Sampling and Analysis Plan Addendum 01, Investigation and Remediation of Releases and Groundwater Protection and Evaluation, Red Hill Bulk Fuel Storage Facility

JOINT BASE PEARL HARBOR-HICKAM, O‘AHU, HAWAI‘I

Administrative Order on Consent in the Matter of Red Hill Bulk Fuel Storage Facility, EPA Docket Number RCRA 7003-R9-2015-01 and DOH Docket Number 15-UST-EA-01, Attachment A, Statement of Work Section 6.2, Section 7.1.2, Section 7.2.2, and Section 7.3.2

September 1, 2017
Revision 00

Comprehensive Long-Term Environmental Action Navy
Contract Number N62742-12-D-1829, CTO 0053

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September 1, 2017
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ACRONYMS AND ABBREVIATIONS

%	percent
%D	percent difference
°C	degree Celsius
^{13}C	carbon-13 isotope
AOC	Administrative Order on Consent
ASTM	ASTM International
BFB	4-bromofluorobenzene
bgs	below ground surface
BTEX	benzene, toluene, ethylbenzene, and xylenes
C	carbon
CA	corrective action
CCV	continued calibration verification
CF&T	contaminant fate and transport
COPC	chemical of potential concern
CSIA	compound-specific isotope analysis
CSM	conceptual site model
DCA	1,2-dichloroethane
DDT	dichlorodiphenyltrichloroethane
DFTPP	decafluorotriphenylphosphine
DL	detection limit
DNA	deoxyribonucleic acid
DoD	Department of Defense, United States
DQI	data quality indicator
DQO	data quality objective
EA/IRMS	elemental analyzer/isotope ratio mass spectrometry
EAL	Environmental Action Level
EDB	1,2-dibromoethane (ethylene dibromide)
ELAP	Environmental Laboratory Accreditation Program
EPA	Environmental Protection Agency, United States
EPH	extractable petroleum hydrocarbon
ft	foot/feet
GC-FID	gas chromatography-flame ionization detector
GC-MS	gas chromatography-mass spectrometry
GC-TCD	gas chromatography/thermal conductivity detector
H_2SO_4	sulfuric acid
HCl	hydrochloric acid
HNO_3	nitric acid
ICAL	initial calibration
ICV	instrument calibration verification
IDW	investigation-derived waste
inHg	inches of mercury
IS	internal standard
JP	Jet Fuel Propellant
L	liter

LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LNAPL	light non-aqueous-phase liquid
LOD	limit of detection
LOQ	limit of quantitation
MA	Massachusetts
MB	method blank
mL	milliliter
MNA	monitored natural attenuation
MS	matrix spike
MSD	matrix spike duplicate
MWIWP	Monitoring Well Installation Work Plan
N	nitrogen
N/A	not applicable
$NaHSO_4$	sodium hydrogen sulfate
NAP	natural attenuation parameter
NAPL	non-aqueous-phase liquid
NAVFAC	Naval Facilities Engineering Command
NELAP	National Environmental Laboratory Accreditation Program
NGS	Next-Generation DNA Sequencing
NIST	National Institute of Standards and Technology
no.	number
NSZD	natural source-zone depletion
PAH	polynuclear aromatic hydrocarbon
PAL	project action level
PFTBA	perfluorotributylamine
PID	photoionization detector
PQO	project quality objective
QA	quality assurance
QC	quality control
qPCR	quantitative polymerase chain reaction
QSM	Quality Systems Manual
RF	response factor
RL	reporting limit
RPD	relative percent difference
RRT	relative retention time
RSD	relative standard deviation
RT	retention time
SAP	Sampling and Analysis Plan
SGC	silica gel cleanup
SIM	selective ion monitoring
SIP	Stable Isotope Probing
SOP	standard operating procedure
SVMP	soil vapor monitoring point
SVOC	semivolatile organic compound

1	TBD	to be determined
2	TCE	trichloroethylene
3	TCEQ	Texas Commission on Environmental Quality
4	TIC	tentatively identified compound
5	TOC	total organic carbon
6	TPH-d	total petroleum hydrocarbons – diesel range organics
7	TPH-g	total petroleum hydrocarbons – gasoline range organics
8	TPH-o	total petroleum hydrocarbons – residual range organics (i.e., TPH-oil)
9	TX	Texas (Texas Commission on Environmental Quality)
10	VOA	volatile organic analysis
11	VOC	volatile organic compound
12	VPH	volatile petroleum hydrocarbon

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1. Introduction

Sampling described in this *Sampling and Analysis Plan* (SAP) *Addendum 01* addresses the analytical program for the proposed new monitoring wells (including multi-level Westbay Systems) to be installed under the *Monitoring Well Installation Work Plan* [MWIWP] *Addendum 02* (DON 2017d) and the attenuation and source studies proposed in the *Attenuation Evaluation Plan* (DON 2017e) in support of the conceptual site model (CSM) and contaminant fate and transport (CF&T) modeling as part of the investigation performed under the Administrative Order of Consent (AOC) Statement of Work Section 6 and Section 7 (EPA Region 9 and DOH 2015).

This SAP Addendum details the analytical program, field study, and sampling procedures for Red Hill's existing and newly proposed groundwater monitoring wells in support of the CSM, the attenuation and source studies proposed in the *Attenuation Evaluation Plan* (DON 2017e), and the CF&T model.

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2. Sampling Rationale

2.1 MULTI-LEVEL WELL SAMPLING

The proposed new groundwater monitoring wells will be installed with multi-level sampling systems manufactured by Westbay Instruments (unless contingency measures are needed, as specified in the *MWIWP Addendum 02* [DON 2017]). Multi-level wells are constructed differently from the previously installed conventional monitoring wells in the Red Hill groundwater monitoring network, which are screened at the surface of the water table. The multi-level wells are expected to be installed with three to ten monitoring zones to profile the saturated zone ranging from the water table surface to approximately 300 feet (ft) below the water table. Multi-level wells will allow for evaluation of preferential pathways, hydraulic conductivities, groundwater flow directions (including vertical gradients), and groundwater chemistry in deeper portions of the basal aquifer.

Table 2-1 presents the proposed monitoring wells to be installed as multi-level well systems, and their locations are shown on Figure 1.

Table 2-1: Proposed Multi-Level Wells

Proposed Multi-Level Well (Westbay MP38)	Surface Elevation (ft msl)	Estimated Depth to Bedrock (ft bgs)	Estimated Depth to Groundwater (ft bgs)	Open Borehole Interval (ft bgs) [a]	Estimated Total Depth (ft bgs)
RHMW01R [b]	103 [c]	N/A	84 [d]	~30–384 [e]	384
RHMW07D	200	25–60	180	170–480	480
RHMW11	230	180–210 [f]	210	200–510	510
RHMW12	243	25–60	223	213–523	523
RHMW13	260	25–60	240	230–540	540
RHMW14	190	140–160 [f]	170	160–470	470
RHMW15	320	25–60	300	290–600	600
RHMW16	260	25–60	240	230–560	540
RHMW17	160	100–180 [f]	140	130–440	440
RHMW18	145	85–165 [f]	125	115–425	425
RHMW19	420	25–60	400	390–700	700
RHMW20	400	25–60	380	370–680	680

bgs below ground surface
N/A not applicable
[a] Based on open borehole 10 ft above water table.
[b] RHMW01R will be installed as a multi-level well only if perched water or evidence of contamination is not encountered in the vadose zone or if light non-aqueous-phase liquid (LNAPL) is not encountered at greater than 0.25-inch thickness on the water table. If these conditions are encountered, RHMW01R will be installed as a conventional monitoring well and sampling will be conducted in accordance with *SAP Revision 01* (DON 2017c).
[c] RHMW01R surface elevation is based on the Facility lower tunnel floor, which is located in bedrock at approximately 312 ft bgs.
[d] Estimated based on groundwater levels in RHMW01.
[e] Depth represents feet below the lower tunnel floor. Casing will end higher in RHMW01R to accommodate soil vapor sampling ports with the Westbay system.
[f] Estimated depth to bedrock is based on Figure 25 in (Wentworth 1942).

The multi-level wells will be included in the groundwater monitoring well network for the AOC Statement of Work Sections 6 and 7 investigation, and will be sampled for the chemical of potential concern (COPC) list identified for existing monitoring wells in accordance with the February 4, 2016, scoping completion letter for AOC Statement of Work Sections 6 and 7 (DON 2017a, Appendix A) and as previously identified in *SAP Revision 01* (DON 2017c).

2.2 SOURCE STUDIES

For the CSM and the CF&T model, it is important to better understand the natural source-zone depletion (NSZD) and monitored natural attenuation (MNA) processes that reduce the mass of fuel in the vadose zone and the groundwater beneath the tank, respectively. To develop a detailed conceptual model of fuel migration and degradation, it is important to understand the composition of fuel at the point of release. Degradation or persistence of the fuel components will be evaluated by comparing the fuel component concentrations at the source to those at downgradient locations (if present). To address NSZD and MNA, source studies will include fuel forensic analyses, TPH fractions analyses, biological testing, analyses for additional natural attenuation parameters (NAPs), compound-specific isotopes analyses, temperature profiles of select groundwater monitoring locations, soil vapor concentrations in the soil vapor sampling points, groundwater monitoring locations, and RHMW01R vadose zone, and a carbon flux study to provide lines of evidence to support presence and potential of biodegradation occurring. The additional analytical program is presented in Table 2-2 and on Figure 2.

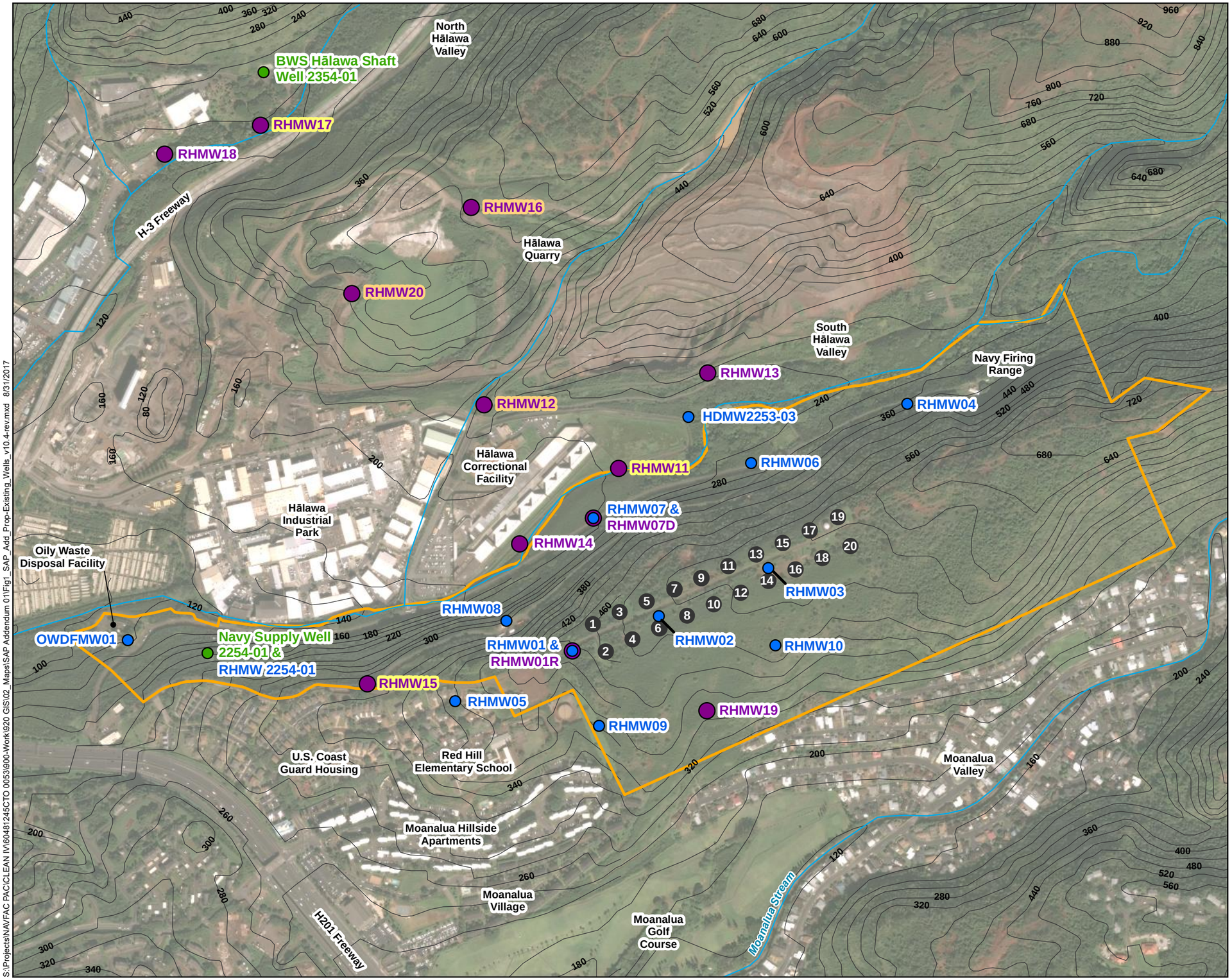
2.2.1 Forensic Analyses and TPH Fractions

To provide further inputs to the CF&T modeling, an analysis to evaluate the chemical composition and carbon fraction distribution will be performed on light non-aqueous-phase liquid (LNAPL) sheen samples from RHMW02 and RHMW03; and on groundwater impacted by TPH at the site.

Forensic and TPH fractions analyses will be performed on sheen samples from RHMW02 and RHMW03 and groundwater collected from RHMW01, RHMW02, and RHMW03 at a minimum, based on long-term groundwater monitoring data showing consistent TPH detections at these locations. Tank bottom water (water collected at the bottom of the tanks [containing JP-5 or F-24] from condensed moisture in tanks or from fuel contamination) will also be collected (if possible) and analyzed to evaluate the partitioning of fuel compounds in water. Forensic and TPH fractions analyses will be performed during at least one groundwater monitoring event for groundwater samples and during at least three events for tank bottom water. Analysis of groundwater samples from other monitoring wells will be contingent on the presence of TPH at those wells.

Forensic analyses will include the following:

- Groundwater samples with positive detections of TPH and in tank bottom water, including:
  - Identifying the specific highest concentration analytes in the volatile and semivolatile range of organic compounds using United States Environmental Protection Agency (EPA) Method 8260 and EPA Method 8270, respectively, and reporting at least the top ten tentatively identified compounds (TICs).
  - Identifying the specific highest concentration analytes (including TICs) in the semivolatile range of organic compounds in the silica-gel-cleaned extract using EPA Method 3630 and analyzed by EPA Method 8270 to evaluate compounds that may be biodegradation by-products.
  - Evaluating TPH polar and non-polar fractionation of TPH-diesel range organics (TPH-d) and TPH-residual range organics (TPH-o) using silica gel cleanup (SGC) by EPA Method 3630 and analyzed by EPA Method 8015.
  - Identifying the parent and alkylated polynuclear aromatic hydrocarbons (PAHs) using EPA Method 8270 Selective Ion Monitoring (SIM) Mod.



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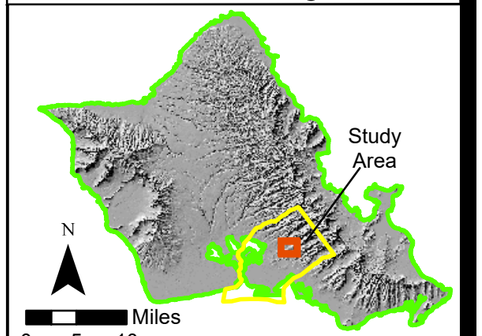
Groundwater Modeling Domain



Legend

- Proposed Monitoring Well Location
- Existing Monitoring Well Location
- Proposed and Existing Monitoring Well Adjacent Locations
- Existing Water Supply Well
- Red Hill Fuel Storage Tank
- Stream
- Topographic Contour (feet above mean sea level)
- Red Hill Installation Boundary
- Regional Model Boundary
- Local Model Boundary

Notes

1. Map projection: NAD 1983 UTM Zone 4N
2. Base Map: DigitalGlobe, Inc. (DG) and NRCS. Publication_Date: 2015
3. Coordinates: NAD 1983 UTM Zone 4N
4. RHMW-- indicates well identified for priority installation.
5. RHMW-- indicates three potential wells, one of which will be identified for priority installation.

N

0 250 500 1,000 Feet

Figure 1
Proposed and Existing Monitoring Well Locations
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Investigation and Remediation of Releases and Groundwater Protection and Evaluation
Red Hill Bulk Fuel Storage Facility
JBPHH, O'ahu, Hawai'i

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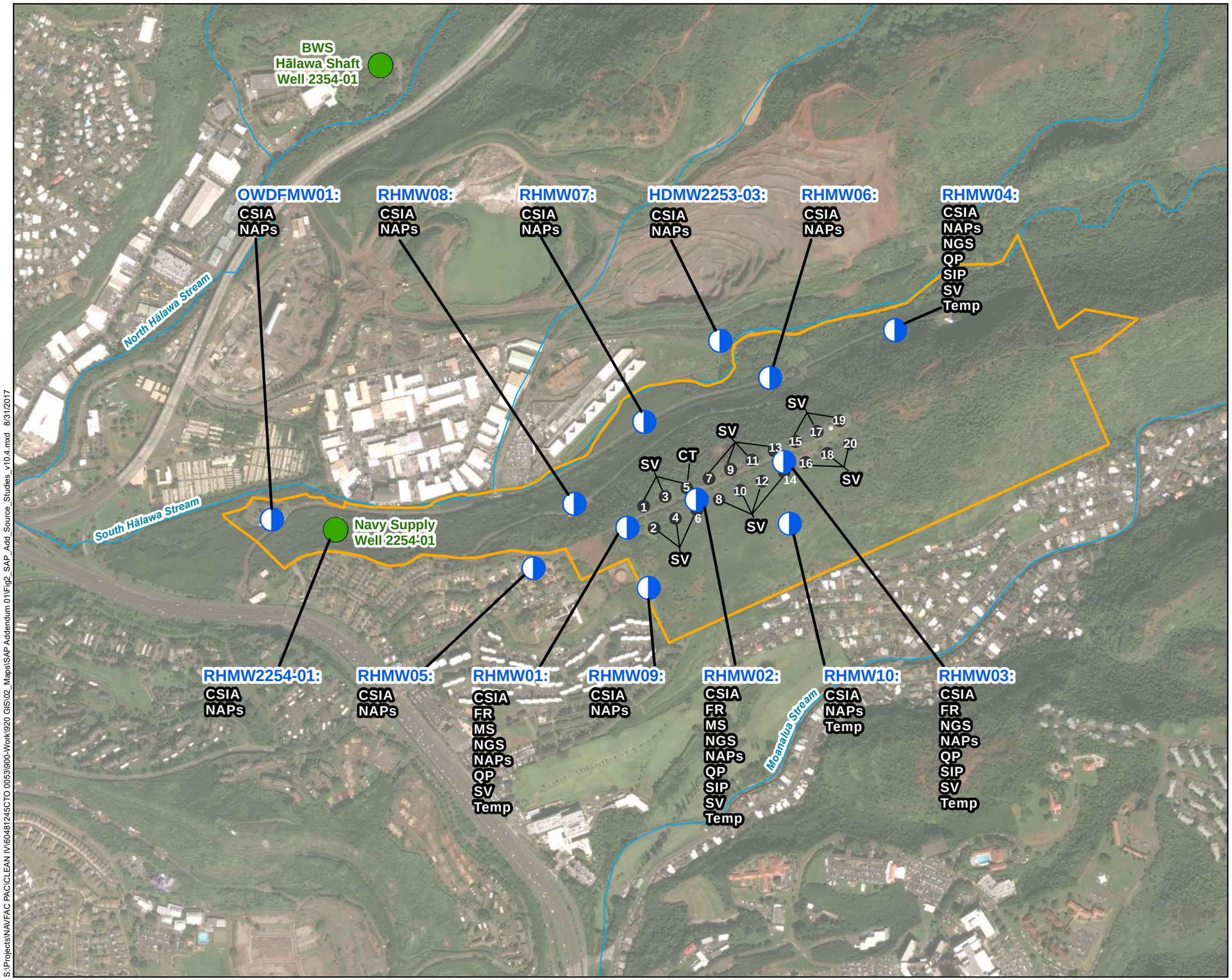

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Legend

- Existing Monitoring Well Location
- Existing Water Supply
- Red Hill Fuel Storage Tank
- Stream
- Red Hill Bulk Fuel Storage Facility Boundary

ACRONYMS:

CSIA	Compound-Specific Isotope Analysis
CT	Carbon Trap
FR	Forensic and TPH Fractions Analyses
MS	Microcosm Study
NGS	Next-Generation Sequencing
NAPs	Natural Attenuation Parameters
QP	QuantArray-Petro
SIP	Stable Isotope Probing
SV	Field Gases and VOCs
Temp	Temperature Probes

Notes

1. Map projection: NAD 1983 UTM Zone 4N
2. Base Map: DigitalGlobe, Inc. (DG) and NRCS. Publication_Date: 2015
3. SIP will be performed using ^{13}C-labeled Naphthalene and ^{13}C-labeled Benzene compounds conducted during separate sampling events. ^{13}C-labeled Benzene will not be sampled at RHMW04.
4. CSIA will be performed using nitrogen and sulfur isotopes.
5. Forensic and TPH fractions analyses will be performed at RHMW01, RHMW02, and RHMW03 at a minimum. Samples from other monitoring wells will be collected based on presence of TPH in the groundwater.

N

0 500 1,000 1,500 2,000 Feet

Figure 2
Source Study Sampling Locations
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Investigation and Remediation of Releases and Groundwater Protection and Evaluation
Red Hill Bulk Fuel Storage Facility
JBPHH, O'ahu, Hawai'i

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Table 2-2: Additional Analytical Program

Type	Parameter	Analytical Method	Analyte(s)	Monitoring Well													Tank water or Shake Test (Water)	Soil Vapor Monitoring Points	RHMW01R Vadose Zone Monitoring Points
				RHMW2254-01	RHMW01	RHMW02	RHMW03	RHMW04	RHMW05	RHMW06	RHMW07	RHMW08	RHMW09	RHMW10	OWDFMW01	HDMW2253-03			
Detailed and forensic analyses	VOCs [a]	EPA 8260	Full suite VOCs with TICs	C [b]	✓	✓	✓	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	✓		
	SVOCs [a]	EPA 8270	Full suite SVOCs with TICs	C [b]	✓	✓	✓	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	✓		
			Full suite SVOCs with TICs (SGC extract)																
	TPH-d/o	EPA 8015	TPH-d, TPH-o	✓[c]	✓[c]	✓[c]	✓[c]	✓[c]	✓[c]	✓[c]	✓[c]	✓[c]	✓[c]	✓[c]	✓[c]	✓[c]	✓[a]		
			TPH-d, TPH-o (SGC extract)	C [b]	✓[c]	✓[c]	✓[c]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	✓[a]		
	Detailed hydrocarbon analysis [a]	EPA 8270 SIM Mod.	Parent and Alkylated PAHs	C [b]	✓	✓	✓	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	✓		
	Sheen sampler [a]	Shell procedure, ASTM D3328	C3–C44 Whole Oil			✓	✓												
TPH fractions	TPH fractions [a]	MADEP VPH	C5–C8 Aliphatics, C9–C12 Aliphatics, C9–C10 Aromatics	C [b]	✓	✓	✓	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	C [b]	✓		
		MADEP EPH	C9–C18 Aliphatics, C13–C18 Aliphatics, C19–C36 Aliphatics, C11–C22 Aromatics																
		TX 1005, TX 1006	Aliphatic Fractions (nC6, >nC6-nC8, >nC8-nC10, >nC10-nC12, >nC12-nC16, >nC16-nC21, >nC21-nC35)																
			Aromatic Fractions (>C7-nC8, >nC8-nC10, >nC10-nC12, >nC12-nC16, >nC16-nC21, >nC21-nC35)																
Biodegradation Studies	Petroleum degradation [a]	QuantArray-Petro	RMO, RDEG, PHE, TOD, TOL, EDO BPH4, PM1, TBA, NAH, NIDA, PHN, ALK, ALMA, BCR, bssA, abcA, NMS, ANC, ASSA, EBAC, APS		✓	✓	✓	✓		C [d]		C [d]	C [d]	C [d]						
	DNA sequencing [a]	Next generation sequencing	Bacterial species identification		✓	✓	✓	✓		C [d]		C [d]	C [d]	C [d]					
	Carbon degradation [a]	Stable isotope probing	^{13}C-Naphthalene			✓	✓	✓		C [d]		C [d]	C [d]	C [d]					
			^{13}C-Benzene			✓	✓												
	Isotope analysis [a]	Compound-specific isotope analysis (CSIA)	Nitrogen, sulfur	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓			
	Microcosm study [a]	Lab Procedure	Microbial community		✓	✓													
NAPs	NAPs	EPA 353.2 [c]	Nitrate-Nitrite as N	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓			
		IsoFlask [a]	Dissolved oxygen, carbon dioxide, methane																
		EPA 415.1/9060 [a]	Total Organic Carbon	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓		
Field	Field parameters	Field (temperature probe)	Temperature (in-well casing measurement)		✓	✓	✓	✓						✓					
Soil Vapor [a, e]	Gases	ASTM D1946	Oxygen, carbon dioxide, methane		✓	✓	✓											✓	✓
	VOCs, SVOCs	TO-15/TO-17	VOC, SVOC		✓	✓	✓											✓	✓
	Gases [f]	Lab Procedure (Carbon Traps)	Carbon traps	Lower and upper access tunnel floor and Red Hill ridge ground surface near Tank 5															

Note: This table presents only additional analyses not presented in *SAP Revision 01*, dated April 19, 2017 (DON 2017c).

