases are miscible, combine the filtered extract with the initial liquid phase and analyze as one sample. Attach the Tedlar bag containing the initial liquid phase to the outlet valve of the ZHE device. Gradually apply pressure to the lower valve and slowly

filter all of the extract into the Tedlar bag. Store at 0-6^{0}C until analysis.

14.7.4.3 If the two phases do not mix, the initial liquid phase and the filtered extract will need to be collected and analyzed separately. Collect all of the extract in a separate Tedlar bag.

14.8 Samples are now ready for analysis.

14.8.1 When the standard list and reporting limits are required the samples can be diluted at least 1:100 and still maintain the reporting limits. The dilution is recommended to reduce the amount of glacial acetic acid introduced into the GC/MS system in order to ensure a longer column lifetime. Samples can be analyzed undiluted but this greatly increases the peak for glacial acetic acid and will potentially shorten column lifetime and will increase the chances of background contamination.

14.8.2 Matrix spikes are prepared after extraction of the sample matrix and prior to analysis by Purge & Trap/GM-MS analysis.

14.8.2 Make sure all proper documentation was entered in the TCLP/SPLP Logbook (Table 3.

15.0 Calculations

15.1 Results are directly obtained from the analysis instrument in ug/L. If using the standard TCLP/SPLP list and reporting limits a dilution of 1:100 is typically perform. To obtain the final results calculate as follows:

$$\text{Results mg/L} = \frac{\text{Analytical result (ug/L) x DF}}{1000}$$

15.2 Calculate the results for multiphasic as follows:

$$\text{Final analyte concentration} = \frac{(V1)\,(C1) + (V2)\,(C2)}{V1 + V2}$$

V1 = volume of the first phase liquid
C1 = concentration of the first phase in mg/L
V2 = volume of the second phase liquid
C2 = concentration of the second phase in mg/L

15.3 See Procedure (14.0) for all other applicable calculations.

16.0 Method Performance

Certified standard solutions, properly maintained extraction equipment and instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external

performance test samples provided by the appropriately accredited agencies.

17.0 Pollution Prevention

See QAM Appendix 9

18.0 Data Assessment & Acceptance Criteria for QC Measures

18.1 If the initial analysis of a sample or a dilution of the sample has a concentration of a particular target analyte that exceeds the calibration range, the sample must be reanalyzed at a dilution that will keep compounds within the calibration range of the instrument.

18.2 Refer to the analysis method (SOP 8260B: *Analysis of Volatile Organic Compounds by GC/MS*) for additional analysis criteria.

19.0 Corrective Measures for Out-of-Control Data

See QAM Appendix 9

20.0 Contingencies for Handling Out-of-Control or Unacceptable Data

See QAM Appendix 9

21.0 Waste Management

See QAM Appendix 9

22.0 Equipment / Instrument Maintenance, Computer Hardware & Software & Troubleshooting

See QAM Appendix 9

23.0 References

23.1 Test Methods for Evaluating Solid Waste, EPA-SW-846. September, 1994. Method 1311.

23.2 Test Methods for Evaluating Solid Waste, EPA-SW-846. September, 1994. Method 1312.

23.3 CT Laboratories Quality Manual, current revision.

23.4 Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013 or most recent revision.

23.5 National Environmental Laboratory Accreditation Conference (NELAC), 2003 NELAC Standard Chapters 1 to 6, EPA/600/R-04/003, June 5, 2003 or most recent version.

23.6 ISO. 2005. General requirements for the competence of testing and calibration laboratories. ISO17025.

24.0 Tables

Table 1
Summary of Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
ZHE Pressure	Pressure applied and set @ 10 psi for all samples and blanks	Pressure must be maintained throughout the extraction process (pressure may increase due to affect from sample matrices)	Do not proceed with analysis until extraction meets criteria. Document problem and re-prepare sample for extraction.
Sample Rotation	30 rpm for 18 hours for all samples and blanks	Rotation rate: 30 ± 2 rpm. Rotation time: 18 ± 2 hours	Do not proceed with analysis until extraction meets criteria. Document problem and re-prepare sample for extraction.
Method Blank (MB)	1 / 20 samples per matrix or at contact/ program specific frequencies. The MB is used to document contamination resulting in the analytical process and shall be carried through the complete sample preparation and analytical procedure.	1. Concentration of analytes of concern shall be less than the highest of either : *Reporting Limit or MDL *Five percent of the measured concentration in the sample. 2. ACOE/QSM: ≤ ½ RL 3. Less than program/project specified limits.	Reanalyze to determine if instrument or laboratory background contamination was the cause. If the method blank is still non-compliant, re-prepare and reanalyze blank and samples.* For ACOE/QSM data if less than ½ MRL no action required.* *If reanalysis of blank still contains contamination above specified limits, affected data shall be qualified (B)

Table 2
FPR2-01
ZHE (Volatiles) Extraction Vessel Usage Log

ZHE (Volatiles)

Usage # >>>>
Blank Analyzed

Vessel #	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	Date / Batch #
1																					/
2																					/
3																					/
4																					/
5																					/
6																					/
7																					/
8																					/

****After 20 uses the vessel must be used for a blank check**

Table 3

FPR2-02
TCLP/SPLP/ASTM Extraction Summary

TCLP/SPLP/ASTM Extraction Summary

Batch:_____ Date:_____ *Analyst (set up):_____ Analyst (take down):_____*

Sample #	Parameter	Test			Vessel / ZHE	Buffer Determination			Sample wt.	**Free Liquids Present (Y/N)	Vol. Ext. Fluid	Initial pH	ZHE Initial Press	Time In	Date / Time Out	Ext. Time	End pH	ZHE End Press	Date / Time Filtered	Volume Filtered
	(M / SV / V / WC)	TCLP	SPLP	ASTM	#	Initial pH	End pH	*Ext. Fluid	(g)		(mL)		(psi)			(hrs)		(psi)		(mL)
															/				/	
															/				/	
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															/				/	

**Extraction Fluid Used:_____ Ext. Fluid ID #:_____ Ext. Fluid pH:_____ Ext. Start Temp (°C):_____*

Ext. End Temp(°C):_____ (min:_____ / max:_____) Filter Manufacturer:_____ Filter Lot #:_____

Balance:_____ pH Meter:_____ 1N HCL:_____ *Tumbler Unit #(s):_____*

Acceptable rotation rate: 30 ±2 rpm Verified Rotation rate: _____ rpm

M=Metals, SV=Semi Volatiles, V=Volatiles, WC=Wet Chemistry

Table 4

FPR2-03
% Solids Calculation Worksheet

% Solids Calculation Worksheet

Tare weights (g) *Transfer weights (g)* *Separation weights (g)* *% Solids Calculations*

Sample #	A Filter wt.	B Filtrate Vessel wt.	C Transfer Vessel wt.	D Sample + Transfer Vessel	E Sample wt {D - C}	F Vessel wt. Post Transfer	G Residue wt. {F – C)	H Total Waste Amount {E – G}	I Filtrate Vessel + Filtrate	J Liquid Phase of Sample {I – B}	K Solid Phase of Sample {H – J}	L. % Solids (**) (K/H*100)	M Waste + Filter (Dry) wt. (g)	% Solids (Dry) ({M – A}/H)*100

(**) If Solids = <0.5%, treat filtrate as TCLP extract, if free liquid present use % Solids Calculation Worksheet

Revision Number	Description of Changes	Date
02	Document changed to incorporated administrative requirements of ISO 17025 and QSM 5.0. Descriptions of changes have not been tracked in previous versions of this document.	03/12/2014
03	Added section 14.8.2 as to when matrix spikes can be prepared for analysis. And updated the TCLP/SPLP/ASTM Extraction Summary form to include min/max temperatures (Table 3).	03/31/2016

Attachment 4 Responses to USEPA and TDEC Comments

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

October 21, 2016

Carolyn Jones
Program Manager
Office of the Chief of Staff for Installation Management
Attn: BRAC Division (DAIM-ODB)
2530 Crystal Drive (Taylor Bldg.), Room 5000
Arlington, VA 22202-3940

Subject: **Membrane Interface Probe Survey Work Plan, Dunn Field**
Defense Depot Memphis, Tennessee
TDoR ID # 79-736
EPA ID # TN 4210020570

Ms. Jones,

TDEC-DoR has reviewed the **Membrane Interface Probe Survey Work Plan-Dunn Field** (August 2016), as compiled by Trinity Development & Analysis Corporation, and approves of the contents of the work plan. If there are questions or concerns, please contact me at (901) 371-3041 or at jamie.woods@tn.gov .