✓ to be analyzed
^{13}C carbon-13 isotope
abcA benzene carboxylase
ALK alkane monooxygenase
ALMA alkane monooxygenase
ANC naphthalene carboxylase
APS sulfate reducing bacteria
ASSA alkylsuccinate synthase
BCR benzoyl coenzyme A reductase
BPH4 biphenyl/isopropylbenzene dioxygenase
bssA benzylsuccinate synthase
C contingent analysis
DNA deoxyribonucleic acid
EBAC total eubacteria
EDO ethylbenzene/isopropylbenzene dioxygenase

EPH	extractable petroleum hydrocarbon
MADEP	Massachusetts Department of Environmental Protection
NAH	naphthalene dioxygenase
NIDA	naphthalene inducible dioxygenase
NMS	naphthylmethylsuccinate synthase
PHE	phenol hydroxylase
PHN	phenanthrene dioxygenase
PM1	methylibium petroliphilum
RDEG	toluene 2 monooxygenase/phenol hydroxylase
RMO	toluene ring hydroxylating monooxygenases
SVOC	semivolatile organic compound
TBA	tert-butyl alcohol monooxygenase
TOD	toluene/benzene dioxygenase
TOL	xylene/toluene monooxygenase
TX	Texas (Texas Commission on Environmental Quality)
VOC	volatile organic compound
VPH	volatile petroleum hydrocarbon

[a] Analysis will be performed during at least one groundwater monitoring event. Additional sampling events will be determined based on evaluation of collected data.
[b] Contingent samples and will be analyzed only if TPH-d and/or TPH-o are detected in the non-silica-gel-cleaned extract for EPA Method 8015.
[c] Analysis will be performed during all future investigation groundwater monitoring events.
[d] Contingent samples and will be collected based on evaluation of groundwater geochemistry properties to determine appropriate background groundwater conditions.
[e] Soil vapor samples will be collected at soil vapor monitoring points under the tanks, and at the air column directly above the water table at all the groundwater monitoring wells.
[f] Carbon flux traps will be installed to the subgrade at several locations along the lower access tunnel. Additional carbon traps sampling events at other locations within the tunnel and the ground surface will be determined based on evaluation of collected data.

- Sheen samples, and shake test fuel fraction (if performed), including:
  - Identifying the parent and alkylated PAHs using EPA Method 8270 SIM Mod.
  - Identifying compounds in the C3–C44 range present in jet fuel using ASTM D3328.

TPH fractions analyses will be performed on groundwater samples with TPH detections and tank bottom water (or shake test water, if tank bottom water is not available) and will include:

- Quantifying aliphatic and aromatic compounds within specific carbon ranges in the volatile and extractable portions using Massachusetts (MA) volatile petroleum hydrocarbon (VPH) and MA extractable petroleum hydrocarbon (EPH) methods, respectively, for risk assessment.
- Quantifying aliphatic and aromatic compounds within the volatile and extractable portions using Texas Commission on Environmental Quality (TCEQ) 1005 and TCEQ 1006, respectively.

The above analyses will provide a comprehensive evaluation of chemicals that may be present in the in the dissolved-phase TPH. Composition of dissolved-phase TPH in the groundwater will provide inputs for attenuation evaluation and to inform the CF&T modeling.

2.2.2 Microbial Testing

To further evaluate attenuation, there is a need to further analyze the COPCs' chemical and physical properties that affect their biodegradation rates. It is also important to understand the microbial biodegradation processes of select site-specific COPCs (i.e., TPH-d and naphthalene) for the CF&T modeling effort to assess the risks posed to potential receptors.

Dissolved-phase TPH concentrations and groundwater NAP results collected from Fourth Quarter 2016 through Second Quarter 2017 groundwater monitoring events indicate biodegradation is occurring at RHMW01, RHMW02, and RHMW03 (DON 2017b). To further evaluate natural attenuation, biological testing will provide information on the presence of petroleum-degrading microbes, profile the microbial community, and rate of degradation of a carbon-labeled COPC.

Biological tests will be performed at wells with consistent TPH detections and thus likely to have robust microbial communities capable of degrading petroleum and COPCs, and at one or more wells with no recent TPH detections for comparison. Tests will include: quantitative polymerase chain reaction (qPCR) testing (QuantArray-Petro) to determine the presence of and quantify petroleum-degrading bacteria in the dissolved-phase TPH; multiple parallel deoxyribonucleic acid (DNA) sequencing tests (Next-Generation DNA Sequencing [NGS]) to identify the microbial community make up down to the genus level; and deployed ^{13}C-naphthalene-labeled and ^{13}C-benzene-labeled passive samplers (Stable Isotope Probing [SIP]) to evaluate the degradation rate of naphthalene and benzene over time. The SIP will focus on naphthalene and benzene based on the following:

1. Naphthalene is one of the COPCs that exceed screening criteria in the groundwater at RHMW02.
2. TPH-d and TPH-o (the most prevalent COPCs detected at the monitoring wells) are combinations of multiple compounds and are impractical to analyze, and are thus precluded from isotope monitoring.

3. Benzene, while not presently identified in the groundwater, will be used for modeling future release scenarios that may include fuel with benzene components.
4. Currently available industry technologies are limited to single compound isotopes.

To further support data collected from the QuantArray-Petro, SIP, and NGS, a laboratory-conducted microcosm study will estimate the bulk attenuation rate due to biodegradation for select COPCs in both aerobic and anaerobic conditions. COPC concentration trends and the abundance of the biomarkers over time from the microcosms will be evaluated. Sediment and groundwater from RHMW01 and RHMW02 will also be collected to construct the laboratory microcosms.

Additionally, compound-specific isotope analysis (CSIA) of $^{14}N/^{15}N$-nitrogen and $^{34}S/^{32}S$-sulfur will allow for evaluation of enrichment of heavier isotopes of nitrogen and sulfur, which would be indicative of electron acceptor use during biodegradation.

Data from these analyses will provide additional evidence to support evaluation of natural attenuation and biodegradation potential at the site.

2.2.3 Additional Natural Attenuation Parameters

In addition to NAPs analyzed for during groundwater monitoring events (including dissolve oxygen, nitrate, sulfate, chloride, methane, ferrous iron, and alkalinity), total organic carbon (TOC) and nitrate-nitrite as nitrogen (N) will be analyzed for. TOC will provide a comparison of relative organic carbon concentrations throughout the monitoring well network, and nitrate-nitrite as N will provide additional support for nitrate as a NAP. TOC will be analyzed for during at least one groundwater sampling event, and nitrate-nitrite as N will be analyzed for during all NAP sampling events.

TPH-d and TPH-o with SGC, which was previously identified to be performed at minimum one dry season and one wet season event, will be performed for all groundwater samples with positive TPH-d and/or TPH-o concentrations in future investigation groundwater sampling events to evaluate the fraction of unweathered TPH and polar compounds generated as metabolites during petroleum biodegradation.

To further evaluate the MNA gases (dissolved oxygen, carbon dioxide, and methane), groundwater will also be collected in IsoFlasks during at least one event. IsoFlasks can provide both gases dissolved in the collected water, as well as gases that volatilized in the headspace of the container. IsoFlask samples will be collected during at least one sampling event.

2.2.4 Temperature Profiles

Temperature in the subsurface is especially informative in evaluating biologically generated heat in the unsaturated zone due to NSZD. Additionally, subsurface temperature profiles may provide information to evaluate preferred pathways and/or groundwater flow directions. Temperature profiles of the subsurface will be collected by lowering a string of thermistors or thermocouples approximately 10 ft long down the monitoring well locations to contact the sensor against the well casing and recording the temperature readings from the bottom of the well to the top of the monitoring well. Additionally, temperatures will be allowed to equilibrate at each depth before a final measurement is taken (approximately 10–15 minutes per depth). Temperature profiles will be collected during at least one field event at RHMW01 (or RHMW01R), RHMW02, RHMW03, and up to two potential background wells (RHMW04 and RHMW10). The temperature survey will be

conducted in wells near potential LNAPL zones and background wells where a "background-corrected temperature" curve can be generated. If no suitable background well can be used, a simple seasonal thermodynamic model (such as the Hillel model) will be used to obtain background-corrected temperatures.

2.2.5 Soil Vapor Testing

To further evaluate whether biodegradation is occurring in the vadose zone, concentrations of methane, carbon dioxide, oxygen, volatile organic compounds (VOCs), and semivolatile organic compounds (SVOCs) will be collected at all operational soil vapor monitoring point (SVMPs) and select groundwater monitoring locations. Vapor concentrations will provide a line of evidence whether off-gassing from fuel biodegradation is occurring at the SVMPs located underneath each tank, directly above the water table at select groundwater monitoring locations, or in monitoring points along the vadose zone of RHMW01R (see below).

Vapor concentrations will be collected from the SVMPs using 1-liter Summa canisters and sorbent tubes during at least one sampling event. Data from the SVMPs will be used to determine the presence of methane, carbon dioxide, and oxygen gases using method ASTM D1946, VOCs using EPA Method TO-15, and VOCs and SVOCs using EPA Method TO-17, and provide information on biodegradation of fuel occurring in the vadose zone.

Vapor concentrations will be also collected from the air column immediately above the groundwater table at select monitoring wells using sorbent tubes during at least one sampling event. These monitoring wells will include wells with TPH detections (i.e., RHMW01, RHMW02, and RHMW03) and a background well for comparison. Sorbent tubes will be analyzed for fixed gases (ASTM D1946) and VOCs and SVOCs (EPA Method TO-17).

RHMW01R, a proposed multi-level monitoring well, will be installed with vapor monitoring zones in the vadose zone. Vapor concentrations will be also collected from select vadose zone monitoring zones using sorbent tubes during at least one sampling event. Sorbent tubes will be analyzed for fixed gases (ASTM D1946) and VOCs and SVOCs (EPA Method TO-17).

2.2.6 Carbon Traps

Carbon dioxide is a by-product of microbial biodegradation of non-aqueous-phase liquid (NAPL) and methane in the subsurface. Carbon dioxide concentrations emitted by the biodegradation of NAPL can be measured by installing carbon traps along the upper and lower access tunnel floors and on the surface adjacent to the tank farm. At least one carbon dioxide sampling event will be performed; however, sampling events may be limited by Facility construction and operation activities, and may be affected by the ventilation setup and flow within the tunnel. Up to four holes will be drilled down to bedrock at the access tunnel floors near Tank 5. Additionally, up to six surface traps will be installed on the ground surface in the vicinity of Tank 5. The traps will capture the carbon dioxide emitted from the subsurface. Actual locations for the traps will be informed by the geology of the site and will be based on possible locations of preferential vapor pathways (e.g., clinker zones); locations will also be dictated by the logistics and feasibility of the trap installation given the activities and operations of the Facility. Traps will be left in place for up to 4 weeks prior to retrieval and lab analysis of the sorbent material in the traps, which will provide an estimate NAPL degradation rate for the area. Carbon traps will be installed during at least one event; additional events may be performed at other locations based on success of the Tank 5 carbon traps.

2.3 INFILTRATION RATE TESTS TO ESTIMATE GROUNDWATER RECHARGE

Evaluation of NAPL movement in the vadose zone includes assessment of groundwater recharge through the vadose zone. The recharge rate of infiltrate through the caprock contributes to the rate of NAPL dissolution and mass transport of dissolved petroleum constituents through the vadose zone. Recharge will be estimated based on dual-ring infiltrometer tests at a minimum of three locations at different elevations within the Facility boundaries, mapped soil characteristics, and precipitation records. The calculated surface recharge rate will be used to inform the CSM and the numerical groundwater model.

3. Field Methods and Procedures

Procedures cited in this section are from the Naval Facilities Engineering Command (NAVFAC) Pacific Environmental Restoration Program *Project Procedures Manual* (DON 2015). Procedures not previously presented in *SAP Revision 01* (DON 2017c) are presented in Appendix A.

Table 3-1 presents the location-specific methods and field standard operating procedures (SOPs) for the activities presented in Sections 3.1 through 3.9.

Table 3-1: Location-Specific Sampling Methods/SOP Requirements

Sampling Location/ID Number	Matrix	Depth	Analytical Group	Number of Samples	Sampling SOP Reference
RHMW01R, RHMW07D, RHMW11, RHMW12, RHMW13, RHMW14, RHMW15, RHMW16, RHMW17, RHMW18, RHMW19, RHMW20	Groundwater	approx. 80–900 ft bgs	VOC (BTEX), TPH (g/d/o), PAHs (1-methylnaphtalene, 2-methylnaphthalene, naphthalene), fuel additives (phenol, 2-[2-methoxyethoxy]-ethanol), lead scavengers (EDB, DCA), NAPs	1 primary per monitoring zone per event [a] 1 duplicate per 10 primary samples per event 1 MS/MSD pair per 20 primary samples per event 1 trip blank per location 1 equipment blank [b] per 20 primary samples per event 1 field blank [b] per event	Fluid Sampling procedures (see Appendix B
Tank 2 S/M/D, Tank 3 S/M/D, Tank 4 S/M/D, Tank 5 S/M/D, Tank 6 S/M, Tank 7 S/M/D, Tank 8 S/M/D, Tank 9 S/M/D, Tank 10 S/D, Tank 11 M/D, Tank 12 S/M/D, Tank 13 S/M/D, Tank 14 S/M/D, Tank 15 S/M/D, Tank 16 S/M/D, Tank 17 S/M/D, Tank 18 S/D, Tank 20 S/M/D	Vapor (canister)	approx. 2–5 ft below tunnel floor	Fixed gases, VOCs	1 primary per event 5 duplicates per event 5 ambient blanks per event	Procedure I-B-3, *Active Soil Gas Sampling*
Tank 2 S/M/D, Tank 3 S/M/D, Tank 4 S/M/D, Tank 5 S/M/D, Tank 6 S/M, Tank 7 S/M/D, Tank 8 S/M/D, Tank 9 S/M/D, Tank 10 S/D, Tank 11 M/D, Tank 12 S/M/D, Tank 13 S/M/D, Tank 14 S/M/D, Tank 15 S/M/D, Tank 16 S/M/D, Tank 17 S/M/D, Tank 18 S/D, Tank 20 S/M/D	Miscellaneous (sorbent tube)	approx. 2–5 ft below tunnel floor	VOCs, SVOCs	1 primary per event 5 duplicates per event 5 ambient blanks per event 1 batch blank per event	Procedure I-B-3, *Active Soil Gas Sampling*

Sampling Location/ID Number	Matrix	Depth	Analytical Group	Number of Samples	Sampling SOP Reference
RHMW01, RHMW02, RHMW03, RHMW04	Miscellaneous (sorbent tube)	approx. 82–100 ft below tunnel floor	Fixed gases, VOCs, SVOCs	1 primary per event 1 duplicate per event 1 ambient blank per event 1 batch blank per event	Procedure I-B-3, *Active Soil Gas Sampling*
RHMW01R	Miscellaneous (sorbent tube)	3 to 4 zones approx. 5–100 ft below tunnel floor	Fixed gases, VOCs, SVOCs	1 primary per event 1 duplicate per event 1 ambient blank per event 1 batch blank per event	Procedure I-B-3, *Active Soil Gas Sampling*
RHMW02, RHMW03	Miscellaneous (sheen sampler)	not applicable	C3–C44 Whole Oil	1 primary per event	Sheen sampling procedure (Section 3.7)
Facility	Water (tank bottom water) or Groundwater (shake test)	not applicable	VOCs with TICs, SVOCs with TICs, SVOCs with TICs with SGC, TPH-d/o, TPH-d/o with SGC, Parent and Alkylated PAHs, VPH, EPH, TX Aliphatics and Aromatics	1 primary per event	Procedure I-C-3, *Monitoring Well Sampling*
RHMW01, RHMW02, RHMW03, contingent locations [c]	Groundwater	approx. 82–100 ft below tunnel floor	VPH, EPH, VOCs with TICs	1 primary per event 1 duplicate per event 1 MS/MSD pair per event 1 trip blank per location [d]	Procedure I-C-3, *Monitoring Well Sampling*
			TX Aliphatics and Aromatics	1 primary per event 1 duplicate per event 1 trip blank per location [d]	Procedure I-C-3, *Monitoring Well Sampling*
			SVOC with TICs	1 primary per event 1 duplicate per event 1 MS/MSD pair per event	Procedure I-C-3, *Monitoring Well Sampling*
			Parent and Alkylated PAHs, TPHd/o with SGC, SVOC with TICs with SGC,	1 primary per event 1 duplicate per event	Procedure I-C-3, *Monitoring Well Sampling*
RHMW01, RHMW02, RHMW03, RHMW04, RHMW05, RHMW06, RHMW07, RHMW08, RHMW09, RHMW10, RHMW2254-01, OWDFMW01, HDMW2253-03	Groundwater	approx. 82–500 ft bgs	CSIA (Nitrogen, Sulfur), Nitrate-nitrite as N, TOC, Dissolved gases (IsoFlask)	1 primary for per event	Procedure I-C-3, *Monitoring Well Sampling*
RHMW01, RHMW02, RHMW03, background location	Miscellaneous (biofilter) or Groundwater	approx. 82–100 ft below tunnel floor	QuantArray-Petro, NGS	1 primary per event 1 duplicate per event	Procedure I-C-3, *Monitoring Well Sampling,* Bio-flo DNA Sampling Protocol, Groundwater DNA Sampling Protocol

Sampling Location/ID Number	Matrix	Depth	Analytical Group	Number of Samples	Sampling SOP Reference
RHMW02, RHMW03, background location	Miscellaneous (passive sampler)	approx. 82–100 ft below tunnel floor	SIP (Naphthalene)	1 primary for per event	Procedure I-C-3, *Monitoring Well Sampling*, Bio-trap SIP Protocol
RHMW02, RHMW03	Miscellaneous (passive sampler)	approx. 82–100	SIP (Benzene)	1 primary for per event	Procedure I-C-3, *Monitoring Well Sampling*, Bio-trap SIP Protocol
RHMW01, RHMW02	Groundwater/ Sediment	approx. 82–100	Microcosm Study	1 primary for per event	Procedure I-C-2, *Monitoring Well Development*
RHMW01	Groundwater	approx. 82 ft below tunnel floor	VOCs with TICs, SVOCs with TICs, TPH-d/o, Parent and Alkylated PAHs, VPH, EPH, TX Aliphatics and Aromatics	1 equipment blank per event [e] 1 field blank per event [e]	Procedure I-C-3, *Monitoring Well Sampling*
RHMW01	Miscellaneous (biofilter) or Groundwater	approx. 82 ft below tunnel floor	QuantArray-Petro, NGS	1 equipment blank per event [e, f]	Procedure I-C-3, *Monitoring Well Sampling*

Notes: Procedures are from the *Project Procedures Manual* (DON 2015).
DCA 1,2-dichloroethane
EDB 1,2-dibromoethane (ethylene dibromide)
MS matrix spike
MSD matrix spike duplicate
TPH-g TPH-gasoline range organics
[a] The actual number of monitoring zones per multi-level well will depend on geology of the saturated zone at each location. The initial sampling event of each multi-level well will involve sampling all monitoring zones. Subsequent sampling events will only include the zone straddling the water table and lower zones that show indications of contamination or demonstrate permeability requiring additional evaluation.
[b] Sampling probes will be used during sampling of multi-level wells. Rinsate from decontamination of the sampling probes will be collected as equipment blank. Field and equipment blanks will be collected during each sampling event to demonstrate field decontamination efficiency.
[c] Analysis of groundwater samples from other monitoring wells will be contingent on detections of TPH-g using EPA Method 8260 and/or TPH-d/o using EPA Method 8015.
[d] One trip blank will be collected for each sampling location.
[e] Rental pump equipment will be used during sampling of groundwater monitoring well RHMW01 for the identified analyses. All other conventional monitoring wells relevant to the indicated analytical group have dedicated pumps installed; therefore, no field or equipment blanks will be collected for those wells.
[f] No field blanks will be collected for biofilter samples. Equipment blanks will be rinsate collected during the rental pump decontamination.

3.1 MULTI-LEVEL WELL GROUNDWATER SAMPLING

Groundwater sampling for multi-level wells will be performed in accordance with Fluid Sampling procedures in Appendix B. Due to the well design, purging will not be required prior to collecting groundwater from each zone. The no-purge Fluid Sampling procedures may also be paired with the monitoring well sampling procedures in accordance with Procedure I-C-3, *Monitoring Well Sampling* (DON 2015) for the zone straddling the water table to demonstrate sampling quality.

Groundwater from a zone will be collected using sampling probes. Sealed sample containers (with up to three auxiliary containers) will be connected to the sampling probe and prepared in accordance with the Fluid Sampling procedures (Appendix B). The probe will be lowered to a monitoring zone and the containers filled with groundwater from the formation. Once sampling containers are filled,

the probe and container string will be brought to the surface, and the groundwater will be transferred to the appropriate lab-supplied containers. Excess groundwater samples will be properly disposed of as investigation-derived waste (IDW) in accordance with Procedure I-A-6, *IDW Management* (DON 2015).

All zones for each multi-level well will be sampled during the first sampling event to profile the groundwater characteristics and COPC and NAP concentrations at each zone. The top three zones (and any lower zones with COPC detections) will be sampled during the second sampling event. Only the zone straddling the water table and select lower zones will be sampled in subsequent events based on whether the lower zones exhibit contamination or based on permeability of the lower zones.

3.2 SOIL VAPOR SAMPLING

3.2.1 Soil Vapor Monitoring Points

At least one round of active soil vapor sampling will be performed at existing SVMPs under each of the 18 active tanks in the Facility. Vapor samples will be collected using batch-certified laboratory-supplied 1-liter Summa canisters and pre-conditioned single-bed sorbent cartridges (capable of testing longer-chain VOCs and SVOCs not captured in canister sampling) in accordance with Procedure I-B-3, *Active Soil Gas Sampling*. One vapor sample will be collected from each SVMP.

Each Summa canister will be fitted with a vacuum gauge and flow controller, and evacuated to create a vacuum pressure of 30 inches of mercury (inHg). The canister initial pressure and canister, controller, and gauge serial numbers will be recorded prior to deployment on field. The canisters will be set up at the tank locations, and tested for tightness against leaks using a helium shroud and a hand-held helium detector. Sorbent cartridges will be checked for any sign of compromise during transport.

SVMP tubing lines range from 25 ft (shallow or S), 60 ft (middle or M), to 93 ft (deep or D) and will be purged of ambient or stagnant air for at least three tubing-volumes using a three way valve and a pump with flow rates not exceeding 200 milliliters (mL) per minute and vacuum pressure not exceeding 7 inHg. Once the lines are purged and the sampling train is free of leaks, soil vapor will be collected into the canister until canister pressure is 5 inHg or no longer than 5 minutes. Sorbent tubes samples will be collected immediately after the canister sample is collected using the same flow rate for up to 10 minutes at flow rates no more than 200 mL per minute. Sampling start and end times and final pressure will be recorded.