Regards,

Jamie A. Woods, P.G.
Project Manager
Division of Remediation
Memphis Environmental Field Office

cc: Todd Calhoun (Trinity)
Thomas C. Holmes (HDRInc)
Diedre Lloyd (EPA-PM)
Joan Hutton (CALIBRE)
TDoR NCO: file 79-736
TDoR MEFO: file 79-736

Responses to:
U.S. Environmental Protection Agency (EPA) Region 4 Comments on:
Uniform Federal Policy-Quality Assurance Project Plan
Membrane Interface Probe Survey Work Plan
Dunn Field, Defense Depot Memphis, Tennessee
August 2016 (Revision 0)

EPA GENERAL COMMENTS

1. In general, the Work Plan does not fully define the problem that the Membrane Interface Probe (MIP) Survey will address at the Defense Depot Memphis Tennessee (DDMT) and how this investigation effort fits within the overall framework of the investigation and remediation efforts underway at the DDMT. For example, Worksheet 10, Problem Definition provides a brief description and history of the DDMT, but does not state what the project objectives and goals are for the site. The Previous Investigations section does not address the previous remediation efforts at Dunn Field and their results. This is key to defining the current problem and explaining why further investigation is needed to fill data gaps. The conclusion for Worksheet 10, Problem Definition should explain in general terms the specific data gap to be addressed by this additional investigation (the MIP Survey). Revise the Work Plan text to provide the requested detail to address this issue.

   Response: The UFP-QAPP Work Plan, specifically Worksheets #10 and #11, has been revised to better clarify the project objectives and goals, introduce the data gaps this investigation intends to address, and how the data will be used and evaluated.

EPA SPECIFIC COMMENTS

1. **Worksheet 10 – Problem Definition, Page 10-1:** The project objectives and goals for the DDMT should be added to the end of the Site Location and History section of Worksheet 10. Consider adding a Table with the maximum contaminant levels (MCLs) for the key contaminants of concern.

   Response: Project objectives for MIP Survey have been added to Worksheet #10. Table 5 Maximum CVOC Concentrations in Background-NE Monitoring Wells – April 2015 has been added to the text identifying the MCLs, location of maximum detected concentrations of PCE, TCE, and 1,1-DCE in Background-NE well from last reported sampling event (April 2015).

2. **Worksheet 10 – Problem Definition, Previous Investigations, Page 10-1:** A brief history of the remediation efforts to date (e.g.; removal of soils, Soil Vapor Extraction and Air Sparging, In-situ Thermal Desorption, disposal units, etc.) at Dunn Field and the results are not presented in this worksheet. As such, in support of the formulation of the problem definition add a discussion of previous investigations and long term monitoring (LTM) results in Worksheet 10.

Response: Remedial actions conducted under the RODs have been limited to Disposal Areas and Source Areas on Dunn Field. No remedial actions have been conducted to date in the study area of this MIP Survey. However, information on remedial actions for the Disposal Sites and Source Areas have been added to the text as requested.

3. **Worksheet 10 – Problem Definition, Previous Investigations, Page 10-1:** The five monitoring wells that had the tetrachloroethylene (PCE) and trichloroethylene (TCE) results above maximum contaminant levels (MCLs) in April 2015 are not identified in the worksheet. Additionally, while Figure 4 depicts total chlorinated volatile organic compounds (CVOC) concentrations, the respective concentrations of the PCE and TCE MCL exceedances in the five monitoring wells is not presented. As such, it is recommended a table is added summarizing the respective MCLs and the results for the monitoring wells. Adding results for several LTM events will also help with formulating the Problem Definition. Revise the Work Plan to address this issue.

   Response: Figure 4 has been revised to provide PCE, TCE, 1,1-DCE concentrations from the last 4 sampling events (2012-2015) for MW-07, MW-08, MW-129, MW-130, and MW-230. As discussed in the response to Specific Comment #1, a table has been added to Worksheet #10 text providing the MCL, and location of maximum detected concentration of PCE, TCE, and 1,1-DCE from the April 2015 sampling event. Note that the original Figure 4 is now presented as Figure 5.

4. **Worksheet 10 – Problem Definition, Page 10-2:** At the end of Worksheet 10, add an explanation of the specific data gap to be addressed by this additional investigation (the MIP Survey) and how the MIP Survey will address the problem with determining the source of contamination of the groundwater.

   Response: Text has been revised to include a subsection for Data Gaps. The data gap has been identified as the lack of an identified source (on or off-site) for the groundwater contaminants observed above MCLs in the Background-NE monitoring wells.

5. **Worksheet 11** – **Data Quality Objectives,** Data Quality Objectives do not meet the format or content requirements specified in EPA's *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA QA/G-4) dated February 2006 [DQO Guidance], and therefore are not sufficient to define the type, quantity, or quality of data needed to meet the project objective to determine where soil samples are needed to quantify the MIP Survey results. Please revise worksheet 11 to list the seven step DQO process, which includes 1) State The Problem; 2) Identify the Goals of the Study; 3) Identify Information Inputs; 4) Define the Boundaries of the Study; 5) Develop the Analytical Approach; 6) Specify Performance or Acceptance Criteria; and 7) Develop the Plan for Obtaining Data. Please revise Worksheet 11 to include descriptive and specific information that meets the guidelines of the DQO Guidance and which details how the sampling and analysis plan will be developed to obtain data which meet the project objective.

   Response: Worksheet #11 has been revised to follow the seven step DQO process and provide additional details as requested.

6. **Worksheet 11 – Data Quality Objectives, Question 2, Page 11-1:** The answer to the question does not clarify the scope of the investigation. As such, revise the answer statement as follows to clarify the scope of the investigation is just within the north east corner of Dunn Field. "The data will be used to determine if an on-site source of contamination does exist in the northeast corner, within the boundaries of the MIP Survey at Dunn Field."

   Response: Note: Worksheet #11 has been revised per Specific Comment #6. Revised text applicable to this comment has been incorporated into the revised Worksheet #11, DQO #2.

7. **Worksheet 11 – Data Quality Objectives, Question 3, Page 11-1:** For clarity regarding the MIP survey methodology, delete "the MIP survey will" and add the following text after "MIP:" "Field screening data will be collected using a Geoprobe to direct push MIP Survey instrumentation into the soil to collect and analyze gas samples…".

   Response: Note: Worksheet #11 has been revised per Specific Comment #7. Revised text applicable to this comment has been incorporated into the revised Worksheet #11, DQO #5.

8. **Worksheet 11 – Data Quality Objectives, Question 6, Page 11-1:** Please add the estimated depth that the MIP Survey instrumentation will be pushed into the soil for sample collection and describe how the decision for the actual depth of the sample collections will be made in the field.

   Response: Note: Worksheet #11 has been revised per Specific Comment #8. Revised text applicable to this comment has been incorporated into the revised Worksheet #11, DQO #4 and DQO #5.

   Text stating that the MIP investigation will be limited to the loess (estimated maximum thickness of 30 feet) has been added to the text for clarification. Additional details regarding monitoring of the MIP electrical conductivity sensor for changes in lithology and the MIP temperature sensor for changes in temperature (groundwater) have also been added to text.

   a. The WP Data Quality Objectives in Worksheet 11 are insufficiently detailed and do not include the decision criteria that will be used to determine where soil samples should be collected. Please revise and include the requested information.

      Response: Note: Worksheet #11 has been revised per Specific Comment #8a. Revised text applicable to this comment has been incorporated into the revised Worksheet #11, DQO #5.

      Soil samples will be collected over full range of ECD readings to correlate ECD and analytical results, with slightly more samples for high ECD readings.

   b. Worksheet 11 does not include a list of the screening and/or regulatory limits that will be used to determine where soils samples are needed to quantitate the MIP Survey

results. Revise the Work Plan to include this information. Please revise and include the requested information.