3.2.2 Monitoring Well Air Column

At least one round of active soil vapor sampling will be performed directly above the water table (at the air/groundwater interface) in monitoring wells RHMW01, RHMW02, RHMW03, and a background well. Vapor samples will be collected using pre-conditioned multi-bed sorbent cartridges in accordance with Procedure I-B-3, *Active Soil Gas Sampling*. One vapor sample will be collected from each monitoring well.

Laboratory-supplied sorbent tubes capable of testing short-chain VOCs and longer-chain VOCs and SVOCs will be used for sampling. Tubing will be deployed down each monitoring well and placed approximately 6 inches from the water table. Using a pump, the tubing will be purged of ambient air as described in Section 3.2.1. The sorbent tubes will be attached to the tubing, and vapor samples will be collected for up to 10 minutes at flow rates no more than 200 mL per minute. Sampling start and end times and final flow rate will be recorded.

3.2.3 Soil Vapor Sampling at RHMW01R

Soil vapor sampling will be conducted in the vadose zone from three to four established packer-isolated zones within the Westbay sampling system. Vapor sampling will be conducted during at least one event. Laboratory-supplied sorbent tubes capable of testing short-chain VOCs and longer-chain VOCs and SVOCs will be used for sampling. Vapor samples will be collected using pre-conditioned multi-bed sorbent cartridges in accordance with Procedure I-B-3, *Active Soil Gas Sampling*. One vapor sample will be collected from each monitoring zone.

The Westbay pumping port valve will be opened at a target monitoring zone, allowing annular gas to flow into the Westbay inner casing. Tubing will be deployed down the monitoring zone. Using a pump, the tubing will be purged of ambient air as described as described in Section 3.2.1. The sorbent tubes will be attached to the tubing, and vapor samples will be collected for up to 10 minutes at flow rates no more than 200 mL per minute. Sampling start and end times and final flow rate will be recorded.

3.3 CARBON TRAPS

At least four carbon traps will be installed below the subgrade (into bedrock) of the upper and lower access tunnel floors near Tank 5. Utility clearance at the sampling locations will be performed in accordance with Procedure I-B-2, *Geophysical Testing*, and using concrete coring hand tools (e.g., electric circular concrete) or a hammer drill advance boreholes down to basalt in accordance with Procedure I-B-1, *Soil Sampling* and Appendix C *Carbon Trap Receiver Pipe Installation Guide*. Carbon traps will be installed directly above the open boreholes, then covered below the subgrade of the tunnel floor and sealed against airflow infiltrating from the tunnel. Traps will be left in place for up to 3 weeks prior to retrieval.

Additionally, up to six surface traps will be installed on the ground surface near and above Tank 5. The traps will capture the carbon dioxide from the subsurface. Traps will be left in place for up to 4 weeks prior to retrieval and lab analysis of the sorbent material in the traps, which will provide an estimated NAPL degradation rate for the area.

3.4 TEMPERATURE PROFILING

Temperature profiles of the subsurface will be collected by lowering a string of thermistors or thermocouples down the monitoring well locations to contact the sensor against the well casing and recording the temperature readings from the bottom of the well to the top of the monitoring well. The temperature will be collected at 10-ft intervals, and readings will be allowed to equilibrate at each interval before a final measurement is taken (approximately 10–15 minutes). The temperature survey will be conducted at RHMW01, RHMW02, RHMW03, and at least one background well. If no suitable background well can be used, a simple seasonal thermodynamic model (such as the Hillel model) will be used to obtain background corrected temperatures.

3.5 TANK BOTTOM WATER (OR SHAKE TEST)

Tank bottom water will be collected from at least one of the tanks for forensic and TPH fractions analysis. Tank bottom water from at least one tank containing either JP-5 or F-24 will be collected for at least three events. If tank bottom water is not available, a laboratory shake test will be performed using jet fuel from the Facility and site groundwater collected from sampling location RHMW2254-01. The laboratory will perform the shake test using a modified EPA Method OPPTS 830.7750. The laboratory will introduce up to 250 milliliters of fuel to 1 liter of groundwater in a

separatory funnel. The funnel will be shaken for up to 5 minutes, and the water and fuel fractions separated. The separated fractions will be extracted and analyzed as specified in Section 4.2.

3.6 GROUNDWATER SAMPLING

Groundwater samples for forensic analyses, TPH fractions analyses, and additional NAP analyses will be collected from monitoring wells identified in Table 2-2. Samples for forensic analyses, TPH fractions analyses, dissolved gases using IsoFlask containers, CSIA, and TOC will be collected during one sampling event at minimum. Nitrate-nitrite as nitrogen will be sampled in all future investigation groundwater sampling events. The monitoring wells will be sampled in accordance with *SAP Revision 01* (DON 2017c) and Procedure I-C-3, *Monitoring Well Sampling* (DON 2015).

3.7 SHEEN SAMPLING

No measurable LNAPL has been detected in the groundwater monitoring locations in previous sampling events, though previous groundwater concentrations at RHMW02 were at or above the TPH-d solubility limit of 4,500 µg/L (DON 2007). To evaluate if very thin LNAPL layers are present at select monitoring wells, sheen samplers capable of sorbing LNAPL will be deployed. Vendor-supplied sheen samplers connected to a decontaminated water level tape (or similar, at least 100 ft long) will be dropped down to the water table at monitoring wells RHMW02 and RHMW03. Once in the water, the sheen sampler's paper wrap will disintegrate to open the sampler. The sampler will be bobbed on the water table to open the sampler "skirt" and expose the sorbent material to the water for a minimum of 5 minutes. Using the water level tape, the sampler will be retrieved and placed in a laboratory-supplied glass jar for analysis. The sampling duration and water level will be recorded.

3.8 MICROBIAL SAMPLING

3.8.1 QuantArray-Petro and NGS

Sampling for the microbes in the groundwater uses in-line biofilters, which are small cased filters intended to be attached to the bladder pump water line. Microbes in the groundwater will be collected in the biofilter, which will be sent to the laboratory for analysis. Biofilter samples will be collected for the QuantArray-Petro analysis and NGS. In the event that biofilter samples cannot be collected due to sampling conditions, groundwater samples will be collected for laboratory processing.

The monitoring well will be purged in accordance with *SAP Revision 01* (DON 2017c) and Procedure I-C-3, *Monitoring Well Sampling* (DON 2015). The biofilter will be attached to the water outflow tubing from the bladder pump. Between 1 and 2 liters of groundwater will be passed through the biofilter, with the groundwater being collected in 1-liter bottles for measurement. Once sufficient water has been filtered, the biofilter will be disengaged from the tubing, capped, and placed in a resealable plastic bag and stored in a cooler with wet ice prior to shipment to the laboratory. The amount of the water filtered through will be recorded on the groundwater sampling forms (DON 2017c).

In the event that less than 1 liter of groundwater passes through the filter prior to the filter clogging (e.g., due to high turbidity), a second biofilter will be collected following the same procedures outlined above. The amount of water passed through each biofilter will be recorded on the groundwater sampling forms.

One biofilter sample will be collected for each monitoring well. In the event that sampling conditions prevent collection of biofilter samples (e.g., clogged filters due to high groundwater turbidity, lack of sufficient seal between the water line and filter), groundwater samples will be collected in accordance with the groundwater sampling procedures described in *SAP Revision 01* (DON 2017c) and Procedure I-C-3, *Monitoring Well Sampling* (DON 2015). For each monitoring location, 1 liter of groundwater will be collected in a 1-liter plastic unpreserved bottle, placed in a resealable plastic bag, and placed in a cooler with double-bagged wet ice for shipment to the laboratory.

3.8.2 Stable Isotope Probing

Naphthalene and benzene degradation at RHMW02, RHMW03, and a background well will be evaluated using SIP. Each monitoring well will be purged in accordance with *SAP Revision 01* (DON 2017c) and Procedure I-C-3, *Monitoring Well Sampling* (DON 2015) prior to deployment of a passive sampler. SIP analysis will be performed for one sampling event at minimum. Additional passive sampling events, likely with different COPCs (e.g., 2-methylnapthalene), will be determined after evaluation of forensic analyses data and naphthalene degradation data.

The passive sampler will contain activated carbon powder or beads tagged with ^{13}C-naphthalene or ^{13}C-benzene. As naphthalene and benzene in the environment is predominantly composed of ^{12}C species, introduction of a known concentration of ^{13}C-naphthalene or ^{13}C-benzene allows for quantification of the rate of naphthalene and benzene degradation over a set duration.

Due to limitations caused by the diameter of the monitoring well (i.e., 2 inch), the dedicated pump will be removed and stored in a secure location immediately after monitoring well purging. Once the pump is removed, the passive sampler will be deployed at the monitoring well in the water column at the same depth as the groundwater pump is set. The passive sampler will remain undisturbed in the water column between 30 and 60 days. Once the sampling duration is complete, the passive sampler will be retrieved and immediately placed in a resealable plastic bag and stored in a cooler with double-bagged wet ice for immediate shipment to the laboratory. SIP for ^{13}C-naphthalene and ^{13}C-benzene will be conducted in sequence, with the naphthalene probe deployed first then the benzene probe immediately deployed after the naphthalene probe has been retrieved. The dedicated pump will be reinstalled into the monitoring well and purged in accordance with Procedure I-C-3, *Monitoring Well Sampling* (DON 2015).

3.8.3 Microcosm Study

Sediment and groundwater from RHMW01 and RHMW02 will be collected for the microcosm study. To collect the sediment and groundwater for the microcosm study, each well will be surged using a surge block, bailer, or submersible pump in accordance with Procedure I-C-2, *Monitoring Well Development*. Groundwater from the well will be collected into laboratory-provided sample containers, immediately chilled, and shipped to the laboratory for processing.

3.9 Infiltration Rate Tests to Estimate Groundwater Recharge

Double-ring infiltrometers will be installed on the ground surface within Facility boundaries at a minimum of three locations. At least one installation point each will be located aboveground near Tanks 1 and 2, Tanks 9 and 10, and Tanks 19 and 20. The infiltration rate tests will be installed and performed in accordance with ASTM D3385, *Standard Test Method for Infiltration Rate of Soils in field Using Double-Ring Infiltrometer*.

Prior to installation, the surficial top soil will be removed to approximately 15–24 inches below ground surface (bgs). The double-ring infiltrometer consists of a 1-ft-diameter steel ring set within a 2-ft-diameter steel ring, both approximately 20 inches tall. The outer ring will be driven to a depth of approximately 6 inches bgs using a heavy sledge, jack, or equivalent. The smaller ring will be installed within the center of the outer ring, and driven to a depth of approximately 3 inches bgs. A depth gage will be installed within the inner wall of each ring. During installation, ground temperature at 12 inches bgs will be recorded prior to adding water. The annular space between the rings and the space of the inner ring will be filled with water until the water level drop rate is constant between the annular space and inner ring. Once the rate is constant between the annular space and inner ring, water will be added until both the annular space and inner ring identically contain approximately 4 inches of water. The amount of water used will be recorded, and the water level recorded down to 1/16 of an inch. Water levels will be taken every 15 minutes for the first hour, 30 minutes for the second hour, and hourly for at least the next 6 hours until the rate of the water passing through the inner ring is within 10% for three consecutive readings. A tarp will be used to secure the infiltrometer against rain and excessive evaporation during the test.

3.10 FIELD QC

Field quality control (QC) samples for groundwater including field blanks, trip blanks, ambient blanks, equipment rinsate, and duplicate samples will be collected according to the procedures described in Procedure III-B, *Field QC Samples (Water, Soil)* (DON 2015). Field QC samples are identified in Table 3-2, and the numbers of samples are presented in Table 3-3 and Table 3-4.

Field blank and equipment rinsate samples will be collected only in association with the rental sampling equipment to be used during sample collection at the wells lacking a dedicated pump (i.e., RHMW01). No field and equipment blanks will be collected for tank bottom water (or shake test), sheen samplers, fuel samples, or biotrap samples. No field blanks will be collected for biofilter samples. Only ambient blanks will be collected for vapor samples.

Table 3-2: Measurement Performance Criteria – Field QC Samples

QC Sample	Analytical Group [a]	Frequency [b]	DQI	Measurement Performance Criteria
Field duplicate	*Multi-Level Wells:* VOC (BTEX), TPH (g/d/o), PAHs (1-methylnaphtalene, 2-methylnaphthalene, naphthalene), fuel additives (phenol, 2-[2-methoxyethoxy]-ethanol), lead scavengers (EDB, DCA), NAPs *Conventional Wells:* VOCs with TICs, SVOCs with TICs, Parent and Alkylated PAHs, C3-C44 Whole Oil, VPH, EPH, TX Aliphatics and Aromatics, QuantArray-Petro, NGS, CSIA *Soil Vapor:* Fixed Gases, VOCs	10% of primary samples collected per matrix per analytical method	Precision	RPD ≤50% water [c] RPD ≤100% vapor [c]

QC Sample	Analytical Group [a]	Frequency [b]	DQI	Measurement Performance Criteria
Field blank	*Multi-Level Wells:* VOC (BTEX), TPH (g/d/o), PAHs (1-methylnaphtalene, 2-methylnaphthalene, naphthalene), fuel additives (phenol, 2-[2-methoxyethoxy]-ethanol), lead scavengers (EDB, DCA) *Conventional Wells:* VOCs with TICs, SVOCs with TICs, TPH-d/o, Parent and Alkylated PAHs, VPH, EPH, TX Aliphatics and Aromatics	Once per source of decontamination water per sampling event	Representativeness; Adequacy of the decontamination water quality or potential for contamination due to field conditions	≤1/2 of LOQ
Equipment rinsate [d]	*Multi-Level Wells:* VOC (BTEX), TPH (g/d/o), PAHs (1-methylnaphtalene, 2-methylnaphthalene, naphthalene), fuel additives (phenol, 2-[2-methoxyethoxy]-ethanol), lead scavengers (EDB, DCA) *Conventional Wells:* VOCs with TICs, SVOCs with TICs, TPH-d/o, Parent and Alkylated PAHs, VPH, EPH, TX Aliphatics and Aromatics, QuantArray-Petro, NGS	5% of primary samples collected per matrix per analytical method	Representativeness; Adequacy of the decontamination process	≤1/2 of LOQ
Trip blank	*Multi-Level Wells:* VOC (BTEX), TPH-g, lead scavengers (EDB, DCA) *Conventional Wells:* VOCs with TICs, VPH, EPH, TX Aliphatics and Aromatics	At minimum, one per cooler containing samples for groundwater	Representativeness; Contamination during sample transport	≤1/2 of LOQ
Ambient blank	Fixed Gases, VOCs, SVOCs	4 per sampling event	Representativeness; Contamination during sampling	≤1/2 of LOQ
Batch blank	Fixed Gases, VOCs, SVOCs	1 per sampling event	Representativeness; Adequacy of sorbent tube quality or contamination during sample transport	≤1/2 of LOQ

% percent
BTEX benzene, toluene, ethylbenzene, and xylenes
DQI data quality indicator
LOQ limit of quantitation
RPD relative percent difference

[a] Refer to Section 4 for the list of analytes within analytical groups.
[b] Per *Project Procedures Manual* Procedure III-B, *Field QC Samples* (DON 2015); refer to Procedure III-B Section 5 for a summary of QC samples by project location, matrix, and analytical group.
[c] Per *Project Procedures Manual* Section II, *Data Validation Procedures* (DON 2015).
[d] Applicable to reusable and/or rental sampling equipment only (i.e., multi-level system sampling probes and rental bladder pumps).

Table 3-3: Summary of Field QC Samples for Groundwater from Multi-Level Wells

Analytical Group	No. of To-Be Installed Sampling Locations	Minimum No. of Field Duplicates	Minimum No. of MS/MSD Pairs	No. of Field Blanks	Minimum No. of Equipment Blanks	No. of VOA Trip Blanks
Groundwater [a]						
VOCs (BTEX), TPH-g	12	2	1	1	1	12 [b]
TPH-d, TPH-o, PAHs (1-methylnaphthalene, 2-methylnaphthalene, naphthalene), fuel additives (phenol, 2-[2-methoxyethoxy]-ethanol)	12	2	1	1	1	—
TPH-d and TPH-o with silica gel cleanup	1 [c]	—	—	—	—	—
Lead scavengers (1,2-dibromoethane, 1,2-dichloroethane)	12 [d]	—	—	—	—	—
NAPs (dissolved oxygen, methane, ferrous iron, nitrate, sulfate, chloride, alkalinity)	12	—	—	—	—	1 [e]
Groundwater chemistry (bromide, chloride, fluoride, sulfate, total calcium, total magnesium, total manganese, total potassium, total sodium, total silica, dissolved silica) [f]	12	—	—	—	—	—

no. number
VOA volatile organic analysis
[a] Groundwater sample counts are based on a per sampling event basis.
[b] Actual number of trip blanks collected during each monitoring event will depend on the number of locations sampled during the monitoring event. It is anticipated that one trip blank will be collected at each sampling location.
[c] Actual number of samples will be contingent on detections of TPH-d and/or TPH-o in the non-silica-gel-cleaned extracts.
[d] Samples collected from each location for at least 1 year of sampling in order to fulfill the requirements of the AOC (EPA Region 9 and DOH 2015).
[e] Trip blanks for NAPs will be analyzed for methane only.
[f] Groundwater chemistry parameters will be collected during one sampling event from all monitoring zones.

Table 3-4: Summary of Field QC Samples per Source Study Event

Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSD Pairs	No. of Field Blanks [f]	No. of Equipment Rinsate [f]	No. of VOA Trip Blanks
Groundwater [a]						
Full suite VOCs with TICs	3	1	1	1	1	3 [b]
Full suite SVOCs with TICs	3	1	1	1	1	—
Full suite SVOCs with TICs with SGC	3	1	1	1	1	—
TPH-d/o with SGC	3	1	1	1	1	—
Parent and alkylated PAHs	3	1	1	1	1	—
VPH	3	1	1	1	1	3 [b]
EPH	3	1	1	1	1	—
TX aliphatics and aromatics	3	1	1	1	1	3 [b]
CSIA (nitrogen and sulfur)	13	1	—	—	—	—
Nitrate-nitrite as N and TOC	13	—	—	—	—	—
Dissolved gases (dissolved oxygen, carbon dioxide, methane)	13	—	—	—	—	—
Fuel (Sheen Sampler)						
C3–C44 whole oil	2	1	—	—	—	—
Tank Bottom Water (or Shake Test) [c]						
Full suite VOCs with TICs	1	1	—	—	—	1 [b]
Full suite SVOCs with TICs	1	1	—	—	—	—
Full suite SVOCs with TICs with SGC	1	1	—	—	—	—
TPH-d/o with SGC	1	1	—	—	—	—
Parent and alkylated PAHs	1	1	—	—	—	—
VPH	1	1	—	—	—	1 [b]
EPH	1	1	—	—	—	—
TX aliphatics and aromatics	1	1	—	—	—	1 [b]
Microbe						
SIP (13C-Naphthalene) [d]	3	—	—	— [g]	— [g]	—
SIP (13C-Benzene) [d]	2	—	—	— [g]	— [g]	—
QuantArray-Petro [e]	3	1	—	— [g]	1 [g]	—
NGS [e]	3	1	—	— [g]	1 [g]	—
Microcosm study	2	—	—	—	—	—
Vapor						
VOCs, fixed gases (SVMPs)	50	5	—	1 [i]	—	4 [h]
VOCs, fixed gases (MWs)	4	1	—	1 [i]	—	1 [i]
VOCs, fixed gases (RHMW01R)	4	1	—	1 [i]	—	1 [i]
Carbon trap	10	—	—	—	—	—

[a] Groundwater sample counts are based on a per sampling event basis.
[b] Samples from each sampling location will be accompanied by a trip blank.
[c] Tank bottom water sample will be collected from the Facility for up to three events.
[d] Samples for naphthalene and benzene degradation will be collected at RHMW02 and RHMW03, at minimum, during consecutive sampling events for at least one event per COPC. Naphthalene degradation will also be performed at one background well.
[e] Samples for QuantArray-Petro and NGS will be collected at RHMW01, RHMW02, and RHMW03 for at least one event.
[f] Field blanks and equipment rinsates will be collected for rental bladder pumps only.
[g] Only equipment rinsates from the rental bladder pump will be collected for the microbial analyses requiring biofilter samples. No field blanks will be collected. No reusable equipment will be used during the SIP sampling, thus no field blanks or equipment rinsates will be collected.
[h] Ambient blanks will be collected in lieu of trip blanks for vapor samples using canister.
[i] Sorbent batch blanks will be collected in lieu of field and trip blanks for vapor samples using sorbent cartridges.

3.11 SAMPLE CONTAINERS AND PRESERVATIVES

Groundwater samples for chemical analyses will be placed in the sample containers listed in Table 3-5 and Table 3-6 and analyzed within the required holding times. These containers, preservatives, and holding times are specified in the respective methods. The analytical laboratories selected for the project will supply the required sample containers.

Table 3-5: Sample Containers, Preservatives, and Holding Times for Multi-Level Wells

SW-846 Parameter	Number/Type of Containers per Sample	Preservative	Holding Time
VOCs			
Benzene, toluene, ethylbenzene, total xylenes	2 × 40-mL vials, Teflon-lined septum caps	No headspace, cool to ≤6°C and adjust to pH <2 with H_2SO_4, HCl, or solid $NaHSO_4$	Maximum holding time is 7 days if pH >2 or 14 days if pH <2.
TPH			
TPH-g	2 × 40-mL vials, Teflon-lined septum caps	No headspace, cool to ≤6°C and adjust to pH <2 with HCl	Maximum holding time is 7 days if pH >2 or 14 days if pH <2.
TPH-d, TPH-o (without and with silica gel cleanup)	2 × 1-L amber glass, Teflon-lined lid	Cool to ≤6°C	7 days/40 days [a]
PAHs			
1-methylnaphthalene, 2-methylnaphthalene, naphthalene	2 × 1-L amber glass, Teflon-lined lid	Cool to ≤6°C	7 days/40 days [a]
Fuel Additives			
Phenol	1 × 1-L amber glass, Teflon-lined lid	Cool to ≤6°C	7 days/40 days [a]
2-(2-methoxyethoxy)-ethanol	1 × 1-L amber glass, Teflon-lined lid	Cool to ≤6°C	7 days/40 days [a]
Lead Scavengers			
1,2-dibromoethane	2 × 40-mL vials, Teflon-lined septum caps	No headspace, cool to ≤6°C	7 days
1,2-dichloroethane	1 × 40-mL vials, Teflon-lined septum caps	No headspace, cool to ≤6°C and adjust to pH <2 with H_2SO_4, HCl, or solid $NaHSO_4$	Maximum holding time is 7 days if pH >2 or 14 days if pH <2.
NAPs			
Chloride, sulfate	1 × 250 mL plastic	Cool to ≤6°C	7 days.
Nitrate	1 × 250 mL plastic	Cool to ≤6°C	48 hours.
Ferrous iron	2 × 250 mL brown plastic	Field filtered, cool to ≤6°C, no headspace	7 days.
Methane	2 × 40-mL vials, Teflon-lined lid	No headspace, cool to ≤6°C and adjust to pH <2 with HCl	Maximum holding time is 7 days if pH >2 or 14 days if pH <2.
Alkalinity (total, bicarbonate, carbonate)	1 × 250 mL plastic	Cool to ≤6°C	14 days
Nitrate-nitrite as N	1 × 250 mL plastic	Cool to ≤6°C and adjust to pH <2 with H_2SO_4,	28 days
TOC	1 × 1-L amber glass, Teflon-lined lid	Cool to ≤6°C and adjust to pH <2 with H_2SO_4, protect from sunlight	28 days

SW-846 Parameter	Number/Type of Containers per Sample	Preservative	Holding Time
Groundwater Chemistry			
Total silica, bromide, chloride, fluoride, and sulfate	1 × 250 mL plastic	Cool to ≤6°C	28 days
Dissolved silica	1 × 250 mL plastic	Field filtered, cool to ≤6°C	28 days
Total calcium, total magnesium, total manganese, total potassium, and total sodium	1 × 500 mL plastic	Cool to ≤6°C and adjust to pH <2 with HNO_3	6 months

°C degree Celsius
H_2SO_4 sulfuric acid
HCl hydrochloric acid
HNO_3 nitric acid
L liter
mL milliliter
$NaHSO_4$ sodium hydrogen sulfate
[a] *x* days/*y* days = *x* days from sample collection to extraction/*y* days for analysis of extracts following extraction.