Response: Note: Worksheet #11 has been revised per Specific Comment #8b. Revised text applicable to this comment has been incorporated into the revised Worksheet #11, DQOs #5 and 6.

Text revised to state that soil analytical results will be screened against the Remediation Goals for Site-Specific Soil Screening Levels to be Protective of Groundwater (Loess Specific Values) established in the Dunn Field ROD (CH2M Hill, 2004). These values have been added to Worksheet #15.

9. **Worksheet 12** – **Measurement Performance Data**, the Measurement Performance Criteria (MPC) does not appear to include any quantitative MPC for the MIP analyses, therefore it is unclear how the results of such analyses will be substantiated as accurate and representative. Please revise and include the requested information.

   Response: The MIP sensors are considered field screening tools and therefore no MPCs exist. As noted in response to Specific Comment 8b, contaminant concentrations cannot be directly determined from the MIP detector responses. Concentrations will be determined from soil samples and the results will be used to correlate ECD results and CVOC concentrations in soil for the study area.

10. **Worksheet 15** – **Reference Limits and Evaluation**, this worksheet does not include the screening or regulatory limits that the MIP data will be compared to, therefore it is unclear whether the laboratory detection limits are sufficient to meet the project objectives. Please revise the worksheet to include the requested information.

    a. Worksheet 15 does not include a list of the screening and/or regulatory limits that will be used to determine where soils samples are needed to quantify the MIP Survey results. Please revise the Work Plan to include the requested information.

       Response: As noted in response to 8a, soil samples will be collected over full range of ECD readings to correlate ECD and analytical results. The sample results and correlated MIP results will be compared to the Remediation Goals for Site-Specific Soil Screening Levels to be Protective of Groundwater (Loess Specific Values) established in the Dunn Field ROD, which have been added to Worksheet #15.

    b. Worksheet #15 does not include the screening or regulatory limits that the MIP data will be compared to, therefore it is unclear whether the laboratory detection limits are sufficient to meet the project objectives. Revise the Work Plan to address this issue.

       Response: The remediation goals have been added to Worksheet #15 and are well above the laboratory detection limits.

11. **Worksheet 17 – Sampling Design and Rationale, Page 17-1:** This worksheet states that up to fifteen soil samples will be collected for off-site analysis at a laboratory based on the MIP

results to verify the absence or presence of volatile organic compounds (VOCs) in soil at the site. However, the text does not explain how a limit of fifteen soil samples was obtained or why this number of samples is sufficient for determining where the contamination exists or originated at the site. Please revise Worksheet 17 to describe how the limit of 15 samples for off-site analysis at a laboratory was determined and why collecting this number of samples is sufficient for supporting decision making at the site regarding the absence or presence of VOCs in soil.

Response: The number of samples of fifteen was selected to represent approximately ¼ of the number of MIP locations and was included in the Scope of Work issued to Trinity Analysis & Development Corp. as a budgetary number for execution of this task order. As stated in the text, the soil samples will be used to quantitate the MIP survey results and will be collected from areas of elevated responses and no responses. This approach is consistent with the MIP investigation conducted as part of the Main Installation Source Area Investigation (e2M, 2009) where soil samples were collected from varying levels of MIP responses.

Worksheet 17, Soil Boring/Sample Locations revised as follows: "Soil samples will be collected from intervals identified during the MIP investigation. Up to 15 soil samples will be collected for VOC analysis by USEPA Method 8260C by CTL to provide definitive analytical results at 25% of the MIP locations. The analytical results will be used to correlate CVOC concentrations to MIP-ECD results. The analytical results will be used to verify the absence or presence of soil contamination, and quantify, if present, so samples will be collected across a range of MIP responses, including no response. Detection of the target CVOCs above remediation goals in soil samples or correlated ECD results will serve as indication of contaminants in soil and will be used to make recommendations for further actions, as necessary."

12. **Figure 8, MIP Boring Layout,** Please explain the cluster of two points indicated in the figure in the upper northwesterly direction on this figure. It appears to be an anomalous configuration of MIP borings.

    Response: Figure has been revised to adjust the boring layout (now presented as Figure 9). The clustered points in the northwestern portion of the original figure were due to automatic placement of the points within a generated grid. Primary selected location was the northwestern quadrant of each grid and in the absence of that location due to boundary conditions (fence line in this case), the default location was the southwestern quadrant of the grid. This boring layout has been modified to eliminate irregular spacing of borings.

Responses to:
U.S. Environmental Protection Agency (EPA) Region 4 Comments on:
Uniform Federal Policy-Quality Assurance Project Plan
Membrane Interface Probe Survey Work Plan
Dunn Field, Defense Depot Memphis, Tennessee
November 2016 (Revision 1)

Responses to additional Comments 1 – 3 assume that the reference to Table 15 is intended to be Worksheet 15.

1) **Can you please add remedial goals and MCLs to Table 15?**

   Response: Remedial goals for Dunn Field are included as the Soil Screening Objectives (Remediation Goals for Site-Specific Soil Screening Levels to be Protective of Groundwater, Loess Specific Values) in Worksheet 15 Table 9 provided in Rev. 1 response to initial EPA comments.
   MCLs not provided since the Work Plan is limited to soil investigation.

2) **What is meant by loess specific values?**

   Response: The loess specific values represent site-specific values of soil concentration that would be protective of groundwater at Dunn Field. These values were established in the 2004 Dunn Field Record of Decision (CH2M Hill) and are provided on Table 2-21B (EMSOFT Calculated SSL) and 2-21G (Site-Specific Soil Screening Levels to be Protective of Groundwater) of the ROD.

3) **How were the screening levels delineated in Table 15 determined?**

   Response: 2004 Dunn Field ROD (Section 2.7.3) states that the EPA Exposure Model for Soil-Organic Fate and Transport (EMSOFT) (EPA 1997) was used to calculate site-specific values of soil concentration for loess and fluvial deposits that would be protective of groundwater at Dunn Field. Full discussion of the calculation of the site-specific remediation goals that would be protective of groundwater at Dunn Field is provided in Appendix C of the Dunn Field Feasibility Study.

4) **The text states that 15 samples (or 25%) of the gridded samples will be chosen for correlation to the ECD readings obtained from the MIP with a bias toward high samples**
   a. **If a sampling of ~25% is an intended target for correlative purposes, then the number of samples should be closer to 17 samples. Will DDMT budget allow for the additional collection of 2 samples?**

      Response: The MIP Survey area (Figure 9) has been revised to include only 60 sampling points (~40 foot x 40 foot grid), therefore 15 samples meets the 25% target.

   b. **Please provide detailed information that supports the proposal to send 25% samples collected to an outside lab for the correlative purposes with all MIP values collected while in the field.**

      Response: Soil sampling at 25% of the MIP Locations is designed to be consistent with the goal stated for the 2009 Main Installation Source Area Investigation, Section 2.0. This

reference has been added to Worksheet 13 Table 8 – Secondary Data Criteria Limitations and Worksheet 17.

There is no definitive statistical basis for the target goal of 25%. The EPA CLU-IN guidance for MIP provides an overview of qualitative assessment of MIP responses for quality control and accuracy but does not provide guidance on determining the number of soil samples best for performing this assessment.
https://clu-in.org/characterization/technologies/mip.cfm

c. Please include how the chosen samples will be determined (ie: the criteria that will be used to choose the samples to send to outside lab), and how correlation between the MIP ECD data and the laboratory data will be determined.

Response: The target intervals for soil samples will be selected to include the full range of ECD readings in the study area, but with a bias toward high readings. The laboratory results will be plotted against the neighboring MIP responses to determine the relationship of concentration versus MIP response. Please see response to 4.e. for additional information.

d. The text outlines the collection of 15 samples over 30 foot intervals at each of the 70 grid locations as depicted in attached figures.