Table 3-6: Sample Containers, Preservatives, and Holding Times for Source Study

Parameter	Number/Type of Containers per Sample	Preservative	Holding Time
Groundwater			
VOCs with TICs	2 × 40-mL vials, Teflon-lined septum caps	No headspace, cool to ≤6°C and adjust to pH <2 with H_2SO_4, HCl, or solid $NaHSO_4$	Maximum holding time is 7 days if pH >2 or 14 days if pH <2.
SVOCs with TICs	1 × 1-L amber glass, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C	7 days/40 days [a]
TPH-d, TPH-o (without and with silica gel cleanup)	2 × 1-L amber glass, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C	7 days/40 days [a]
Parent and alkylated PAHs	2 × 1-L amber glass, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C	7 days
VPH	2 × 40-mL vials, Teflon-lined septum caps	No headspace, cool to ≤6°C and adjust to pH <2 with HCl	14 days
EPH	2 × 1-L amber glass, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C and adjust to pH <2 with HCl	7 days/40 days [a]
TX aliphatics and aromatics (TCEQ 1005)	2 × 40-mL vials, Teflon-lined septum caps	No headspace, cool to ≤6°C and adjust to pH <2 with HCl	14 days
TX aliphatics and aromatics (TCEQ 1006)	1 × 1-L amber glass, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C and adjust to pH <2 with H_2SO_4, HCl, or solid $NaHSO_4$	14 days/14 days [a]
Chloride, sulfate	1 × 250 mL plastic, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C	7 days
Nitrate	1 × 250 mL plastic, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C	48 hours
Alkalinity (total, bicarbonate, carbonate)	1 × 250 mL plastic, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C	14 days

Parameter	Number/Type of Containers per Sample	Preservative	Holding Time
Ferrous iron	1 × 250 mL brown plastic, Teflon-lined closed-top lid (no septum)	Field filtered, cool to ≤6°C, no headspace	7 days
Methane	2 × 40-mL vials, Teflon-lined septum caps	No headspace, cool to ≤6°C and adjust to pH <2 with HCl	Maximum holding time is 7 days if pH >2 or 14 days if pH <2.
Nitrate-nitrite as N	1 × 250 mL plastic, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C and adjust to pH <2 with H_2SO_4,	28 days
TOC	1 × 1-L amber glass, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C and adjust to pH <2 with H_2SO_4, protect from sunlight	28 days
Dissolved gases (oxygen, carbon dioxide, methane)	1 × IsoFlask (700 mL plastic)	None	30 days
CSIA (nitrogen, sulfur)	2 × 250 mL plastic, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C and adjust to pH <2 with HCl.	28 days
Sheen Sample			
C3–C44 whole oil	1 × sheen sampler in 8-oz glass jar, Teflon-lined closed-top lid (no septum)	None	365 days
Tank Bottom Water Sample (or Shake Test)			
VOCs with TICs	Water only: 2 × 40-mL vials, Teflon-lined septum caps Water with product layer: 1 × 1-L amber glass, Teflon-lined closed-top lid (no septum)	Water only: No headspace, cool to ≤6°C and adjust to pH <2 with H_2SO_4, HCl, or solid $NaHSO_4$ Water with product layer: Cool to ≤6°C	Maximum holding time is 7 days if pH >2 or 14 days if pH <2.
SVOCs with TICs	1 × 1-L amber glass, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C	7 days/40 days [a]
TPH-d, TPH-o (without and with silica gel cleanup)	2 × 1-L amber glass, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C	7 days/40 days [a]
Parent and alkylated PAHs	2 × 1-L amber glass, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C	7 days
VPH	2 × 40-mL vials, Teflon-lined septum caps	No headspace, cool to ≤6°C and adjust to pH <2 with HCl	14 days
EPH	2 × 1-L amber glass, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C and adjust to pH <2 with HCl	7 days/40 days [a]
TX aliphatics and aromatics (TCEQ 1005)	2 × 40-mL vials, Teflon-lined septum caps	No headspace, cool to ≤6°C and adjust to pH <2 with HCl	14 days
TX aliphatics and aromatics (TCEQ 1006)	1 × 1-L amber glass, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C and adjust to pH <2 with H_2SO_4, HCl, or solid $NaHSO_4$	14 days/14 days [a]
Microbe			
QuantArray-Petro	1 × biofilter	Cool to ≤6°C	48 hours
NGS	1 × biofilter	Cool to ≤6°C	48 hours
SIP	1 × passive sampler	Cool to ≤6°C	48 hours
Microcosm study	10 × 1-L amber glass, Teflon-lined closed-top lid (no septum)	Cool to ≤6°C	48 hours

Parameter	Number/Type of Containers per Sample	Preservative	Holding Time
Vapor (SVMPs)			
VOCs	1 × 1-L Summa canister	None	30 days
Fixed gases			
VOCs and SVOCs	1 × sorbent tube, single-bed	None	30 days
Vapor (MWs and RHMW01R)			
VOCs and SVOCs	2 × sorbent tube, multi-bed	None	30 days
Fixed gases			
Carbon Trap			
Carbon dioxide	1 × Carbon Trap	None	30 days

[a] *x* days/*y* days = *x* days from sample collection to extraction/*y* days for analysis of extracts following extraction.

3.12 SAMPLE CUSTODY REQUIREMENTS

Sample naming, labeling, handling, and shipping procedures will be performed in accordance with the sample custody requirement procedures described in *SAP Revision 01* (DON 2017c), and with the additional study type and matrix identifiers listed in Table 3-7.

Table 3-7: Sample Type and Matrix Identifiers

Identifier	Sample Type	Matrix
BF	Biofilter	Miscellaneous Solid
BT	Biotrap	Miscellaneous Solid
SH	Sheen Sampler	Miscellaneous Solid
SV	Canister	Vapor/Gas
ST	Sorbent Tube	Miscellaneous Solid
CT	Carbon Trap	Miscellaneous Solid

3.13 DECONTAMINATION

Equipment decontamination will be performed in accordance with the decontamination procedures described in *SAP Revision 01* (DON 2017c) and Procedure I-F, *Equipment Decontamination* (DON 2015).

For the decontamination procedures associated with collecting biofilter samples, the rental pump equipment rinsate will be collected in a satellite container (e.g., new, decontaminated bucket). The rinsate will be pushed through a clean biofilter using a hand pump or equivalent, and the effluent collected in 1-liter bottles for measurement. After collection of 1–2 liters of effluent, the biofilter will be sent to the laboratory in accordance with the sample custody procedures described in Section 3.8 and *SAP Revision 01* (DON 2017c). Effluent samples will be handled as IDW.

3.14 DISPOSAL OF RESIDUAL MATERIALS

IDW disposal will be performed in accordance with the IDW management procedures described in *SAP Revision 01* (DON 2017c) and Procedure I-A-6, *Investigation-Derived Waste Management* (DON 2015).

3.15 FIELD VARIANCES

As conditions in the field may vary, it may become necessary to implement modifications to the sampling procedures presented in this *SAP Addendum*. When appropriate, the Navy consultant quality assurance (QA) program manager and the Navy Contracting Officer's Representative (COR) will be notified and a verbal approval will be obtained before implementing the changes. Modifications to the approved plan will be documented in the project report.

3.16 DOCUMENTATION OF FIELD ACTIVITIES

Document and records will be kept in accordance with the documentation procedures described in *SAP Revision 01* (DON 2017c), Procedure III-D, *Logbooks* (DON 2015), and the current Navy guidance on photographs (COMNAVREG Hawaii Instruction 5510.14D).

4. Analytical Program

The sample analytical program is presented in Table 2-2, Table 3-3, and Table 3-4. In addition to information presented in *SAP Revision 01* (DON 2017c), the following reference limits and laboratory analytical methods, QC, equipment testing, and instrument calibration requirements are presented in tables in Sections 4.1 through 4.5.

4.1 REFERENCE LIMITS AND EVALUATION

No reference limits are proposed for the full suite VOCs, SVOCs, TICs, Texas (TX) aliphatics and aromatics, microbial analyses, NAPs, and fixed gases. Additional reference limits for source study apply only to VPH and EPH for groundwater samples, and VOCs and SVOCs for soil vapor samples. The reference limits presented in Table 4-1 are in addition to those presented in *SAP Revision 01* (DON 2017c).

Table 4-1: Analyte List and Reference Limits

	Screening criterion (µg/L)		Project Goal (µg/L)		Laboratory-Specific Limits (µg/L)		
Analyte	EPA RSL [a]	EAL [b]	LOQ	LOD	LOQ	LOD	DL
Groundwater							
VPH/EPH							
C5–C8 Aliphatics	1,300	N/A	433	130	100	100	50
C9–C10 Aromatics	5.5	N/A	1.8	0.55	*100*	*40*	*20*
C9–C12 Aliphatics	100	N/A	33	10	100	100	50
C9–C18 Aliphatics	100	N/A	33	10	30	30	30
C11–C22 Aromatics	5.5	N/A	1.8	0.55	*40*	*40*	*40*
C13–C18 Aliphatics	100	N/A	33	10	TBD	TBD	TBD
C19–C36 Aliphatics	60,000	N/A	20,000	6,000	50	50	50

Note: *Italic text* indicates laboratory limit exceeding the screening criterion.

	Screening criterion (µg/m^3)	Project Goal (µg/m^3)		Laboratory-Specific Limits (µg/m^3)		
Analyte	EAL [c]	LOQ	LOD	LOQ	LOD	DL
Soil Vapor						
VOCs and SVOCs						
Acenaphthene	100,114	33,371	10,011	TBD	TBD	TBD
Acenaphthylene	66,743	22,248	6,674	TBD	TBD	TBD
Acetone	12,931,429	4,310,476	1,293,143	TBD	TBD	TBD
Anthracene	500,571	166,857	50,057	TBD	TBD	TBD
Benzene	720	240	72	TBD	TBD	TBD
Benzo(a)anthracene	1,843	614	184	TBD	TBD	TBD
Biphenyl, 1,1-	167	56	17	TBD	TBD	TBD
Bis(2-chloroethyl)ether	17	5.7	1.7	TBD	TBD	TBD
Bis(2-chloro-1-methylethyl)ether	562	187	56	TBD	TBD	TBD
Bromodichloromethane	152	51	15	TBD	TBD	TBD
Bromoform	5,105	1,702	510	TBD	TBD	TBD
Bromomethane	2,086	695	209	TBD	TBD	TBD
Carbon tetrachloride	936	312	94	TBD	TBD	TBD
Chlorobenzene	20,857	6,952	2,086	TBD	TBD	TBD
Chloroethane	4,171,429	1,390,476	417,143	TBD	TBD	TBD
Chloroform	244	81	24	TBD	TBD	TBD
Chloromethane	37,543	12,514	3,754	TBD	TBD	TBD
Chlorophenol, 2-	8,343	2,781	834	TBD	TBD	TBD
Dibromochloromethane	267	89	27	TBD	TBD	TBD
Dibromoethane, 1,2-	9.4	3.1	0.94	TBD	TBD	TBD
Dichlorobenzene, 1,2-	83,429	27,810	8,343	TBD	TBD	TBD
Dichlorobenzene, 1,3-	50,057	16,686	5,006	TBD	TBD	TBD
Dichlorobenzene, 1,4-	510	170	51	TBD	TBD	TBD
Dichloroethane, 1,1-	3,510	1,170	351	TBD	TBD	TBD
Dichloroethane, 1,2-	216	72	22	TBD	TBD	TBD
Dichloroethylene, 1,1-	83,429	27,810	8,343	TBD	TBD	TBD
Dichloroethylene, cis 1,2-	3,337	1,112	334	TBD	TBD	TBD
Dichloroethylene, trans 1,2-	33,371	11,124	3,337	TBD	TBD	TBD
Dichloropropane, 1,2-	562	187	56	TBD	TBD	TBD
Dichloropropene, 1,3-	1,404	468	140	TBD	TBD	TBD

	Screening criterion (µg/m^3)	Project Goal (µg/m^3)		Laboratory-Specific Limits (µg/m^3)		
Analyte	EAL [c]	LOQ	LOD	LOQ	LOD	DL
Ethylbenzene	22,462	7,487	2,246	TBD	TBD	TBD
Fluorene	66,743	22,248	6,674	TBD	TBD	TBD
Hexachlorobenzene	12	4.1	1.2	TBD	TBD	TBD
Hexachlorobutadiene	255	85	26	TBD	TBD	TBD
Hexachloroethane	510	170	51	TBD	TBD	TBD
Methyl ethyl ketone	2,085,714	695,238	208,571	TBD	TBD	TBD
Methyl isobutyl ketone	1,251,429	417,143	125,143	TBD	TBD	TBD
Methyl tert butyl ether	21,598	7,199	2,160	TBD	TBD	TBD
Methylene chloride	202,778	67,593	20,278	TBD	TBD	TBD
Methylnaphthalene, 1-	7,745	2,582	775	TBD	TBD	TBD
Methylnaphthalene, 2-	6,674	2,225	667	TBD	TBD	TBD
Naphthalene	1,251	417	125	TBD	TBD	TBD
Nitrotoluene, 2-	102	34	10	TBD	TBD	TBD
Phenanthrene	66,743	22,248	6,674	TBD	TBD	TBD
Pyrene	50,057	16,686	5,006	TBD	TBD	TBD
Styrene	417,143	139,048	41,714	TBD	TBD	TBD
Tert-butyl alcohol	7,487	2,496	749	TBD	TBD	TBD
Tetrachloroethane, 1,1,1,2-	759	253	76	TBD	TBD	TBD
Tetrachloroethane, 1,1,2,2-	97	32	10	TBD	TBD	TBD
Tetrachloroethylene	921	307	92	TBD	TBD	TBD
Toluene	2,085,714	695,238	208,571	TBD	TBD	TBD
TPH (gasolines)	586,086	195,362	58,609	TBD	TBD	TBD
TPH (middle distillates)	262,800	87,600	26,280	TBD	TBD	TBD
Trichlorobenzene, 1,2,4-	775	258	77	TBD	TBD	TBD
Trichloroethane, 1,1,1-	2,085,714	695,238	208,571	TBD	TBD	TBD
Trichloroethane, 1,1,2-	83	28	8.3	TBD	TBD	TBD
Trichloroethylene	834	278	83	TBD	TBD	TBD
Trichloropropane, 1,2,3-	0.27	0.090	0.027	TBD	TBD	TBD
Trichloropropene, 1,2,3-	125	42	13	TBD	TBD	TBD
Vinyl chloride	340	113	34	TBD	TBD	TBD
m-Xylene	N/A	N/A	N/A	N/A	N/A	N/A
o-Xylene	N/A	N/A	N/A	N/A	N/A	N/A

	Screening criterion (µg/m^3)	Project Goal (µg/m^3)		Laboratory-Specific Limits (µg/m^3)		
Analyte	EAL [c]	LOQ	LOD	LOQ	LOD	DL
p-Xylene	N/A	N/A	N/A	N/A	N/A	N/A
Xylenes	41,714	13,905	4,171	TBD	TBD	TBD
TICs	N/A	N/A	N/A	N/A	N/A	N/A

Note: Information currently identified as TBD (to be determined) is pending based on at least one of the following: completion of analytical laboratory procurement; confirmation of laboratory schedule capability; and confirmation of laboratory limits.

DL detection limit
N/A not applicable
LOD limit of detection

[a] EPA Tapwater Residential Screening Levels THQ=1, May 2016.
[b] DOH Tier 1 EALs, Table D1-b Groundwater Action Levels, for groundwater is a current or potential drinking water resource and surface water body is not located within 150 meters of release site.
[c] DOH Tier 1 EALs, Table C-2 Shallow Soil Vapor Action Levels, for evaluation of potential vapor intrusion hazards (volatile chemicals only).

4.2 LABORATORY ANALYTICAL METHODS

Water, fuel, and vapor samples will be analyzed by a Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP)-accredited laboratory, as applicable, or a National Environmental Laboratory Accreditation Program (NELAP)-accredited laboratory using the analytical method specified for each analytical group. Samples for microbial analyses will be analyzed in accordance with The NELAC Institute standards (TNI 2016) and DoD *Quality Systems Manual* (QSM) Version 5.1 (or most current version) (DoD 2017) as applicable.

In addition to information presented in *SAP Revision 01* (DON 2017c), the additional preparation and analytical requirements for water, fuel, microbe, and vapor samples are presented in Table 4-2, Table 4-3, and Table 4-4.

Table 4-2: Preparation and Analytical Requirements for Groundwater and QC Water

Matrix	Analytical Group	Preparation Reference/Method SOP Analytical Reference/Method SOP
Water	Parent and Alkylated PAH Composition	Preparation Method: EPA 3510 Preparation SOP: TBD Analysis Method: EPA 8270 SIM Mod. Analysis SOP: TBD
	VPH	Preparation/Analysis Method: MA VPH Preparation/Analysis SOP: T-GC-WI9685
	EPH	Preparation/Analysis Method: MA EPH Preparation/Analysis SOP: T-OE-GC-WI10914/T-GC-WI9672
	TX Aliphatic and Aromatic	Preparation/Analysis Method: TCEQ 1005 Preparation/Analysis SOP: T-GC-WI9770
	TX Aliphatic and Aromatic	Preparation/Analysis Method: TCEQ 1006 Preparation/Analysis SOP: T-OE-GC-WI11364
	Nitrate-nitrite as N	Preparation/Analysis Method: EPA 353.2 Preparation/Analysis SOP: ANA353.2
	TOC	Preparation/Analysis Method: EPA 9060 Preparation/Analysis SOP: ANA9060
	Dissolved gases	Preparation/Analysis Method: TBD Preparation/Analysis SOP: TBD
	CSIA	Preparation/Analysis Method: TBD Preparation/Analysis SOP: TBD
Sheen Sample	C3–C44 Whole Oil	Preparation Method: EPA 3580 Preparation SOP: TBD Analysis Method: ASTM D3328 Analysis SOP: TBD

Matrix	Analytical Group	Preparation Reference/Method SOP Analytical Reference/Method SOP
Microbe	QuantArray-Petro	Preparation/Analysis Method: Lab Procedure Preparation/Analysis SOP: MI SOP-DNAEXT/QuantArray
	NGS	Preparation/Analysis Method: Lab Procedure Preparation/Analysis SOP: MI SOP- Amplicon PCR
Microbe (cont.)	SIP	Preparation/Analysis Method: Lab Procedure Preparation/Analysis SOP: MI SOP-PLFA/MI SOP- SIP Data
Vapor	VOCs, Fixed Gases (canister)	Preparation/Analysis Method: TO-15, ASTM D1946 Preparation/Analysis SOP: TBD
	VOCs, SVOCs, Fixed Gases (sorbent tube)	Preparation/Analysis Method: TO-17, ASTM D1946 Preparation/Analysis SOP: TBD
	Carbon Trap	Preparation/Analysis Method: Lab Procedure Preparation/Analysis SOP: TBD

Note: Information currently identified as TBD (to be determined) is pending based on at least one of the following: completion of analytical laboratory procurement; confirmation of laboratory schedule capability; and confirmation of laboratory method performance.

Table 4-3: Analytical Services

Matrix	Analytical Group	Sampling Locations/ ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (address)
Groundwater	Multi-Level Wells: VOC (BTEX), TPH (g/d/o), PAHs (1-methylnaphtalene, 2-methylnaphthalene, naphthalene), fuel additives (phenol, 2-[2-methoxyethoxy]-ethanol), lead scavengers (EDB, DCA), NAPs, groundwater chemistry (bromide, chloride, fluoride, sulfate, total calcium, total magnesium, total manganese, total potassium, total sodium, total silica and dissolved silica) Conventional Wells: VOCs with TICs, SVOCs with TICs, Nitrate-Nitrite as N, TOC	Multi-level Wells: RHMW01R, RHMW07D, RHMW11, RHMW12, RHMW13, RHMW14, RHMW15, RHMW16, RHMW17, RHMW18, RHMW19, RHMW20 Conventional Wells: RHMW01, RHMW02, RHMW03, contingent wells	ANA8260, ANA8011, ANA8015, ANA8270SIM, ANA RSK175, ANA3500FeBc, HPL9056, ANA2320B, ANA8270, ANA6010, ANA4500SiD, ANA353.2, ANA9060	14 days after samples are received at the laboratory	APPL (908 N. Temperance Ave., Clovis, CA 93611)
	Parent and Alkylated PAHs, C3–C44 Whole Oil	RHMW01, RHMW02, RHMW03, contingent wells	TBD	14 days after samples are received at the laboratory	PACE (220 William Pitt Way, Pittsburgh, PA 15238)
	VPH, EPH, TX Aliphatics and Aromatics	RHMW01, RHMW02, RHMW03, contingent wells	T-GC-WI9685, T-GC-WI9672, T-GC-WI9770, T-GC-WI9792	14 days after samples are received at the laboratory	EarthToxics/Eurofins|Lancaster (2425 New Holland Pike, Lancaster, PA 17601)
	CSIA (nitrogen, sulfur)	RHMW01, RHMW02, RHMW03, RHMW04, RHMW05, RHMW06, RHMW07, RHMW08, RHMW09, RHMW10, RHMW2254-01, OWDFMW01, HDMW2253-03	TBD	14 days after samples are received at the laboratory	Isotech (1308 Parkland Court, Champaign, IL 61821)
	Dissolved gases (oxygen, carbon dioxide, methane) (IsoFlask)	RHMW01, RHMW02, RHMW03, RHMW04, RHMW05, RHMW06, RHMW07, RHMW08, RHMW09, RHMW10, RHMW2254-01, OWDFMW01, HDMW2253-03	TBD	14 days after samples are received at the laboratory	Isotech (1308 Parkland Court, Champaign, IL 61821)

Matrix	Analytical Group	Sampling Locations/ ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/Organization (address)
Tank bottom water (or shake test)	VOCs with TICs, SVOCs with TICs, TPH-d/o	Facility	ANA8260, ANA8270, ANA8015	14 days after samples are received at the laboratory	APPL (908 N. Temperance Ave., Clovis, CA 93611)
	Parent and Alkylated PAHs, C3–C44 Whole Oil	Facility	TBD	14 days after samples are received at the laboratory	PACE (220 William Pitt Way, Pittsburgh, PA 15238)
	VPH, EPH, TX Aliphatics and Aromatics	Facility	T-GC-WI9685, T-GC-WI9672, T-GC-WI9770, T-GC-WI9792	14 days after samples are received at the laboratory	EarthToxics/Eurofins|Lancaster (2425 New Holland Pike, Lancaster, PA 17601)
Sheen sampler	C3–C44 Whole Oil	RHMW02, RHMW03	TBD	14 days after samples are received at the laboratory	PACE (220 William Pitt Way, Pittsburgh, PA 15238)
Biofilter	QuantArray-Petro, NGS	RHMW01, RHMW02, RHMW03, background well	QuantArray, MI SOP- Amplicon PCR	21 days after samples are received at laboratory	Microbial Insights (10515 Research Dr., Knoxville, TN 37932)
Biotrap	SIP (13C-Naphthalene)	RHMW02, RHMW03, RHMW04	MI SOP- SIP Data	21 days after samples are received at laboratory	Microbial Insights (10515 Research Dr., Knoxville, TN 37932)
Biotrap	SIP (13C-Benzene)	RHMW02, RHMW03	MI SOP- SIP Data	21 days after samples are received at laboratory	Microbial Insights (10515 Research Dr., Knoxville, TN 37932)
Vapor/sorbent tube	VOC, SVOC, Fixed Gases	SVMPs RHMW01, RHMW02, RHMW03, background well RHMW01R	TBD	21 days after samples are received at laboratory	Eurofins|AirToxics (180 Blue Ravine Rd., Folsom CA 95630)
Carbon trap	Carbon Dioxide	Lower and upper access tunnels, ridge ground surface	TBD	21 days after samples are received at laboratory	E-Flux (3185-A Rampant Rd. #250D, Fort Collins, CO 80521)

Note: Information currently identified as TBD (to be determined) is pending based on at least one of the following: completion of analytical laboratory procurement; confirmation of laboratory schedule capability; and confirmation of laboratory method performance.

Table 4-4: Analytical SOP References

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM (Yes/No)	Modified for Project Work? (Yes/No)
Preparatory Methods						
T-OE-GC-WI10914	Separatory Funnel Extraction for EPH in Waters by Massachusetts, New Jersey, and Louisiana Protocol, Rev. 6, 6/15/16	Definitive	MA EPH (Water)	Preparation	No	No
T-OE-GC-WI11364	Extraction of Total Petroleum Hydrocarbon Organics in Waters by Texas Methodology. Rev. 5, 7/21/17	Definitive	TX Aliphatic and Aromatic (TCEQ 1006) (Water)	Preparation	No	No
TBD	TBD	Definitive	Parent and Alkylated PAH (Water)	Preparation	TBD	TBD
TBD	TBD	Definitive	C3–C44 Whole Oil (Sheen Sampler)	Preparation	TBD	TBD
MI SOP-DNAEXT	Extraction of DNA from Environmental samples (Matrix-Soil, Water, BioFlo, Beads), 04/12/2016, Rev 1.2	Definitive	QuantArray-Petro (Biofilter)	Preparation	No	No
MI SOP-Amplicon PCR	16s Amplicon PCR from Environmental Samples for Next Generation Sequencing, 1/1/2017, 1.0	Definitive	NGS (Biofilter)	Preparation	No	No
MI SOP-PLFA	Modified Bligh and Dyer Lipid Extraction, 7/6/2016, Rev 2.1	Definitive	SIP (Biotrap)	Preparation	No	No
Analytical Methods						
T-GC-WI9685	MA DEP VPH in Waters and Solids Using GC, Rev 13, 7/27/16	Definitive	MA VPH (Water, Product)	GC/PID/FID	No	No
T-GC-WI9672	EPH by Massachusetts Protocol (MAEPH) in Waters and Solids Using GC, Rev. 11, 6/6/17	Definitive	MA EPH (Water)	GC/FID	No	No
T-GC-WI9770	TNRCC TX Method 1005 - Total Petroleum Hydrocarbons (Gasoline Range, Diesel Range, and Extended Range Organics) in Waters and Solids, Rev. 11, 5/10/17	Definitive	TX1005 (Water)	GC/FID	No	No
T-GC-WI9792	TX 1006 Characterization of C6-C35 Petroleum Hydrocarbons in Waters and Solids Rev. 6, 8/4/16	Definitive	TX1006 (Water)	GC/FID	No	No
TBD	TBD	Definitive	Parent and Alkylated PAH Composition (Water)	GC/MS	TBD	TBD
TBD	TBD	Definitive	C3–C44 Whole Oil (Sheen Sampler)	GC/MS	TBD	TBD
TBD	TBD	Definitive	CSIA (nitrogen, sulfur) (Water)	EA/IRMS	TBD	TBD

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Variance to QSM (Yes/No)	Modified for Project Work? (Yes/No)
ANA353.2	Total Oxidizable Nitrogen, Nitrate, and Nitrite Analysis by EPA 353.2, Rev 8, 1/25/17	Definitive	Nitrate-Nitrite as N, calculation (Water)	Spectrophotometer	TBD	TBD
ANA9060	TBD	Definitive	TOC (Water)	Carbonaceous Analyzer	TBD	TBD
QuantArray	QuantArray, 04/01/2013, Rev 1.1	Definitive	QuantArray-Petro (Biofilter)	RealTime PCR	No	No
MI SOP- Amplicon PCR	16s Amplicon PCR from Environmental Samples for Next Generation Sequencing, 1/1/2017, 1.0	Definitive	NGS (Biofilter)	MiSeq System	No	No
MI SOP- SIP Data	SIP Data, 5/19/2017, Rev 1.1	Definitive	SIP (Biotrap)	GC/IRMS	No	No
TBD	TBD	Definitive	VOCs (Vapor)	GC/MS	TBD	TBD
TBD	TBD	Definitive	VOCs, SVOCs (Sorbent)	GC/MS	TBD	TBD
TBD	TBD	Definitive	Fixed Gases (Vapor)	GC	TBD	TBD
TBD	TBD	Definitive	Fixed Gases (Sorbent)	GC/TCD	TBD	TBD
TBD	TBD	Definitive	Carbon Dioxide (Carbon trap)	TBD	TBD	TBD

Note: Information currently identified as TBD (to be determined) is pending based on at least one of the following: completion of analytical laboratory procurement; confirmation of laboratory schedule capability; and confirmation of laboratory method performance.

EA/IRMS elemental analyzer/isotope ratio mass spectrometry
GC/IRMS gas chromatography/isotope ratio mass spectrometry
GC/MS gas chromatography/mass spectrometry
GC/TCD gas chromatography/thermal conductivity detector
PID photoionization detector

4.3 LABORATORY QUALITY CONTROL

Corrective actions will be implemented when control limits for field or laboratory QC measurements are not met, as described in Section 5.

In addition to laboratory QC samples identified in *SAP Revision 01* (DON 2017c), laboratory QC samples for water, fuel, and vapor samples identified in this *SAP Addendum* will include method blanks, laboratory control samples, matrix spikes/matrix spike duplicates (MS/MSDs, as applicable), and duplicates (as applicable) and as described in the DoD QSM Version 5.1 (DoD 2017) and Table 4-5 Laboratory QC Samples.

Table 4-5: Laboratory QC Samples

Matrix	**Groundwater**
Analytical Group	**VPH**
Analytical Method/SOP Reference	Analytical/Preparation Method: MA VPH Laboratory SOPs: T-GC-WI9685
Analytical Organization	EarthToxics/Eurofins|Lancaster

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
LOD determination and verification	At initial set-up and verified quarterly. If a laboratory uses multiple instruments for a given method, the LOD must be verified on each.	The apparent signal to noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must: 1) Repeat the detection limit determination and LOD verification at a higher concentration; or 2) Perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	Analyst Lab QA Officer Project Chemist	Bias/ Representativeness	QC acceptance criteria as specified by Lab SOP T-GC-WI9685.
LOQ establishment and verification	At initial setup: 1) Verify LOQ; and 2) Determine precision and bias at the LOQ. Subsequently, verify LOQ quarterly. If a laboratory uses multiple instruments for a given method, the LOQ must be verified on each.	1) The LOQ and associated precision and bias must meet client requirements and must be reported; or 2) In the absence of client requirements, must meet control limits of the LCS. 3) If the method is modified, precision and bias at the new LOQ must be demonstrated and reported. See Volume 1, Module 4, Section 1.5.2 of the DoD QSM 5.1 (DoD 2017).	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	QC acceptance criteria as specified by Lab SOP T-GC-WI9685and at least as stringent as specified by DoD QSM 5.1 (DoD 2017).

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
CCV	Before sample analysis, after every 10 field samples, after every 12 hours of analysis time, and at the end of the analysis sequence.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ±20% of true value. All reported analytes and surrogates within ±50% for the end of the analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	Results may not be reported without a valid CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. If the specific version of a method requires additional evaluation (e.g., average response factors) these additional requirements must also be met.
MB	Each time analytical batch.	No analytes detected >1/2 LOQ or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common lab contaminants, no analytes detected >LOQ.	Correct problem. If required, re-prep and reanalyze MB and all samples processed with the contaminated blank.	Analyst Lab QA Officer Project Chemist	Bias	No analytes detected >1/2 LOQ or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common laboratory contaminants, no analytes detected >LOQ.
LCS	One per batch of at most 20 samples analyzed of similar matrix per analytical method.	Per Method MA VPH and Lab SOP T-GC-WI9685.	Correct problem. If required, re-prep and reanalyze the LCS and all samples processed in the associated preparatory batch for the failed analytes. Results may not be reported without a valid LCS.	Analyst Lab QA Officer Project Chemist	Accuracy	QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017) for GC-FID.
MS/MSD pair	One per analytical method for each batch of at most 20 samples.	Per Method MA VPH and Lab SOP T-GC-WI9685. MSD or Matrix Duplicate: RPD of all analytes ≤50%.	Examine the PQOs. Notify Lab QA officer and project chemist about additional measures to be taken.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	For matrix evaluation, use QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017) for GC-FID.
Surrogate spike	All field and QC samples.	Per Method MA VPH and Lab SOP T-GC-WI9685.	For QC and field samples, correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision/ Representativeness	QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017) for GC-FID.
Trip blank	1 per cooler.	Target analytes ≤1/2 LOQ.	Reanalyze for confirmation through a second analysis of the trip blank. Examine the PQOs.	Analyst Lab QA Officer Project Chemist	Accuracy/Bias, Representativeness/ Contamination	Target analytes ≤1/2 LOQ.

CCV continued calibration verification
GC-FID gas chromatography-flame ionization detector
LCS laboratory control sample
MB method blank
PQO project quality objective
RT retention time

Matrix	**Groundwater**
Analytical Group	**MA EPH**
Analytical Method/SOP Reference	Analytical/Preparation Method: MA EPH Laboratory SOPs: T-GC-WI9672
Analytical Organization	EarthToxics/Eurofins|Lancaster

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
LOD determination and verification	At initial set-up and verified quarterly. If a laboratory uses multiple instruments for a given method, the LOD must be verified on each.	The apparent signal to noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must: 1) Repeat the detection limit determination and LOD verification at a higher concentration; or 2) Perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	Analyst Lab QA Officer Project Chemist	Bias/ Representativeness	QC acceptance criteria as specified by Lab SOP T-GC-WI9672.
LOQ establishment and verification	At initial setup: 1) Verify LOQ; and 2) Determine precision and bias at the LOQ. Subsequently, verify LOQ quarterly. If a laboratory uses multiple instruments for a given method, the LOQ must be verified on each.	1) The LOQ and associated precision and bias must meet client requirements and must be reported; or 2) In the absence of client requirements, must meet control limits of the LCS. 3) If the method is modified, precision and bias at the new LOQ must be demonstrated and reported. See Volume 1, Module 4, Section 1.5.2 of the DoD QSM 5.1 (DoD 2017).	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	QC acceptance criteria as specified by Lab SOP T-GC-WI9672and at least as stringent as specified by DoD QSM 5.1 (DoD 2017).
CCV	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ±25% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	Results may not be reported without a valid CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.
MB	Each time samples are extracted and one per matrix per analytical method for each batch of at most 20 samples.	No analytes detected >1/2 LOQ or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common lab contaminants, no analytes detected >LOQ.	Correct problem. If required, re-prep and reanalyze MB and all samples processed with the contaminated blank.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	No analytes detected >1/2 LOQ or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common laboratory contaminants, no analytes detected >LOQ.

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
LCS	One per batch of at most 20 samples analyzed of similar matrix per analytical method.	Per Method MA EPH and Lab SOP T-GC-WI9672.	Correct problem. If required, re-prep and reanalyze the LCS and all samples processed in the associated preparatory batch for the failed analytes.	Analyst Lab QA Officer Project Chemist	Accuracy	QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017) for GC-FID.
Surrogate spike	All field and QC samples.	Per Method MA EPH and Lab SOP T-GC-WI9672.	For QC and field samples, correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision/ Representativeness	QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017) for GC-FID.
Fractionation surrogate	All field and QC samples.	Per Method MA EPH and Lab SOP T-GC-WI9672.	For QC and field samples, correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision/ Representativeness	QC acceptance criteria per Method MA EPH.
Fractionation check solution	All field and QC samples.	Per Method MA EPH and Lab SOP T-GC-WI9672.	For QC and field samples, correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	QC acceptance criteria per Method MA EPH.
MS/MSD pair	One per analytical method for each batch of at most 20 samples.	Per Method MA EPH and Lab SOP T-GC-WI9672. MSD or Matrix Duplicate: RPD of all analytes ≤50%.	Examine the PQOs. Notify Lab QA officer and project chemist about additional measures to be taken.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	For matrix evaluation, use QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017) for GC-FID.

Matrix	**Groundwater**
Analytical Group	**TX Aliphatics and Aromatics**
Analytical Method/SOP Reference	Analytical/Preparation Method: TCEQ 1005 Laboratory SOPs: T-GC-WI9770
Analytical Organization	EarthToxics/Eurofins|Lancaster

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
LOD determination and verification	At initial set-up and verified quarterly. If a laboratory uses multiple instruments for a given method, the LOD must be verified on each.	The apparent signal to noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must: 1) Repeat the detection limit determination and LOD verification at a higher concentration; or 2) Perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	Analyst Lab QA Officer Project Chemist	Bias/ Representativeness	QC acceptance criteria as specified by Lab SOP T-GC-WI9770.
LOQ establishment and verification	At initial setup: 1) Verify LOQ; and 2) Determine precision and bias at the LOQ. Subsequently, verify LOQ quarterly. If a laboratory uses multiple instruments for a given method, the LOQ must be verified on each.	1) The LOQ and associated precision and bias must meet client requirements and must be reported; or 2) In the absence of client requirements, must meet control limits of the LCS. 3) If the method is modified, precision and bias at the new LOQ must be demonstrated and reported. See Volume 1, Module 4, Section 1.5.2 of the DoD QSM 5.1 (DoD 2017).	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	QC acceptance criteria as specified by Lab SOP T-GC-WI9770and at least as stringent as specified by DoD QSM 5.1 (DoD 2017).
CCV	Before sample analysis, after every 10 field samples, after every 12 hours of analysis time, and at the end of the analysis sequence.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ±20% of true value. All reported analytes and surrogates within ±50% for the end of the analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	Results may not be reported without a valid CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative. If the specific version of a method requires additional evaluation (e.g., average response factors) these additional requirements must also be met.

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
Retention time check	One per batch of at most 20 samples	RF for nC35 is ≥75% the RF for nC28.	Correct problem and reestablish RT windows. Reanalyze all samples since last acceptable retention time check.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	Results may not be reported without a valid retention time check. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.
MB	Each time analytical batch.	No analytes detected >1/2 LOQ or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common lab contaminants, no analytes detected >LOQ.	Correct problem. If required, re-prep and reanalyze MB and all samples processed with the contaminated blank.	Analyst Lab QA Officer Project Chemist	Bias	No analytes detected >1/2 LOQ or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common laboratory contaminants, no analytes detected >LOQ.
LCS	One per batch of at most 20 samples analyzed of similar matrix per analytical method.	Per Method TCEQ 1005 and Lab SOP T-GC-WI9770.	Correct problem. If required, re-prep and reanalyze the LCS and all samples processed in the associated preparatory batch for the failed analytes. Results may not be reported without a valid LCS.	Analyst Lab QA Officer Project Chemist	Accuracy	QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017) for GC-FID.
MS/MSD pair	One per analytical method for each batch of at most 20 samples.	Per Method TCEQ 1005 and Lab SOP T-GC-WI9770. MSD or Matrix Duplicate: RPD of all analytes ≤50%.	Examine the PQOs. Notify Lab QA officer and project chemist about additional measures to be taken.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	For matrix evaluation, use QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017) for GC-FID.
Surrogate spike	All field and QC samples.	Per Method TCEQ 1005 and Lab SOP T-GC-WI9770.	For QC and field samples, correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision/ Representativeness	QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017) for GC-FID.
Trip blank	1 per cooler.	Target analytes ≤1/2 LOQ.	Reanalyze for confirmation through a second analysis of the trip blank. Examine the PQOs.	Analyst Lab QA Officer Project Chemist	Accuracy/Bias, Representativeness/ Contamination	Target analytes ≤1/2 LOQ.

1 RF response factor

Matrix	**Groundwater**
Analytical Group	**TX Aliphatics and Aromatics**
Analytical Method/SOP Reference	Analytical/Preparation Method: TCEQ 1006 Laboratory SOPs: T-GC-WI9792
Analytical Organization	EarthToxics/Eurofins|Lancaster

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
LOD determination and verification	At initial set-up and verified quarterly. If a laboratory uses multiple instruments for a given method, the LOD must be verified on each.	The apparent signal to noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must: 1) Repeat the detection limit determination and LOD verification at a higher concentration; or 2) Perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	Analyst Lab QA Officer Project Chemist	Bias/ Representativeness	QC acceptance criteria as specified by Lab SOP T-GC-WI9792.
LOQ establishment and verification	At initial setup: 1) Verify LOQ; and 2) Determine precision and bias at the LOQ. Subsequently, verify LOQ quarterly. If a laboratory uses multiple instruments for a given method, the LOQ must be verified on each.	1) The LOQ and associated precision and bias must meet client requirements and must be reported; or 2) In the absence of client requirements, must meet control limits of the LCS. 3) If the method is modified, precision and bias at the new LOQ must be demonstrated and reported. See Volume 1, Module 4, Section 1.5.2 of the DoD QSM 5.1 (DoD 2017).	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	QC acceptance criteria as specified by Lab SOP T-GC-WI9792 and at least as stringent as specified by DoD QSM 5.1 (DoD 2017).
CCV	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ±25% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	Results may not be reported without a valid CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.
MB	Each time samples are extracted and one per matrix per analytical method for each batch of at most 20 samples.	No analytes detected >1/2 LOQ or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common lab contaminants, no analytes detected >LOQ.	Correct problem. If required, re-prep and reanalyze MB and all samples processed with the contaminated blank.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	No analytes detected >1/2 LOQ or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common laboratory contaminants, no analytes detected >LOQ.

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
LCS	One per batch of at most 20 samples analyzed of similar matrix per analytical method.	Per Method TCEQ 1006 and Lab SOP T-GC-WI9792.	Correct problem. If required, re-prep and reanalyze the LCS and all samples processed in the associated preparatory batch for the failed analytes.	Analyst Lab QA Officer Project Chemist	Accuracy	QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017) for GC-FID.
Surrogate spike	All field and QC samples.	Per Method TCEQ 1006 and Lab SOP T-GC-WI9792.	For QC and field samples, correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision/ Representativeness	QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017) for GC-FID.
Fractionation surrogate	All field and QC samples.	Per Method TCEQ 1006 and Lab SOP T-GC-WI9792.	For QC and field samples, correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision/ Representativeness	QC acceptance criteria per Method TCEQ 1006.
Fractionation check solution	All field and QC samples.	Per Method TCEQ 1006 and Lab SOP T-GC-WI9792.	For QC and field samples, correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	QC acceptance criteria per Method TCEQ 1006.
MS/MSD pair	One per analytical method for each batch of at most 20 samples.	Per Method TCEQ 1006 and Lab SOP T-GC-WI9792. MSD or Matrix Duplicate: RPD of all analytes ≤50%.	Examine the PQOs. Notify Lab QA officer and project chemist about additional measures to be taken.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	For matrix evaluation, use QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017) for GC-FID.

Matrix	**Groundwater**
Analytical Group	**Parent and Alkylated PAHs**
Analytical Method/SOP Reference	Analytical Method: EPA Method 8270 SIM Mod. Preparation Method: EPA 3510 Laboratory SOPs: TBD
Analytical Organization	PACE

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
RL establishment and verification	At initial setup: 1) Verify RL; and 2) Determine precision and bias at the RL. Subsequently, verify RL quarterly. If a laboratory uses multiple instruments for a given method, the RL must be verified on each.	1) The RL and associated precision and bias must meet client requirements and must be reported; or 2) In the absence of client requirements, must meet control limits of the LCS. 3) If the method is modified, precision and bias at the new RL must be demonstrated and reported.	If the RL verification fails, the laboratory must either establish a higher RL or modify method to meet the client-required precision and bias.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	QC acceptance criteria as specified by Lab SOP TBD, and at least as stringent as specified by Method EPA SW-846 8270 SIM.
Performance check	Before ICAL and sample analysis, and at the beginning of each 12-hour shift.	Degradation of DDT must be ≤20%. Benzidine and pentachlorophenol will be present at their normal responses, and will not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	Degradation of DDT must be ≤20%; and benzidine and pentachlorophenol must be present at normal responses and tailing factor is ≤2. No samples must be analyzed until performance check is within criteria.
Tune check	Prior to the ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	No samples may be analyzed without a passing tune.
CCV	Before sample analysis, after every 10 field samples, after every 12 hours of analysis time, and at the end of the analysis sequence.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ±20% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	Results may not be reported without a valid CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.
MB	Each time samples are extracted and one per matrix per analytical method for each batch of at most 20 samples.	No analytes detected >1/2 RL or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common lab contaminants, no analytes detected >RL.	Correct problem. If required, re-prep and reanalyze MB and all samples processed with the contaminated blank.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	No analytes detected >1/2 RL or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common laboratory contaminants, no analytes detected >RL.

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
LCS	One per batch of at most 20 samples analyzed of similar matrix per analytical method.	Per Method EPA SW-846 8270 SIM and Lab SOP TBD.	Correct problem. If required, re-prep and reanalyze the LCS and all samples processed in the associated preparatory batch for the failed analytes.	Analyst Lab QA Officer Project Chemist	Accuracy	QC acceptance criteria at least as stringent as specified by Method EPA SW-846 8270 SIM.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ±10 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision/ Representativeness	Laboratory in-house method manual to be followed for acceptance criteria.
Surrogate spike	All field and QC samples.	Per Method EPA SW-846 8270 SIM and Lab SOP TBD.	For QC and field samples, correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision/ Representativeness	QC acceptance criteria at least as stringent as specified by Method EPA SW-846 8270 SIM.
MS/MSD pair	One per analytical method for each batch of at most 20 samples.	Per Method EPA SW-846 8270 SIM and Lab SOP TBD.	Examine the PQOs. Notify Lab QA Officer and project chemist about additional measures to be taken.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	For matrix evaluation, use QC acceptance criteria at least as stringent as specified by Method EPA SW-846 8270 SIM.

BFB 4-bromofluorobenzene
DDT dichlorodiphenyltrichloroethane
DFTPP decafluorotriphenylphosphine
ICAL initial calibration
RL reporting limit

Matrix	**Sheen Sample**
Analytical Group	**C3–C44 Whole Oil**
Analytical Method/SOP Reference	Analytical/Preparation Method: ASTM D3328 Laboratory SOP: TBD
Analytical Organization	TBD

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
Reporting limit establishment and verification	At initial setup: 1) Verify RL; and 2) Determine precision and bias at the RL. Subsequently, verify RL quarterly. If a laboratory uses multiple instruments for a given method, the RL must be verified on each.	1) The RL and associated precision and bias must meet client requirements and must be reported; or 2) In the absence of client requirements, must meet control limits of the LCS. 3) If the method is modified, precision and bias at the new RL must be demonstrated and reported.	If the RL verification fails, the laboratory must either establish a higher RL or modify method to meet the client-required precision and bias.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	QC acceptance criteria as specified by Lab SOP TBD, and at least as stringent as specified by ASTM D3328.
Resolution check	Before sample analysis.	Pristane and phytane resolution ≥80% for peak pairs n-C17 and pristane, and peak pairs n-C18 and phytane.	Correct problem, then repeat performance checks. If resolution is <50% either or both peak pairs, replace column and check instrument operating conditions.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	Pristane and phytane resolution ≥80% for peak pairs n-C17 and pristane, and peak pairs n-C18 and phytane. No samples must be analyzed until resolution check is within criteria.
MB	Each time samples are extracted and one per matrix per analytical method for each batch of at most 20 samples.	No analytes detected >1/2 RL or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common lab contaminants, no analytes detected >RL.	Correct problem. If required, re-prep and reanalyze MB and all samples processed with the contaminated blank.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	No analytes detected >1/2 RL or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is higher. For common laboratory contaminants, no analytes detected >RL.
LCS/LCSD	One per batch of at most 20 samples analyzed of similar matrix per analytical method.	Per ASTM D3328 and Lab SOP TBD.	Correct problem. If required, re-prep and reanalyze the LCS/LCSD and all samples processed in the associated preparatory batch for the failed analytes.	Analyst Lab QA Officer Project Chemist	Accuracy	QC acceptance criteria at least as stringent as specified by Lab SOP TBD.