Response: Text describes the collection of 15 soil samples at selected locations based on MIP responses. MIP grid has been revised to include a total of 60 MIP points. Within the main survey area, the spacing is approximately 40 feet x 40 feet.

i. Please outline how MIP readings will be taken, specifically will MIP readings be taken continuously or at a predefined foot interval? If at a predefined foot interval – what interval?
Response: The following text added to Worksheet 14, MIP Borings – "Target operation of the MIP tooling will be as follows based on manufacturer recommendation. The MIP probe will be advanced in 1-foot increments at a rate of approximately 0.5 feet/second. The probe will be stopped at each 1-foot interval for 45 seconds for the MIP block and membrane to heat up and for consistent sample collection. The membrane will also be exposed for 45 seconds in the response test and which is approximately the length of time required to add a drill rod. Stopping at each interval for 45 seconds keeps the advancement and membrane exposure consistent. The professional judgement of the MIP operator will be used to determine if a response above instrument detection levels exists."

ii. Please explain how sampling locations will be determined with respect to depth at each of the 70 grid locations.
Response: Worksheet 14, Soil Borings/Sampling – "The locations and target depths of the soil borings will be based on the MIP results and will be used to confirm both absence and presence of detected results. The number and locations of borings advanced will be determined in the field based on the observed MIP results which could allow for the collection of soil samples from multiple intervals within the same boring."
The following text added to Worksheet 14, Soil Borings/Sampling – "The field geologist in communication with the project team will review the 'real-time' MIP data and concur on soil sample locations and depths."

e. Please state the number of samples that will:

Response: In response to comments e.i., e.ii, and e.iii, during the MIP survey performed as part of the Main Installation Source Area Investigation (e^2m, 2009), the ECD responses of those borings were categorized as 0 – 0.3 Volts, 0.3 – 3 Volts, and >3 Volts. This grouping was based on results of the initial Dunn Field survey performed by CH2M Hill. For the Main Installation Source Area Investigation, about half the borings had ECD responses above 3 Volts and half between 0.3 and 3; very few locations with ECD responses below 0.3 Volts.

As indicated in response to 4.c, the target intervals for soil samples will be selected to include the full range of ECD readings in the study area, but with a bias toward high readings. Based on the lack of historical reference data and expected responses in this area of Dunn Field, the "measurability" of a response cannot be scaled to determine a category until the data is collected. Once the full data is collected, it can be plotted to see where the bulk of the responses fall.

i. **Collected for non-detect (ie: low readings) = # samples?**
ii. **Collected for high level = # samples?**
iii. **Collected for medium level - # samples?**
iv. **Which samples will be chosen for duplicates and how many will be chosen for duplicates? = high, low, mid-level? And what number?**

Response: Consistent with Worksheet 18 Table 11, two field duplicates will be collected. One sample each will be collected from the medium level and high level response categories.
One MS/MSD pair will be collected from the low/no response category.

v. **What criteria will be used to determine where to sample and at what level?**

Response: The professional judgement of the MIP operator will be used to determine if a response above instrument detection levels exists. The field geologist in communication with the project team will review the "real-time" MIP data and concur on soil sample locations and depths.

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

January 10, 2017

FED-EX
RETURN RECEIPT REQUESTED

Mr. James Foster
Assistant Chief of Staff for Installation Management
Base Realignment and Closure Division (ACSIM-ODB)
2530 Crystal Drive (Taylor Building), Room 5000
Arlington, VA 22202-3940

Dear Mr. Foster:

The U.S. Environmental Protection Agency (EPA) has received and reviewed the Department of the Army, Defense Depot of Memphis Depot Uniform Federal Policy-Quality Assurance Project Plan Membrane Interface Probe Survey Work Plan, dated August, 2016 along with the additional 2 rounds of comment responses and teleconference on January 10, 2017.

EPA provides approval for the above referenced report along with the submitted comment responses and appreciates the U.S. Army's efforts to address U.S. EPA's concerns. If you have any questions or concerns, please contact me at (404) 229 -9500.

Sincerely,

Diedre Lloyd
Remedial Project Manager
Restoration & Sustainability Branch
Superfund Division

cc: Mr. James Foster, (Signed Original), United Parcel Service, Return Receipt
Mr. Jamie A. Woods, PG, Tennessee, Department of Environment and Conservation, Memphis Environmental Field Office, 8383 Wolf Lake Drive, Bartlett, TN 38133-4119
Ms. Joan Hutton, CALIBRE, 3898 Mountain View Road, Kennesaw, GA 30152
Mr. Thomas Holmes, HDR Environmental, P.O. Box 728, Highlands, NC 28741