1 LCSD laboratory control sample duplicate

Matrix	**Soil Vapor**
Analytical Group	**VOCs**
Analytical Method/SOP Reference	Analytical/Preparation Method: EPA TO-15 Laboratory SOPs: TBD
Analytical Organization	Eurofins|AirToxics

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
LOD determination and verification	At initial set-up and verified quarterly. If a laboratory uses multiple instruments for a given method, the LOD must be verified on each.	The apparent signal to noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must: 1) Repeat the detection limit determination and LOD verification at a higher concentration; or 2) Perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	Analyst Lab QA Officer Project Chemist	Bias/ Representativeness	QC acceptance criteria as specified by Lab SOP TBD.
LOQ establishment and verification	At initial setup: 1) Verify LOQ; and 2) Determine precision and bias at the LOQ. Subsequently, verify LOQ quarterly. If a laboratory uses multiple instruments for a given method, the LOQ must be verified on each.	1) The LOQ and associated precision and bias must meet client requirements and must be reported; or 2) In the absence of client requirements, must meet control limits of the LCS. 3) If the method is modified, precision and bias at the new LOQ must be demonstrated and reported. See Volume 1, Module 4, Section 1.5.2 of the DoD QSM 5.1 (DoD 2017).	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	QC acceptance criteria as specified by Lab SOP TBD and at least as stringent as specified by DoD QSM 5.1 (DoD 2017).
Canister cleanliness check	Each canister batch prior to shipment.	No reported analytes detected > ½ LOQ.	Correct problem, then repeat cleaning of canister batch and recertify.	Analyst Lab QA Officer Project Chemist	Representativeness	No samples shall be collected until canisters are certified clean.
Tune check	Prior to ICAL and prior to each 24-hour period of sample analysis.	Specific ion abundance criteria of BFB from method.	Retune instrument and verify.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	No samples shall be analyzed without a valid tune.
ICV	Once after each ICAL.	All reported analyzes within ±30% of true value.	Correct problem and rerun ICV. If rerun fails, repeat ICAL.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	No samples shall be analyzed without a valid ICV.

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
CCV	Before sample analysis, after every 10 field samples, after every 12 hours of analysis time, and at the end of the analysis sequence.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ±30% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	Results may not be reported without a valid CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.
MB	Each time samples are extracted and one per matrix per analytical method for each batch of at most 20 samples.	No analytes detected >1/2 LOQ and greater than 1/10 the amount measured in any sample. For common lab contaminants, no analytes detected >LOQ.	Correct problem. If required, re-prep and reanalyze MB and all samples processed with the contaminated blank.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	No analytes detected >1/2 LOQ. For common laboratory contaminants, no analytes detected > LOQ.
LCS	One per batch of at most 20 samples analyzed of similar matrix per analytical method.	Per Method TO-15 and Lab SOP TBD.	Correct problem. If required, re-prep and reanalyze the LCS and all samples processed in the associated preparatory batch for the failed analytes.	Analyst Lab QA Officer Project Chemist	Accuracy	QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017).
Internal standards verification	Every field sample, standard, and QC sample.	Retention time for field and QC samples ± 20 seconds from most recent ICAL midpoint standard. Area response for IS in ICAL within 40% of the mean area response of ICAL standards; retention time shift within ± 20 seconds from ICAL standards.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision/ Representativeness	If corrective action fails, field sample data must be qualified and explained If reanalysis cannot be performed, data must be qualified and explained in the case narrative.
Surrogate spike	All field and QC samples.	Per Method TO-15 and Lab SOP TBD.	For QC and field samples, correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision/ Representativeness	QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017) for GC/MS.

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
Sample duplicate or LCSD	One measure of precision per analytical batch.	Per Method TO-15 and Lab SOP TBD. For LCSD, if more than 5% are outside of %RPD criteria, then inspect the system and re-analyze. Narrate exceedances. For samples, %RPD <25% for hits greater than 5× LOQ.	For LCSD, if more than 5% are outside of %RPD criteria, then inspect the system and re-analyze. Narrate exceedances. For samples, if %RPD exceeds criterion for hits greater than 5x LOQ, inspect system and re-analyze. Narrate non-compliance if still outside criterion.	Analyst Lab QA Officer Project Chemist	Precision	Target analytes ≤1/2 LOQ.

1 ICV instrument calibration verification

Matrix	**Soil Vapor**
Analytical Group	**VOCs, SVOCs**
Analytical Method/SOP Reference	Analytical/Preparation Method: EPA TO-17 Laboratory SOPs: TBD
Analytical Organization	Eurofins|AirToxics

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
LOD determination and verification	At initial set-up and verified quarterly. If a laboratory uses multiple instruments for a given method, the LOD must be verified on each.	The apparent signal to noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must: 1) Repeat the detection limit determination and LOD verification at a higher concentration; or 2) Perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	Analyst Lab QA Officer Project Chemist	Bias/ Representativeness	QC acceptance criteria as specified by Lab SOP TBD.
LOQ establishment and verification	At initial setup: 1) Verify LOQ; and 2) Determine precision and bias at the LOQ. Subsequently, verify LOQ quarterly. If a laboratory uses multiple instruments for a given method, the LOQ must be verified on each.	1) The LOQ and associated precision and bias must meet client requirements and must be reported; or 2) In the absence of client requirements, must meet control limits of the LCS. 3) If the method is modified, precision and bias at the new LOQ must be demonstrated and reported. See Volume 1, Module 4, Section 1.5.2 of the DoD QSM 5.1 (DoD 2017).	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	QC acceptance criteria as specified by Lab SOP TBD and at least as stringent as specified by DoD QSM 5.1 (DoD 2017).
Tune check	Prior to ICAL and prior to each 24-hour period of sample analysis.	Specific ion abundance criteria of BFB from method.	Retune instrument and verify.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	No samples shall be analyzed without a valid tune.
ICV	Once after each ICAL.	All reported analyzes within ±30% of true value.	Correct problem and rerun ICV. If rerun fails, repeat ICAL.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	No samples shall be analyzed without a valid ICV.
CCV	Before sample analysis, after every 10 field samples, after every 12 hours of analysis time, and at the end of the analysis sequence.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ±30% of true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision	Results may not be reported without a valid CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
MB	Each time samples are extracted and one per matrix per analytical method for each batch of at most 20 samples.	No analytes detected >1/2 LOQ and greater than 1/10 the amount measured in any sample. For common lab contaminants, no analytes detected >LOQ.	Correct problem. If required, re-prep and reanalyze MB and all samples processed with the contaminated blank.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	No analytes detected >1/2 LOQ. For common laboratory contaminants, no analytes detected > LOQ.
LCS	One per batch of at most 20 samples analyzed of similar matrix per analytical method.	Per Method TO-17 and Lab SOP TBD.	Correct problem. If required, re-prep and reanalyze the LCS and all samples processed in the associated preparatory batch for the failed analytes.	Analyst Lab QA Officer Project Chemist	Accuracy	QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017).
Internal standards verification	Every field sample, standard, and QC sample.	Retention time for field and QC samples ± 20 seconds from most recent ICAL midpoint standard. Area response for IS in ICAL within 40% of the mean area response of ICAL standards; retention time shift within ± 20 seconds from ICAL standards.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision/ Representativeness	If corrective action fails, field sample data must be qualified and explained. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.
Surrogate spike	All field and QC samples.	Per Method TO-17 and Lab SOP TBD.	For QC and field samples, correct problem then re-prep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Analyst Lab QA Officer Project Chemist	Accuracy/Precision/ Representativeness	QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017) for GC/MS.
LCSD	One measure of precision per analytical batch.	Per Method TO-17 and Lab SOP TBD. For LCSD, if more than 5% are outside of %RPD criteria, then inspect the system and re-analyze. Narrate exceedances. For samples, %RPD <25% for hits greater than 5× LOQ.	For LCSD, if more than 5% are outside of %RPD criteria, then inspect the system and re-analyze. Narrate exceedances. For samples, if %RPD exceeds criterion for hits greater than 5x LOQ, inspect system and re-analyze. Narrate non-compliance if still outside criterion.	Analyst Lab QA Officer Project Chemist	Precision	Target analytes ≤1/2 LOQ.

Matrix	**Soil Vapor**
Analytical Group	**Fixed Gases**
Analytical Method/SOP Reference	Analytical/Preparation Method: ASTM D1946 Laboratory SOPs: TBD
Analytical Organization	Eurofins|AirToxics

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Personnel Responsible for Corrective Action	DQI	Measurement Performance Criteria
LOD determination and verification	At initial set-up and verified quarterly. If a laboratory uses multiple instruments for a given method, the LOD must be verified on each.	The apparent signal to noise ratio must be at least 3 and the results must meet all method requirements for analyte identification.	If the LOD verification fails, the laboratory must: 1) Repeat the detection limit determination and LOD verification at a higher concentration; or 2) Perform and pass two consecutive LOD verifications at a higher concentration. The LOD is set at the higher concentration.	Analyst Lab QA Officer Project Chemist	Bias/ Representativeness	QC acceptance criteria as specified by Lab SOP TBD.
LOQ establishment and verification	At initial setup: 1) Verify LOQ; and 2) Determine precision and bias at the LOQ. Subsequently, verify LOQ quarterly. If a laboratory uses multiple instruments for a given method, the LOQ must be verified on each.	1) The LOQ and associated precision and bias must meet client requirements and must be reported; or 2) In the absence of client requirements, must meet control limits of the LCS. 3) If the method is modified, precision and bias at the new LOQ must be demonstrated and reported. See Volume 1, Module 4, Section 1.5.2 of the DoD QSM 5.1 (DoD 2017).	If the LOQ verification fails, the laboratory must either establish a higher LOQ or modify method to meet the client-required precision and bias.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	QC acceptance criteria as specified by Lab SOP TBD and at least as stringent as specified by DoD QSM 5.1 (DoD 2017).
MB	Each time samples are extracted and one per matrix per analytical method for each batch of at most 20 samples.	No analytes detected >1/2 LOQ and greater than 1/10 the amount measured in any sample. For common lab contaminants, no analytes detected >LOQ.	Correct problem. If required, re-prep and reanalyze MB and all samples processed with the contaminated blank.	Analyst Lab QA Officer Project Chemist	Sensitivity/Bias	No analytes detected >1/2 LOQ. For common laboratory contaminants, no analytes detected > LOQ.
LCS	One per batch of at most 20 samples analyzed of similar matrix per analytical method.	Per Method ASTM D1946 and Lab SOP TBD.	Correct problem. If required, re-prep and reanalyze the LCS and all samples processed in the associated preparatory batch for the failed analytes.	Analyst Lab QA Officer Project Chemist	Accuracy	QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017).
End check	At the end of the analytical sequence.	85–115%	Re-analyze standard. If still out of acceptance limits, then re-analyze samples as needed.	Analyst Lab QA Officer Project Chemist	Accuracy	QC acceptance criteria at least as stringent as specified by DoD QSM 5.1 (DoD 2017).

4.4 LABORATORY EQUIPMENT TESTING, INSPECTION AND MAINTENANCE

The analytical laboratory is responsible for inspecting and maintaining laboratory equipment as described in their laboratory QA plan (as specified by the analytical method used), and as described in *SAP Revision 01* (DON 2017c) and supplemented in Table 4-6.

Table 4-6: 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 [a]
GC-FID, GC-PID, GC-MS, EA-IRMS	Change gas purifier.	N/A.	Visually inspect if traps are changing color.	Every 6–12 months	No moisture	Replace indicating traps.	Analyst or certified instrument technician	TBD
	Change syringes/syringe needles.	N/A.	Visually inspect for wear or damage.	Every 3 months	N/A	Replace syringe if dirt is noticeable in the syringe.	Analyst or certified instrument technician	
	Change inlet liner, liner O-rings, and inlet septum.	N/A.	Visually inspect for dirt or deterioration.	Weekly for liner Monthly for O-rings Daily for septum	N/A	Replace and check often.	Analyst or certified instrument technician	
	Change front-end column.	N/A.	Check peak tailing, decreased sensitivity, retention time changes, etc.	Weekly, monthly, or when needed	N/A	Remove 1/2 to 1 meter from the front of the column when experiencing problems.	Analyst or certified instrument technician	
	Clean injector ports.	N/A.	N/A.	As needed	N/A	N/A.	Analyst	
	Replace trap on purge-and-trap systems.	N/A.	N/A.	Bi-monthly or as needed	N/A	N/A.	Analyst	
	Replace columns.	N/A.	N/A.	If chromatograms indicate possible contamination	N/A	N/A.	Analyst	
GC-FID	Replace detector jets.	N/A.	N/A.	As needed	N/A	N/A.	Analyst	TBD
	Replace hydrocarbon traps and oxygen traps on helium and hydrogen gas lines.	N/A.	N/A.	Every 4–6 months	N/A	N/A.	Analyst	
	Replace chemical trap.	N/A.	N/A.	Yearly or as needed	N/A	N/A.	Analyst	
	Replace converter tube in gas purifier system.	N/A.	N/A.	Yearly or as needed	N/A	N/A.	Analyst	
GC-PID	Replace PID lamp.	N/A.	Visually inspect	As needed	N/A	Change when lamp burns out	Analyst	TBD
	Clean PID lamp.	N/A.	Visually inspect	Decreased sensitivity	N/A	Clean PID lamp window to remove any residue	Analyst	

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference [a]
GC-MS	Change tune MSD, check the calibration vial, and replace the foreline pump oil.	N/A.	Visually inspect and monitor the fluid becoming discolored.	As needed or every 6 months	In accordance with manufacturer's recommendation or lab SOP	Keep plenty of PFTBA; refill the vial and check the fluid; change when the fluid becomes discolored.	Analyst or certified instrument technician	TBD
	Run tuning program to determine if source is functioning properly.	N/A.	N/A.	Daily	N/A	Cool system, vent, disassemble, and clean.	Analyst	TBD
	N/A	Tune instrument.	N/A.	Daily or every 12 hours	Per method	Liner and septa are replaced; tune file used is manually adjusted.	Analyst	
	Vacuum rough pump oil level is checked.	N/A.	N/A.	Every 4-6 weeks	N/A	Add oil if needed.	Analyst	
	Replace/refill carrier gas line oxygen and moisture traps.	N/A.	N/A.	Yearly or as needed	N/A	N/A.	Analyst	
Water bath (precision microprocessor controlled)	Check instrument connections, water level, and thermometer.	Measure water temperature against a calibrated thermometer.	Visually inspect for wear or damage and indicator from computer controls.	Daily and annual maintenance from manufacturer	Refer to manufacturer's recommendation	Return to manufacturer for recalibration or call for maintenance service.	Analyst or certified instrument technician	TBD
Analytical balance	Check digital LCD display and ensure a flat base for the Instrument.	Calibrate against verified (NIST) mass.	Visually inspect for wear or damage and indicator from computer controls.	Daily and annual maintenance from manufacturer	Refer to manufacturer's recommendation	Return to manufacturer for recalibration or call for maintenance service.	Analyst or certified instrument technician	TBD
pH meter	Check LCD display and pH probe.	3 point calibration using known standards.	Visually inspect for wear or damage and indicator from computer controls.	Daily and annual maintenance from manufacturer	±0.05 units	Return to manufacturer for recalibration or call for maintenance service.	Analyst or certified manufacture instrument technician	TBD

N/A not applicable
NIST National Institute of Standards and Technology
PFTBA perfluorotributylamine

4.5 LABORATORY INSTRUMENT CALIBRATION AND FREQUENCY

The analytical laboratory is responsible for calibrating laboratory equipment as specified by the analytical method used and as specified in Table 4-7.

Table 4-7: Analytical Instrument Calibration

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference [a]
GC-MS EPA Methods 8720 SIM Mod, TO-15, TO-17	Tuning	Prior to ICAL and at the beginning of each 12-hour period	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Lab Manager/Analyst or certified instrument technician	ANA8260, ANA8270, TBD
	Breakdown check (DDT-Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation ≤20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	Correct problem, then repeat breakdown checks.	Lab Manager/Analyst or certified instrument technician	
	Minimum 5-point ICAL for linear calibration Minimum 6-point ICAL for quadratic calibration	Prior to sample analysis	RSD for each analyte ≤15% or least square regression ≥0.995. Non-linear least squares regression (quadratic) for each analyte ≤0.995.	Correct problem then repeat ICAL.	Lab Manager/Analyst or certified instrument technician	
	Second source calibration verification	After ICAL	All analytes within ±20% of expected value.	Correct problem and verify second source standard; rerun second source verification. If fails, correct problem and repeat ICAL.	Lab Manager/Analyst or certified instrument technician	
	RT window position for each analyte and surrogate	Once per ICAL	Position will be set using the midpoint standard for the ICAL.	N/A.	Lab Manager/Analyst or certified instrument technician	
	RRT	With each sample	RRT of each target analyte in each calibration standard within ±0.06 RRT units of ICAL.	Correct problem, then reanalyze all samples analyzed since the last RT check. If fails, then rerun ICAL and samples.	Lab Manager/Analyst or certified instrument technician	
	CCV	Daily, before sample analysis, unless ICAL performed same day and after every 10 samples and at the end of the analysis sequence	All analytes within ±20% of expected value (%D). All reported analytes and surrogates within ±50% for end of analytical batch CCV.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Lab Manager/Analyst or certified instrument technician	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference [a]
GC-MS EPA Methods 8720 SIM Mod, TO-15, TO-17 (cont.)	IS	Each CCV and sample	RT ±10 seconds from RT of the ICAL mid-point standard. EICP area within -50% to +100% of area from IS in ICAL mid-point standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed during failure is mandatory.	Lab Manager/Analyst or certified instrument technician	ANA8260, ANA8270, TBD (cont.)
GC-FID, GC-FID-PID, GC-TCD MA VPH, MA EPH, TCEQ 1005, TCEQ 1006, ASTM D3328, ASTM D1946	Minimum 5-point ICAL for linear calibration Minimum 6-point ICAL for quadratic calibration (for MA VPH and MA EPH methods only)	Prior to sample analysis	RSD for each analyte ≤20% or least square regression ≥0.995. Non-linear least squares regression (quadratic) for each analyte ≤0.995.	Correct problem then repeat ICAL.	Lab Manager/Analyst or certified instrument technician	T-GC-WI9685, T-GC-WI9672, T-GC-WI9770, T-GC-WI9792, TBD
	Second source calibration verification (for MA VPH, MA EPH, TCEQ 1005, and TCEQ 1006 methods only)	Once after each ICAL	Analytes within ±20% of expected value (initial source), and within established RT windows.	Correct problem and verify second source standard. Rerun second source verification. If fails, correct problem and repeat ICAL.	Lab Manager/Analyst or certified instrument technician	
	RT window width	At method set-up and after major maintenance	RT width is ±3 times standard deviation for each analyte RT from 72-hour study. For TPH-d: calculate RT based on C12 and C25 alkanes.	N/A.	Lab Manager/Analyst or certified instrument technician	
	Establishment and verification of the RT window for each analyte and surrogate (for MA VPH MA EPH, TCEQ 1005, and TCEQ 1006 methods only)	Once per ICAL and at the beginning of the analytical shift for establishment of RT; and with each CCV for verification of RT	Using the midpoint standard or the CCV at the beginning of the analytical shift for RT establishment; and analyte must fall within established window during RT verification.	N/A.	Lab Manager/Analyst or certified instrument technician	
	Resolution Check (ASTM methods only)	Prior to sample analysis.	Pristane and phytane resolution ≥80% for peak pairs n-C17 and pristane, and peak pairs n-C18 and phytane.	Correct problem, then repeat performance checks. If resolution is <50% either or both peak pairs, replace column and check instrument operating conditions.	Lab Manager/Analyst or certified instrument technician	
	Run second source calibration verification (ICV) (for MA VPH MA EPH, TCEQ 1005, and TCEQ 1006 methods only)	ICV: Daily, before sample analysis, unless ICAL performed same day	All analytes within ±20% of expected value (%D).	Correct problem and rerun ICV. If fails, repeat ICAL.	Lab Manager/Analyst or certified instrument technician	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference [a]
GC-FID, GC-FID-PID, GC-TCD MA VPH, MA EPH, TCEQ 1005, TCEQ 1006, ASTM D3328, ASTM D1946 (cont.)	CCV (for MA VPH MA EPH, TCEQ 1005, and TCEQ 1006 methods only)	Daily, before sample analysis, unless ICAL performed same day and after every 10 samples and at the end of the analysis sequence	All analytes within ±20% of expected value (%D).	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Lab Manager/Analyst or certified instrument technician	T-GC-WI9685, T-GC-WI9672, T-GC-WI9770, T-GC-WI9792, TBD (cont.)
Water bath	Measure water temperature against a calibrated thermometer	Annually	In accordance with unit model and manufacturer's recommendation or laboratory SOP.	Terminate analysis, recalibrate, and verify before sample analysis.	Lab Manager/Analyst or certified instrument technician	INS001
Drying oven	Measure oven temperature against a calibrated thermometer	Annually	In accordance with unit model and manufacturer's recommendation or laboratory SOP.	Terminate analysis, recalibrate, and verify before sample analysis.	Lab Manager/Analyst or certified instrument technician	INO003
Analytical balance	Calibrate against verified (NIST) mass	Daily or prior to analyzing samples	In accordance with unit model and manufacturer's recommendation or laboratory SOP.	Terminate analysis, recalibrate, and verify before sample analysis.	Lab Manager/Analyst or certified instrument technician	INO011
pH meter	Run a minimum 3-point calibration; run CCV	Daily or prior to analyzing samples; one CCV for every 10 samples	±0.05 unit.	Terminate analysis, recalibrate, and verify before sample analysis.	Lab Manager/Analyst or certified instrument technician	INO038

%D percent difference
CA corrective action
CCV continued calibration verification
DDT dichlorodiphenyltrichloroethane
ICAL initial calibration
ICV initial calibration verification
IS internal standard
RRT relative retention time
RSD relative standard deviation
RT retention time
TBD to be determined
[a] See Table 4-2 for analytical SOP references.

5. Data Validation, Management, and Usability

Data review, verification and validation, and data management will be performed in accordance with procedures outlined in Section 8 of *SAP Revision 01* (DON 2017c) and supplemented as described below.

5.1 Additional Data Validation Procedures

All analytical laboratory data results for forensic, TPH-fractions, microbial, NAPs, and soil vapor analyses will be validated by the third-party data validation firm to assess method compliance, calibration frequency and acceptability, QC frequency and acceptability, and data usability in accordance with the DoD QSM Version 5.1 (DoD 2017) and associated NAVFAC Pacific Environmental Restoration Program Data Validation Procedures (DON 2015) previously identified in *SAP Revision 01* (DON 2017c) and supplemented below:

- Procedure II-W, *Level C and Level D Data Validation for GC/FID/ECD Volatile Organics and Fixed Gases in Soil Gas/Vapor by EPA Method TO-03 and ASTM D1946*
- Procedure II-X, *Level C and Level D Data Validation for GC/MS Volatile Organics and Fixed Gases in Soil Gas/Vapor by EPA Method TO-14, TO-15, and TO-17*

For analyses that have no applicable Data Validation Procedures (DON 2015), data will be validated in accordance with the analytical methods and the DoD QSM Version 5.1 (DoD 2017) as applicable. For analyses on which DoD QSM Version 5.1 (DoD 2017) does apply, data will be validated for QC compliance in accordance with the analytical methods and laboratory SOPs and qualified in accordance with the National Functional Guidelines for organic and inorganic data (EPA 2014b, 2014a).

5.2 Reconciliation with User Requirements

Data that have undergone review as discussed in Sections 5.1, 7, and 8 of the SAP (DON 2017c) will be evaluated against data quality objectives (DQOs) and project action levels (PALs). Any limitations associated with the data will be discussed in detail in the reporting document.

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Appendix A:
Standard Operating Procedures

NAVFAC Pacific Environmental Restoration Program (DON 2015):

- I-B-3 – Active Soil Gas Sampling
- I-C-2 – Monitoring Well Development
- III-C – Field QC Samples (Air)
- II-W – Level C and Level D Data Validation for GC/FID/ECD Volatile Organics and Fixed Gases in Soil Gas/Vapor by EPA Method TO-03 and ASTM D1946
- II-X – Level C and Level D Data Validation for GC/MS Volatile Organics and Fixed Gases in Soil Gas/Vapor by EPA Methods TO-14, TO-15, and TO-17

Microbial Insights:

- Bio-Flo – DNA Sampling Protocol
- Groundwater – DNA Sampling Protocol
- Bio-Trap – Stable Isotope Probing Protocol

Isotech Laboratories:

- Collection of Ground Water Samples from Domestic and Municipal Water Wells for Dissolved Gas Analysis Using IsoFlasks

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Active Soil Gas Sampling

1. Purpose

This standard operating procedure describes recommended soil gas sampling procedures for use by the United States (U.S.) Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command (NAVFAC), Pacific personnel.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in most recent version of the the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Definitions

3.1 ACTIVE SOIL GAS SAMPLING

The process of collecting a soil gas sample using mechanical equipment to create a hole, typically to a depth of 3 feet (or greater), and then using a vacuum pump to "actively" withdraw a soil-gas sample through stainless steel probes or plastic tubing. For whole gas sampling, the soil-gas sample is collected in a container that generally has a volume of 0.025 to 6.0 liters; for sorbent tube sampling, a volume of soil gas is drawn through a sorbent-packed tube, trapping contaminants onto the sorbent material. The gas/sorbent sample is then analyzed onsite by a mobile laboratory or sent to a fixed laboratory for analysis. The results provided by active soil-gas systems are most often reported in volume of gas per unit volume of air (e.g., parts per million by volume or part per billion by volume) or in units of mass per unit volume (e.g., milligrams per liter or micrograms per cubic meter).

3.2 PERMEABILITY

Permeability is the resistance of fluids, air, and gases to flow through a porous medium.

3.3 SATURATED ZONE

The saturated zone is the zone in which the voids in the rock (consolidated) or soil (unconsolidated) are filled with water at a pressure greater than atmospheric.

3.4 SOIL GAS

Soil gas is the atmospheric gases, vapor, and gaseous compounds present between soil particles in the subsurface. It can flow and migrate in response to pumping or pressure differentials.

3.5 VADOSE ZONE

The vadose zone is the unsaturated zone between the land surface and the water table. It includes the root zone, intermediate zone, and capillary fringe. The pore spaces contain water at less than atmospheric pressure, as well as air and other gases. Saturated bodies, such as perched groundwater, may exist in the unsaturated zone

3.6 FIXED GAS

Fixed gases are gases that are considered permanent by percent in the atmosphere (e.g. nitrogen gas [N_2] and oxygen gas [O_2]).

3.7 BIOGENIC GAS

Biogenic gas is a gas that is synthesized by biological activity (e.g. methane [CH_4] and carbon dioxide [CO_2]).

4. Responsibilities

The prime contractor CTO Manager is responsible for ensuring that the soil gas survey activities conducted during the NAVFAC Pacific ER Program comply with this procedure. It is recommended that supervisory personnel have an understanding of the principles of soil gas and the physical characteristics of the vadose zone. This should be determined in consultation with the QA Manager or Technical Director. To a certain extent, adequate understanding of the physical characteristics of the vadose zone by field supervisory personnel is site specific and is subject to the judgment of the QA Manager or Technical Director. The CTO Manager is responsible for ensuring that all personnel involved in sampling and/or testing shall have the appropriate education, experience, and training to perform their assigned tasks as specified in Chief of Naval Operations Instruction 5090.1, under *Specific Training Requirements* (DON 2014).

The prime contractor QA Manager or Technical Director is responsible for ensuring overall compliance with this procedure.

The Field Manager is responsible for ensuring that all project field staff are familiar with these procedures.

Field sampling personnel are responsible for the implementation of this procedure.

5. Procedures

5.1 CONSIDERATIONS FOR MUNITIONS AND EXPLOSIVES OF CONCERN

Potential Munitions and Explosives of Concern (MEC) hazards may be encountered in any area formerly or currently occupied or used by the Department of Defense (DoD). MEC hazards may occur on the ground surface, in the subsurface, and within bodies of water, and may not always be readily observable, or identifiable. As a result, whether or not munitions-related activities ever occurred on the specific work area or within waters in which Navy operations/activities will take place, special care should always be taken when conducting field operations, especially intrusive activities, in the event that MEC may be encountered.

If the site is currently recognized as belonging in the Military Munitions Response Program and has a current, Naval Ordnance Safety and Security-accepted, site-specific Explosives Safety Submission (ESS) (per DON 2010), then field activities, especially intrusive activities, shall adhere to the safety procedures outlined within the ESS.

If suspected MEC is encountered on an active DoD installation, immediately notify your supervisor, DoD Point of Contact, and installation Point of Contact, who will contact and facilitate military Explosive Ordnance Disposal response.

5.2 BACKGROUND INFORMATION

The soil gas survey is a semi-quantitative technique for evaluating the distribution of contaminants in soil gas. The resulting data can be used to qualitatively evaluate the potential for, and extent of, certain types of contamination in soil and groundwater.

Soil gas sampling is most often used to:

- Help locate and characterize areas of contaminated soil or groundwater.
- Evaluate subsurface contaminant vapor concentrations in areas where significant concentrations of volatile or semivolatile contaminants are known to be present.
- Identify the potential for contaminated vapor migration and intrusion into overlying and nearby buildings or structures.

If the extent of contamination in soil and groundwater is already known, then soil gas samples are typically not required for site characterization. Soil gas is also not an appropriate characterization technique for non-volatile contaminants such as metals.

There are two broad categories of active soil gas:

1. *Whole air sampling* is the collection of a volume of gas in a sample container. As the name implies, “whole air” samples remain in a gas matrix while in the sample container, thus concentrations of targeted compounds are directly reported. Sample containers associated with whole air sampling are the sample bag (e.g., Tedlar bag) and the canister (e.g. Summa canister).
2. *Sorbent tube sampling* is the drawing a volume of air through a sorbent tube using a pump or other vacuum source and trapping contaminants onto the sorbent material. Unlike whole air sampling, concentrations of targeted compounds are measured by determining the mass of contaminant on the sorbent material, and dividing that mass by the volume of air that was drawn through the sorbent material.

The use of active soil gas surveying to locate potential source areas of subsurface contamination is based on aqueous phase/vapor phase equilibrium in the subsurface. Volatile organic compounds (VOCs) have a tendency to partition from the aqueous phase into the soil vapor phase because of their relatively low solubilities and high vapor pressures. Certain semivolatile compounds also behave in this manner. Generally, an organic compound with a relatively high Henry’s Law constant (i.e., the ratio of a compound’s equilibrium concentration in air to its equilibrium concentration in water) is likely to partition from soil or groundwater into soil gas. The presence of VOCs in shallow

soil gas depends on the following factors: (1) the volatilization of VOCs from soil or groundwater into the soil gas, (2) the presence of a chemical gradient in soil gas between the contaminant source and the ground surface, and (3) the physical properties of the soil. If VOCs are present in the soil gas in large enough quantities, they can be detected during a soil gas survey.

Typical compounds detected in soil gas include aromatic hydrocarbons (benzene, toluene, ethylbenzene, xylenes), aliphatic hydrocarbons with carbon ranges from $C_1 – C_{10}$ (methane, butane, pentane), mixtures (gasoline, jet propellant-4), and chlorinated hydrocarbons (chloroform, carbon tetrachloride, vinyl chloride).

Fixed gas (i.e., O_2 and N_2) and biogenic gas (i.e., CO_2, CH_4, nitrous oxide, and hydrogen sulfide) data obtained during a soil gas investigation also provide an indication of potential subsurface contamination. A concurrent increase in CO_2 and decrease in O_2 often indicates increased chemical or biological breakdown of organic compounds. This phenomenon is usually associated with the degradation of petroleum hydrocarbons; however, moisture content, natural organic content, and reduction/oxidation (redox) conditions in the soil can also affect fixed gas/biogenic gas ratios.

5.3 EQUIPMENT AND SUPPLIES

Soil gas sampling requires specialized equipment to install sampling points and obtain air samples. The following equipment and supplies are typically required to conduct soil vapor sampling:

- Hydraulic driving/hammering system designed to drill through pavement and install sampling probes and soil vapor extraction wells at depth
- Stainless steel drive points for setting vapor probes at depth
- For installation of sub-slab vapor probes: a hammer drill and various sized drill bits to establish sampling locations through concrete
- Vapor probes (permanent or temporary)
- Tubing (Teflon preferably for sample lines directly in contact with vapor sample) and fittings
- Oil-less air pump, syringe or evacuation chamber for purging sample lines of ambient air, checking for air leaks in the sampling train, etc.
- Sample containers: Summa canister, sorbent tubes, vials etc.
- Photoionization Detector
- Fittings, tools, syringes
- Helium gas and a Helium Detector (if used for field leak-testing)

The following sections discuss the equipment considerations for whole air sampling and sorbent cartridge sampling.

5.3.1 Whole Air Sampling Equipment and Supplies

Whole air samples may be collected in Summa canisters, gas-tight vials, or sample bags. Canisters are the preferred sampling containers, as gas-tight vials can contain only small sample volumes and sample bags should not be air shipped because the bags may be compromised due to

pressure/temperature differentials during transport and tend to sorb contaminants. When low-level definitive data is required, it is recommended that canisters be used. Consideration to the canister volume should be taken when low-level data is required or re-analysis is anticipated. For the purposes of this standard operating procedure, discussion of whole air samples will be focused on air samples collected using canisters. Refer to the State of Hawaii Department of Health (DOH) Technical Guidance Manual for the Implementation of the Hawaii State Contingency Plan (TGM) for guidance on using other sampling containers (DOH 2009).

5.3.1.1 SUMMA CANISTER

A Summa canister is a stainless steel container that has had the internal surfaces chemically deactivated to produce a surface that is nearly chemically inert which is important for minimizing reactions with the sample and maximizing recovery of target analytes from the container. Recovery of compounds from canisters is limited to 10 carbon aromatic hydrocarbons compounds and 12 carbon aliphatic compounds.

Canister Certification for Cleanliness

The Summa canister is provided by a vendor, who certifies that the canister is clean. It is important to verify that the certificate sufficiently documents that project contaminants are not present in the canister at a detection limit appropriate for the site investigation.

Typically, Summa canisters are either “batched” or “individually” certified clean. The definitions of cleanliness will vary among vendors, thus it is important to review the project goals with the vendor providing the Summa canisters. In general, the cleaning process involves a combination of dilution, heat, and high vacuum.

For a “batched” certified clean canister, a canister within the same “batch” has been certified for a subset of VOCs using a gas chromatography/mass spectrophotometer (GC/MS) to have less than a specified concentration. Canisters that are “batch” certified are appropriate for collecting samples with anticipated high concentrations and routine ambient air applications. U.S. Environmental Protection Agency (EPA) methods TO-14 or TO-15 are the analytical methods used to analyze air samples where low level detections are not required (EPA 1999).

For an “individually” certified clean canister, each canister is certified clean for a client-specified list of target analytes below the laboratory’s specified detection limits. Canisters that are “individually” certified are appropriate for collecting ambient and indoor air samples which are driven by risk or litigation. When collecting air samples with an individually certified clean canister, it is important to use the flow controller and pressure gauge associated with the canister. EPA method TO-15 selective ion monitoring (SIM) analysis is the analytical method used to analyze air samples requiring low level detection.

Canister Holding Times

Once a canister is certified clean, the recommend time for sample collection is within 30 days of certification. Following sampling, laboratories prefer to have canisters returned within 14 days of air sampling although 30 days has been recognized by the EPA as acceptable. However, the stability of the chemicals of concern should be the driver on the number of days to analysis. Consult the laboratory for appropriate holding times.

Canister Volumes (Sizes)

Summa canisters are available in various volumes, but the most commonly used sizes are 1 liter and 6 liters. Six liter volumes are typically used for collecting ambient or indoor air samples that require low levels of detection. Ambient and indoor air samples are usually collected over an 8 hour or 24 hour period. One liter canister volumes are typically used for soil vapor sampling and is the minimum volume the State of Hawaii recommends for final decision making purposes. Sample collection periods will ultimately define the size of the Summa canister used.

Associated Canister Hardware

Hardware associated with Summa canisters includes vacuum gauges, flow controllers (critical orifice) and particulate filters.

The flow controller limits the rate that a sample can be drawn into the Summa canister and ensures the sample flow rate over the targeted sampling period (i.e., time-integrated sampling). In general, sampling flow rates should not exceed 200 milliliters per minute (mL/min) or sampling pressures greater than 7 inches of mercury. The vacuum drawn should not strip vapor from the soil but rather sample the vapor within the pore spaces of the subsurface at equilibrium.

Built into most flow controllers are 2 micron particulate filters which eliminate particulates larger than two microns. Grab air samples are usually fitted with a 7 micron particulate filter which removes particles larger than 7 microns.

The vacuum gauge is used to indicate the initial pressure of the canister before sampling and the final pressure after sampling. Laboratories typically prepare Summa canisters with a vacuum of approximately 30 inches of mercury. The vacuum gauges provided by the supplier are typically not calibrated and are meant to provide relative measures of change. Summa canister providers should be consulted to ensure the appropriate size canister and appropriate critical orifice is used to meet project requirements. In addition, if more stringent requirements are needed to monitor the canister pressure, then the canister supplier should be consulted for alternative pressure gauges.

5.3.2 Sorbent Tube Sampling Equipment and Supplies

Sorbent tube sampling may be used for short-chain VOCs or longer chained VOC and semivolatile compounds with molecular weights up to 200 grams per mole which cannot be recovered from canisters. Sorbent tubes are typically stored and shipped chilled (prior to and after sampling) at 4 degrees Celsius but should be brought to ambient temperatures prior to use.

Flow rates drawn through the sorbent tubes should be no more than 200 mL/min. A syringe or a pump may be used to draw air through the sorbent tubes. A pump is used when large volumes of air are required for sampling.

5.3.2.1 Selection of Sorbent Tubes

Selection of sorbent cartridges, solid phase sorbent and the evacuating system used to draw air through the sorbent will depend upon the target analytes of interest and the intent of data use. Sorbent tubes have a maximum sorption capacity before break through occurs. Consideration to the level of contamination will also influence the sorbent tube assembly. Consult with the analytical laboratory for selection of the appropriate sorbent material for the project analytes.

5.4 SELECTION OF SAMPLING LOCATIONS AND ANALYSES

The design of a soil gas survey program depends on the objectives of the program and the types of contaminants anticipated being present. The following items shall be considered when designing a soil gas program.

Number of Samples: This depends upon the extent of anticipated contamination, the size of the site, and the selected sample spacing.

Soil Types Expected to be Encountered (if known): The lithology of the subsurface must be considered when determining sampling locations, distance between samples, and sampling depth.

Depth of Samples: This will depend on the type of contamination, the depth to groundwater, and the objectives of the survey. For instance, evaluation of surface contamination may require only a 3- to 5-foot sampling depth whereas evaluation of deeper soil gas quality may require penetration to 20 feet. Samples may also be collected at several discrete intervals to provide a depth profile. Some flexibility exists in choosing a sampling depth or depths; however, once chosen, consistency across the site should be used.

Distance between Samples: For detecting the limits of plumes, spacing may be 50 to 100 feet or greater. Around a buried tank, spacing may be a few feet. Also consider the relative air permeability of the soil type(s) present. Soils with low air permeability (i.e., clays) may require closer sample spacing. Select spacing based on the objective(s) of the survey, subsurface conditions, and the nature of the target compounds. These factors shall be addressed in the project-specific work plan (WP).

Sampling Point Selection: Large spills, leaks, or plumes are often sampled on a predetermined sampling grid or by using real-time field data. Sample point selection for vapor intrusion concerns suggest locating subslab sample points within a building in areas where utilities or cracks serve as conduits for vapor intrusion, or commonly used spaces. Location access may also be an important factor.

Objectives of the Survey: If the objective is to define and delineate a soil vapor plume, then locate points throughout the suspected area. If the objective is to evaluate the potential for vapor intrusion into a building, then strategically locate subslab sampling locations within the building, at the source areas or around the building. In addressing vapor intrusion concerns, soil gas sample collection generally focuses in and around buildings within 100 feet of the source area. Point samples are used during the initial phases of an investigation to determine the extent and magnitude of subsurface vapor contamination and to assess potential exposure pathways. Soil gas samples are recommended from beneath the building slab and the potential source area. The depth beneath the subsurface and the target analytes should be taken into consideration when developing a soil vapor sampling strategy.

Timing of Sampling: Probe locations can be sampled in stages to meet the objectives of the survey. The first stage of sampling may involve widespread spacing of the probes. Later sampling should focus on areas where VOCs were detected during the first stage of sampling to define the lateral extent of soil gas contaminants, or delineate a source area. Later sampling events should include some overlap with earlier sampling points in order to provide a basis for correlation between data sets.

The frequency of a soil vapor investigation may also depend upon the objectives of the investigation. If vapor intrusion is a concern, collection of a soil vapor sample may occur in two distinct events to determine if seasonal/temporal variations change the soil vapor concentration beneath the slab or within the subsurface.

Selection of Analytes: Generally, only contaminants with relatively high Henry's Law constants are amenable to detection using soil gas; thus, analysis should focus on known indicator compounds at the site. Analytes should be selected to sample the compounds necessary to meet the objectives of the study and to maximize the number of locations sampled in a given period of time.

5.5 SAMPLING PROCEDURES

Summary Overview

Insert the soil gas probes into the ground using a hydraulic ram, pneumatic hammer, or other similar device. When the soil gas probe is at the desired sampling depth, a section of inert tubing is fitted to the top of the probe and connected to an air withdrawal system. The air withdrawal system is used to apply a vacuum to the system and draw soil gas from the surrounding formation into the probe. Purge the system for a sufficient amount of time to allow all of the atmospheric air to be removed, and ensure that a representative soil gas sample can be obtained. The amount of air to be removed is proportional to the volume of the sampling probe. Maintain an airtight seal around the soil gas probe at the ground surface to help prevent possible short-circuiting from atmospheric air diluting soil vapor gas concentrations. Purging of approximately 1.5 volumes permits removal of atmospheric air from the system with a minimum disturbance of the soil gas around the probe tip. Unlike purging of a groundwater monitoring well, purging of a soil gas probe should remove only the ambient air in the system. If a vacuum pump is used, record vacuum pressure and time required to purge the prescribed volume of gas from each probe to permit estimation of relative soil permeabilities.

When purging is complete, end the air withdrawals and allow the sampling system to return to atmospheric pressure. Withdraw the appropriate soil gas sample volume from the system using the vacuum in a Summa canister or pull soil gas through a sorbent tube using a vacuum pump.

Other methods of sample withdrawal and collection are acceptable as long as approval is obtained from the CTO Manager and QA Manager or Technical Director.

As part of the sampling procedure, record probe locations on a site map in accordance with Procedure I-I, *Land Surveying*. In addition, use field data forms (and chain-of-custody forms if necessary) to record observations regarding vapor sampling and probe installation. These field data forms may include, but are not limited to, vacuum pressures corresponding to steady flow, time required for the sampling system to reach atmospheric pressure, sampling depth, volume of soil gas extracted, soil characteristics, and procedures that are necessary to drive sampling probes to the target depth.

For additional information on the installation of temporary and permanent sample probes, please refer to the DOH TGM (DOH 2009, Section 7.9).

5.5.1 Purging Vapor Probe Locations

Once the vapor probe has been installed at the designated sampling location, the vapor probe should be purged of ambient or stagnant air. The volume of air space in sand packs installed with the vapor point should be included in purging if less than 24 hours has lapsed since installation of the probe.

5.5.2 Equilibrating Vapor Probe Locations

Following purging, vapor probe locations should be allowed to equilibrate. Equilibration times will depend upon the method of installation. The DOH TGM indicates that temporary probes reach equilibrium within 2 hours or so of installation, while permanent probes installed with a direct push rig typically require 8 to 24 hours. Vapor probes installed with a hollow auger are expected to require 48 hours of equilibration time. Equilibration times should be clearly communicated in the planning phases of a project and agreed upon by all project stakeholders.

5.5.3 Purging Sampling Trains

Following equilibration, connect an airtight canister assembly or sorbent cartridge assembly to the vapor probe using inert, rigid-walled tubing (i.e., Teflon, nylon, or stainless steel) and appropriate fittings.

This sampling train should be purged from ambient air prior to sampling. The rule of thumb for purging sample tubing is three tubing-volumes. The goal of purging is to have the tubing and other equipment filled with soil vapor prior to sample collection. Purging sample tubing can be accomplished by using a syringe, a pump or a canister and a three way valve. The purging of sample lines should not cause an excess vacuum on the soil. Stripping of vapor from soil should be avoided. The DOH TGM recommends that purging flow rates should not exceed 200 milliliters (mL) per minute and vacuum pressures should not exceed 7 inches of mercury.

5.5.4 Permeability Testing

While evacuating sample tubing of ambient air, the permeability of the sample location can be tested with a 20 or 50 mL syringe. If the syringe has difficulty drawing air from the probe location, the flow rates for sampling soil vapor need to be lowered. Alternatively, the sample location could be abandoned and another sampling location installed.

5.5.5 Leak Testing

Prior to sampling, the sample train should be leak tested. Leaks in a sampling train and the vapor probe surface seal can result in dilution of the soil vapor samples with ambient air resulting in low biased reported values. There are two types of leak tests: 1) a tightness test which checks the tightness of the sampling train and 2) a tracer test which measures the presence/absence of a compound introduced near the vapor probe surface seal. The design of the system used to perform a leak test will depend upon the installation design of the vapor probe.

The design of the tightness test should include isolating the sample train from the soil vapor probe and drawing a vacuum on the sampling train. The applied vacuum should hold for at least 60 seconds. If the vacuum does not hold, retighten connections of the sampling train and perform tightness test again.

There are two options for performing the tracer test: 1) *surface seal testing*, where the tracer compound (i.e., isopropanol) is applied to an absorbing material and placed on the vapor probe surface seal *or* a tracer gas (i.e., helium or difluoroethane) is introduced into a small shroud which is placed over the vapor probe surface seal; and 2) *whole apparatus testing*, where a tracer gas is introduced into a shroud which covers the entire sampling apparatus.

Of the two options, the surface seal testing is the easiest to implement and uses less tracer material. Feed-back on the integrity of the vapor point is immediate if helium is used as the tracer gas because presence of helium can be tested for in the field with a hand-held helium detector. The whole apparatus tracer test option can be implemented if the integrity testing of the entire sampling apparatus is required. Selection of the option used for leak testing should be agreed upon by all stakeholders.

There are multiple tracer compounds that can be used for leak testing. Three of the commonly used compounds are isopropanol, helium and difluoroethane. Each has advantages and disadvantages. The advantages of using isopropanol and difluoroethane, are that they are both inexpensive, readily available, and isopropanol does not require a shroud. There are several disadvantages of using these tracer compounds: 1) there are no available field meters that are selective for these compounds, so the presence of a leak is not known until the samples are analyzed by the laboratory; 2) if the compounds are present in high concentrations in the sample, they may interfere with laboratory analysis and elevate reporting levels above project action levels; and 3) the quantification of a leak can only be estimated because the initial concentration of the compounds in the field are unknown. The advantage of using helium is that it can be detected and quantified in the field. As a result, leaks at the sampling point can be determined immediately and the size of the leak can be quantified if the sample is tested for helium in the laboratory and the concentration of helium under the shroud maintained for the duration of the sampling period.

5.5.6 Prepare Summa Canister (Whole Gas Sampling)

Prior to sampling, verify the evacuated pressure of the canister (typically 30 inches of mercury). Do not use the canister if the pressure is less than 25 inches of mercury (or as appropriate for canister volume) and contact canister supplier. Record the initial pressure on the sample chain of custody (COC).

For grab sampling, verify the canister valve is closed and attach particulate filter. For time integrated sampling, close canister value and assemble the canister, flow controller and pressure gauge. Close assembly by capping the sample inlet. Check for leaks in assembly by opening and quickly closing the canister valve. If the needle on the vacuum gauge drops the assembly is not air tight. Refit and/or tighten connection until the needle on the vacuum gauge remains steady. Do not use assembly if it is not air tight.

5.5.7 Prepare the Sorbent Tube (Sorbent Tube Sampling)

Prior to sampling, tubes should be kept in their storage and transportation container and allowed to equilibrate with ambient temperature. The flow rates should be set using a dummy tube of identical construction. Using clean gloves, the sample tubes should be removed from the container and attached to the sampling lines. Any flow rate adjustments should be made quickly to avoid sampling errors. Then the flow rate should be monitored throughout the sampling process.

5.5.8 Collect Soil Gas

To begin, open canister valve at a half turn or start vacuum pump for the sorbent tube assembly. Record start date and time. Periodically monitor the progress of the air sampling by verifying sufficient vacuum pressure remains for entire sampling period for canister sampling. Monitor and record the flow rate periodically stable over the sampling period for sorbent tube sampling.

For canister sampling, the final vacuum pressure should preferably be 5 inches of mercury but are acceptable between 2 to 10 inches of mercury. If the vacuum is less than 1 inch of mercury, the air sample integrity may have been compromised. Record the final pressure and end date and time as well as the canister, controller and gauge serial numbers.

For sorbent tube sampling, turn off pump at pre-defined times, and seal sorbent tubes. Record the final flow rate, end date and end time, and the sorbent serial number.

5.5.9 Analytical Methods

The analytical method for soil gas samples collected using a sorbent cartridge is EPA Method TO-17 (EPA 1999). The analytical method for soil gas samples collected using a Summa canister depends upon the project objectives. For projects requiring indoor air risk drivers is typically analyzed by EPA Method TO-15 GC/MS SIM. For soil-gas data, EPA TO-14 or TO-15 should be sufficient. However, ultimately, the analytical method used, will depend upon the project specific needs. Consult the analytical laboratory for recommended analytical method to use.

5.5.10 Active Soil Gas Sampling Field Quality Control Samples

Duplicate soil vapor samples must be collected from the same sample location, using the same equipment and procedure as used for the original. The percentage of duplicates submitted for laboratory analysis depends on project-specific objectives and regulatory specifications that shall be defined in the WP. Purge the pump between sampling locations and check for residual VOC contamination by collecting field blanks for analysis.

5.5.11 Active Soil Gas Sampling Equipment Decontamination

Soil gas samples should not contact potentially sorbing materials such as the pump diaphragm or soft tubing. Check all components of the sampling system randomly for contamination by drawing atmospheric air through the system, subjecting it to analysis, and comparing the resulting chromatogram with that of ambient air. Use pre-cleaned probes for each sampling location in order to minimize the possibility of cross-contamination among sampling locations.

Clean sampling components, such as the probes, using steam or pressurized water and detergent at the conclusion of each day and clean immediately after use with a portable sprayer as described in Procedure I-F, *Equipment Decontamination*. Sampling syringes can be decontaminated and reused only if GC analyses indicate no residual contamination is present. Drive points placed at the ends of the steel sampling tubes are dedicated to one sampling location. Note that this procedure assumes that syringe sampling will be conducted. If other sampling techniques are preferred or required, document them in the project-specific WP.

5.5.12 Active Soil Gas Analytical Quality Control Samples

Blanks shall be run at least once for every 20 samples and after "hot" samples with concentrations outside the calibration range. A quality control standard containing concentrations in the middle range of those expected at the site shall be run at least once for every 20 samples, or at a minimum of once per day. In addition, a minimum of two ambient air samples shall be collected over the course of each day and analyzed for background concentrations of target compounds.

6. Documentation/Records

The subcontractor shall document each soil gas sampling event in a bound logbook or appropriate field log sheets. The following information will be recorded for each soil gas sampling event:

- Sample number
- Project name and number
- Sampling location and depth
- Date and time
- Name(s) of sampling personnel
- Site location
- Miscellaneous observations
- Analytical equipment utilized (e.g., GC, column, detector)

Other documentation will be recorded on a daily basis in the bound field notebook, and will include:

- Calibration results
- Blank measurement results

The original field records will be placed in the project files immediately upon completion of fieldwork. Subcontractors will prepare a detailed report summarizing the methodologies used during the survey, the results obtained, and an interpretation of the results. This report will be incorporated into the site characterization report or equivalent document.

7. Health and Safety

Field Personnel shall perform work in accordance with the current (or as contractually obligated) United States Army Corps of Engineers Safety and Health Requirements Manual EM-385-1-1 (USACE 2008) and site-specific health and safety plan.

8. References

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Environmental Protection Agency, United States (EPA). 1999. *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air.* 2nd ed. EPA-625/R-96-010b. Center for Environmental Research Information. January.

United States Army Corps of Engineers (USACE). 2008. *Consolidated Safety and Health Requirements Manual.* EM-385-1-1. Includes Changes 1–7. 13 July 2012.

Procedure I-F, *Equipment Decontamination*

Procedure I-I, *Land Surveying.*

Procedure I-A-5, *Utility Clearance*.

9. Attachments

None.

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Monitoring Well Development

1. Purpose

This section describes the standard operating procedures for monitoring well development to be used by United States Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command (NAVFAC), Pacific personnel.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the Uniform Federal Policy-Quality Assurance Project Plan (DoD 2005). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Definitions

None.

4. Responsibilities

The prime contractor CTO Manager is responsible for ensuring that these monitoring well development procedures are followed during projects conducted under the NAVFAC Pacific ER Program. The CTO Manager is responsible for ensuring that all personnel involved in monitoring well development shall have the appropriate education, experience, and training to perform their assigned tasks as specified in Chief of Naval Operations Instruction 5090.1, under *Specific Training Requirements* (DON 2014).

The prime contractor QA Manager or Technical Director is responsible for ensuring overall compliance with this procedure.

The Field Manager is responsible for ensuring that all project field staff follow these procedures.

Field personnel are responsible for the implementation of this procedure.

5. Procedure

5.1 INTRODUCTION

Well development procedures are crucial in preparing a well for sampling. They enhance the flow of groundwater from the formation into the well and remove the clay, silt, and other fines from the formation so that produced water will not be turbid or contain suspended matter that can interfere with chemical analyses. A monitoring well should be a “transparent” window into the aquifer from

which samples can be collected that are truly representative of the quality of water that is moving through the formation.

The goal of well development is to restore the area adjacent to a well to its natural condition by correcting damage to the formation during the drilling process. Well development shall accomplish the following tasks:

- Remove a filter cake or any drilling fluid within the borehole that invades the formation.
- Remove fine-grained material from the filter pack.
- Increase the porosity and permeability of the native formation immediately adjacent to the filter pack.

Well development shall not occur until 24 hours after the completion of well installation to allow the annular seal to fully set up.

5.2 FACTORS AFFECTING MONITORING WELL DEVELOPMENT

5.2.1 Type of Geologic Materials

Different types of geologic materials are developed more effectively by using certain development methods. Where permeability is greater, water moves more easily into and out of the formation and development is accomplished more quickly. Highly stratified deposits are effectively developed by methods that concentrate on distinct portions of the formation. If development is performed unevenly, a groundwater sample will likely be more representative of the permeable zones. In uniform deposits, development methods that apply powerful surging forces over the entire screened interval will produce satisfactory results.

5.2.2 Design and Completion of the Well

Because the filter pack reduces the amount of energy reaching the borehole wall, it must be as thin as possible if the development procedures are to be effective in removing fine particulate material from the interface between the filter pack and natural formation. Conversely, the filter pack must be thick enough to ensure a good distribution of the filter pack material during emplacement. The general rule is that filter pack material must be at least 2 inches thick.

The screen slot size must be appropriate for the geologic material and filter pack material in order for development to be effective. If slot size is too large, the removal of too much sediment may cause settlement of overlying materials and sediment accumulation in the casing. When screen openings are too small, full development may not be possible and well yield will be below the potential of the formation. Additionally, incomplete development coupled with a narrow slot size can lead to blockage of the screen openings.

5.2.3 Drilling Method

The drilling method influences development procedure. Typical problems associated with specific drilling methods include the following:

- If a mud rotary method is used, mud cake builds up on the borehole wall and must be removed during the development process.

- If drilling fluid additives have been used, the development process must include an attempt to remove all fluids that have infiltrated into the native formation.
- If driven casing or hollow-stem auger methods have been used, the interface between the casing or auger flights and the natural formation may have been smeared with fine particulate matter that must be removed during the development process.
- If an air rotary method has been used in rock formations, fine particulate matter is likely to build up on the borehole walls and may plug pore spaces, bedding planes, and other permeable zones. These openings must be restored during the development process.

5.3 PREPARATION

In preparing for monitoring well development, development logs for any other monitoring wells in the vicinity should be reviewed to determine the general permeability of the water-bearing formation and the appropriate development method.

Depth to groundwater and information from the well construction log should be used in calculating the required quantity of water to be removed. The distance between the equilibrated water level and the bottom of the screen is the saturated section. The saturated section (feet) multiplied by the unit well volume per foot (gallons/linear foot) equals the gallons required to remove one total well volume of water. The unit well volume is the sum of the casing volume and the filter pack pore volume, both of which depend upon casing and borehole diameter and the porosity of the filter pack material. Well volume can be calculated using Table I-C-2-1, Table I-C-2-2, or Table 1-C-2-3.

Table I-C-2-1: Casing Volume*

Casing Diameter (inches)	Volume (gallon/linear foot)
2	0.16
4	0.65
6	1.47

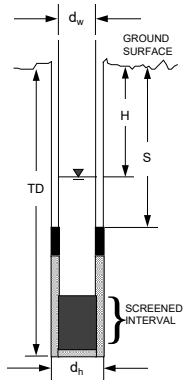
Table I-C-2-2: Filter Pack Pore Volume

Casing Diameter (inches)	Borehole Diameter (inches)	Volume [a] (gallon/linear foot)
2	6	0.52
2	8	0.98
4	10	1.37
4	12	2.09
6	12	1.76

* The above two volumes must be added together to obtain one unit well volume.
[a] Assumes a porosity of 40% for filter pack.

Table I-C-2-3: Well Volume Calculation

HOLE DIAMETER	d_h	=
WELL CASING INSIDE DIAMETER	d_wID	=
OUTSIDE DIAMETER	d_wOD	= _____
DEPTH TO: WATER LEVEL	H	=
BASE OF SEAL	S	= _____
BASE OF WELL	TD	=
EST. FILTER PACK POROSITY	P	= _____



WELL VOLUME CALCULATION:

CASING VOLUME = $V_c = \Pi\left(\frac{d_w ID}{2}\right)^2 (TD - H) = 3.14\left(\frac{\_\_\_\_}{2}\right)^2 (\_\_\_\_ - \_\_\_\_) = \_\_\_\_\_$

FILTER PACK PORE VOLUME = $V_f = \Pi\left[\left(\frac{d_h}{2}\right)^2 - \left(\frac{d_w OD}{2}\right)^2\right](TD - (S \text{ or } H^*)(P) = \_\_\_\_\_$

(* if S > H, use S; if S < H, use H)

$= 3.14\left[\left(\frac{\_\_\_\_}{2}\right)^2 - \left(\frac{\_\_\_\_}{2}\right)^2\right](\_\_\_\_ - \_\_\_\_)(\_\_\_\_) = \_\_\_\_\_$

TOTAL WELL VOLUME = VT = Vc + Vf = ____ + ____ = _____ $ft.^3$ x 7.48 = _____ gal.

5.4 DECONTAMINATION

The purpose of decontamination of development equipment is to prevent cross-contamination between monitoring wells. Use disposable equipment where appropriate. Use a steam-cleaner, if available, to decontaminate development equipment. Clean the equipment away from the monitoring well in such a fashion that decontamination effluent can be intercepted and drummed.

A triple rinse decontamination procedure is acceptable for equipment, such as bailers, or if access to a steam cleaner is not possible (Procedure I-F, *Equipment Decontamination*).

During well development, place visqueen around the well to prevent contamination at ground surface. Properly dispose of this sheeting after each use.

5.5 WELL DEVELOPMENT MONITORING

Throughout the well development process, maintain a development record using the form presented in Attachment I-C-2-1. The record should include the following information:

- General:
  - Project name and number
  - Well name/number and location
  - Date, time, and weather conditions
  - Names of personnel involved
- Development volume:
  - Initial and final water level
  - Casing total depth and diameter
  - Borehole diameter
  - Casing volume, filter pack pore volume, total well volume
  - Volume of water to be evacuated
  - Method and rate of removal
  - Appearance of water before and after development
- Monitoring data for each sample point:
  - Date, time, elapsed time
  - Cumulative gallons removed, removal method, removal rate
  - Temperature, pH (indicates the hydrogen ion concentration – acidity or basicity), specific conductivity, turbidity, dissolved oxygen, redox potential, and salinity

Part of the well development procedure shall consist of acquisition and analysis of water samples at appropriate intervals considering the total quantity of water to be removed. Measure conductivity, pH, temperature, dissolved oxygen, redox potential, turbidity, and salinity in each sample using a multi-parameter meter and flow-through cell. Collect readings on a periodic basis (approximately every 3 to 5 minutes) during development and obtain at least one reading after removal of each well

volume. At the time each sample is analyzed, record the cumulative water removed, the time, the time elapsed during development, and calculated flow rate. Continue development until at least 3 borehole volumes have been removed, turbidity stabilizes at or below 5 nephelometric turbidity units, and three successive readings of the parameters have stabilized (values within 10 percent of each other). If stabilization has not been attained, if turbidity remains high, or if the well does not readily yield water, allow the water level in the well to recover, conduct an additional 15 minutes of mechanical surging and/or bailing, then continue development until stabilization can be achieved or for a reasonable time.

Section 5.7 describes well development in special situations, such as low yield formations and 2-inch wells.

5.6 METHODS OF MONITORING WELL DEVELOPMENT

The methods available for the development of monitoring wells have been inherited from production well practices. Methods include: (1) mechanical surging with a surge block or swab, and (2) surge pumping. Development methods using air or jetting of water into the well are generally inappropriate for development of monitoring wells due to the potential for affecting water quality.

Containerize and appropriately label all development water (unless it is permissible to discharge it on site). All development efforts must utilize mechanical surging or surge pumping, followed by bailing or groundwater removal with a pump. More detailed descriptions of appropriate development methods are presented below.

5.6.1 Mechanical Surging and Bailing

For mechanical surging and bailing, a surge block or swab is operated either manually or by a drill rig. The surge block or swab should be vented and be of sufficient weight to free-fall through the water in the well and create a vigorous outward surge. The equipment lifting the tool must be strong enough to extract it rapidly. A bailer is then used to remove fine-grained sediment and groundwater from the well.

Procedures:

1. Properly decontaminate all equipment entering the well.
2. Record the static water level and the total well depth.
3. Lower the surge block or swab to the top of the screened interval.
4. Operate in a pumping action with a typical stroke of approximately 3 feet.
5. Gradually work the surging downward through the screened interval during each cycle.
6. Surge for approximately 10 to 15 minutes per cycle.
7. Remove the surge block and attach the bailer in its place.
8. Bail to remove fines loosened by surging until the water appears clear.

9. Repeat the cycle of surging and bailing at least three times or until turbidity is reduced and stabilization of water quality parameters occurs.
10. The surging shall initially be gentle and the energy of the action should gradually increase during the development process.

The advantages (+) and disadvantages (–) of this method are listed below:

+ Reversing the direction of flow reduces bridging between large particles, and the inflow then moves the fine material into the well for withdrawal.
+ It affects the entire screened interval.
+ It effectively removes fines from the formation and the filter pack.
– It might cause upward movement of water in the filter pack that could disrupt the seal.
– Potential exists for damaging a screen with a tight-fitting surge block or with long surge strokes.

5.6.2 Surge Pumping

Procedures:

1. Properly decontaminate all equipment entering the well.
2. Record the static water level and the total well depth.
3. Lower a submersible pump or airlift pump without a check valve to a depth within 1 to 2 feet of the bottom of the screened section.
4. Start pumping and increase discharge rate to maximum capacity (overpumping), causing rapid drawdown of water in the well.
5. Periodically stop and start the pump, allowing the water in the drop pipe to fall back into the well and surge the formation (backwashing), thus loosening particulates.
6. The pump intake shall be moved up the screened interval in increments appropriate to the total screen length.
7. At each pump position, the well shall be pumped, overpumped, and backwashed alternately until satisfactory development has been attained as demonstrated by reduction in turbidity and stabilization of water quality parameters.

The advantages (+) and disadvantages (–) of this method are listed below:

+ Reversing the direction of flow reduces bridging between large particles, and the inflow then moves the fine material into the well for withdrawal.
+ It effectively removes fines from the formation and filter pack.
– The pump position or suction line must be changed to cover the entire screen length.

- Submersible pumps suitable to perform these operations may not be available for small diameter (1 inch or less) monitoring wells.
- It is not possible to remove sediment from the well unless particle size is small enough to move through the pump.

For additional information on well development, consult the references included in Section 8 of this procedure.

5.7 SPECIAL SITUATIONS

5.7.1 Development of Low Yield Wells

Development procedures for monitoring wells in low-yield (<0.25 gallons per minute), water-bearing zones are somewhat limited. Due to the low hydraulic conductivity of the materials, surging of water in and out of the well casing is difficult. Also, the entry rate of water is inadequate to remove fines from the well bore and the gravel pack when the well is pumped. Additionally, the process may be lengthy because the well can be easily pumped dry and the water level is very slow to recover.

Follow the procedures for mechanical surging and bailing for low yield wells. During surging and bailing, wells in low yield formations should be drawn down to total depth twice, if possible. Development can be terminated, however, if the well does not exhibit 80 percent recovery after 3 hours.

5.7.2 Development of 2-inch Wells

It is easier to develop monitoring wells that are large in diameter than small diameter wells. Mechanical surging or bailing techniques that are effective in large diameter wells are much less effective when used in wells 2 inches or less in diameter. Mechanical surge blocks and bailers have a high potential for damaging a small diameter well. As a result, the CTO Manager shall obtain approval from the QA Manager or Technical Director prior to installing groundwater monitoring wells with inside diameters of 2 inches or less.

Develop two-inch or smaller diameter wells by surging with a specially designed, hand-operated surge block or by pumping with a bladder or airlift pump. Information related to development of wells 2 inches or less in diameter shall be included in the CTO work plan.

6. Records

Well development information should be documented in indelible ink on well development monitoring forms (Attachment I-C-2-1). Copies of this information shall be sent to the CTO Manager and to the project files. The CTO Manager shall review all well development logs on a minimum monthly basis.

7. Health and Safety

Field personnel shall perform work in accordance with the current (or as contractually obligated) United States Army Corps of Engineers Safety and Health Requirements Manual EM-385-1-1 (USACE 2008) and site-specific health and safety plan.

8. References

Department of Defense, United States (DoD). 2005. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

Department of the Navy (DON). 2014. *Environmental Readiness Program Manual*. OPNAV Instruction 5090.1D. 10 January.

United States Army Corps of Engineers (USACE). 2008. *Consolidated Safety and Health Requirements Manual.* EM-385-1-1. Includes Changes 1–7. 13 July 2012.

Procedure I-F, *Equipment Decontamination.*

9. Attachments

Attachment I-C-2-1: Well Development Record

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Attachment I-C-2-1
Well Development Record

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WELL DEVELOPMENT LOG

PROJECT	WELL ID	SITE	PREPARED BY

METHOD
- OVERPUMPAGE ☐
- BAILER ☐
- SURGE BLOCK ☐
- AIR LIFT ☐
- OTHER _____

INITIAL WATER LEVEL:
INITIAL TOTAL DEPTH:

FINAL WATER LEVEL:
FINAL TOTAL DEPTH:

REMARKS:

*CAPACITY OF CASING (GALLONS/LINEAR FOOT)

1" - 0.012
2" - 0.16
4" - 0.65
6" - 0.1.47

*VOLUME BETWEEN CASING AND HOLE (GALLONS/LINEAR FOOT) (ASSUMING 40% POROSITY)

1" CASING AND 2" HOLE - 0.013
2" CASING AND 6" HOLE - 0.52
2" CASING AND 8" HOLE - 0.98
4" CASING AND 10" HOLE - 1.37
4" CASING AND 12" HOLE - 2.09

*The two volumes for the saturated portion of the well must be added together to obtain one unit well volume.

DEVELOPMENT LOG:					CUMULATIVE WATER REMOVED	WATER QUALITY							COMMENTS
DATE	TIME	METHOD	ELAPSED TIME	FLOW RATE (gpm)	GALLONS	pH	TEMP (ºC)	SP. COND (mS/cm)	DO (mg/L)	ORP (mV)	TURBIDITY (ntu)	SALINITY (ppt)	

DO dissolved oxygen
ORP oxidation-reduction potential

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Field QC Samples (Air)

1. Purpose

This standard operating procedure describes the standard quality assurance (QA) and quality control (QC) procedures for air monitoring field samples for use by United States (U.S.) Navy Environmental Restoration (ER) Program, Naval Facilities Engineering Command (NAVFAC), Pacific personnel.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility. Specific guidance for collecting field QC samples will be addressed in project-specific planning documents.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the most recent version of the Uniform Federal Policy-Quality Assurance Project Plan Part 1 (DoD 2005a), 2A (DoD 2012), and 2B (2005b), as well as the DoD Quality Systems Manual (DoD 2013). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the Contract Task Order (CTO) Manager and the QA Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Definitions

3.1 DIRECT WHOLE AIR SAMPLING AND SORBENT SAMPLING

The following two soil gas and indoor air sampling methods are described in detail in this procedure: (1) whole air sampling and (2) sorbent tube sampling using an adsorbent material or combination of sorbents. Impinger and filter methods will be not be discussed.

3.1.1 Direct Whole Air Sampling

Whole air sampling describes air samples collected and analyzed in the gaseous phase. This sampling method may be used for collection of fixed gases, reduced sulfur compounds, and volatile organic compounds (VOCs).

Air samples may be collected in a sample canister (e.g., Summa canister) or a sample bag (e.g., Tedlar bag). Sample canisters are stainless steel vessels designed to hold a vacuum and have specially treated interior surfaces that are nearly chemically inert. Evacuated canisters do not require a sampling pump for sample collection. However, because canisters may be prone to contamination issues, they must pass a canister cleaning certification, as well as vacuum check readings. Bags, however, require a sample pump or lung sampler, but can be purchased inexpensively in bulk, require little preparation, and are certified by the manufacturer. U.S. Environmental Protection Agency (EPA) Methods TO-13 (polycyclic aromatic hydrocarbons [PAHs]), TO-14 (VOCs), and TO-15 (VOCs) are all commonly used for analysis of whole air samples.

3.1.2 Sorbent Sampling

Sorbent sampling describes a technique where air is drawn through a tube filled with a solid sorbent (or combination of sorbents) material and contaminants in the air are chemically adsorbed onto the material in the tube. This sampling method may be used for collection of many VOCs and semi-volatile organic compounds (SVOCs), including PAHs, polychlorinated biphenyls (PCBs), and pesticides. However, many compounds in the PAH, PCB, and pesticide classes would not normally be volatile at normal room temperature. Therefore, air sampling for these compounds is not definitive for their presence as contaminants in the non-volatile phase (i.e., in soil or water). Sorbent materials are specific to capture a particular chemical or class of chemicals. To collect samples, a personal sampling pump is needed. The pump should be calibrated with an air flow calibrator to pull sample air through the sorbent tube at a known flow rate for a known amount of time, to determine an accurate sample volume. EPA Method TO-17 is commonly used for analysis of VOCs in sorbent tube samples.

3.2 TRIP BLANK

Trip blanks typically pertain to sorbent tubes; trip blanks for air sample bags and canisters can be prepared by the laboratory by filling with zero air. For tubes, the trip blank is prepared and added to the site samples after sampling has been completed, but prior to shipment. The ends of the sorbent tube should be broken, but no air drawn through the tube when it is added to the group of samples. It should then remain unsealed throughout the shipping process. The trip blanks for bags and canisters are shipped to the field and remain unopened (i.e., are not used for sampling), and are treated in the same manner as project samples (i.e., the trip blank will accompany project samples throughout the sample collection and analysis process; including return to the laboratory for analysis). Analysis of a trip blank provides information about the storage, shipping and handling procedures and evaluates if contaminants were introduced into the sample or if the original packing material or laboratory equipment was potentially contaminated. Trip blanks are typically not necessary for air samples collected in passivated canisters, but may be requested by the CTO Manager.

3.3 FIELD BLANK

A field blank is a sample collected in the field from a certified air or nitrogen source. Field blanks are associated with active air sampling with summa canisters or sorbent tubes and are recommended if decontamination procedures are employed on non-disposable air sampling equipment. A field blank shall be submitted for sorbent tubes for each day of sampling. The ends of the sorbent tube are broken at the beginning of the day and resealed, but no air is drawn through the tube. Analysis of a field blank provides information about the equipment used for air sample collection (i.e., stainless steel lines, pressure gauges, and sample tubing, vapor probe parts). If a field blank is collected with a summa canister, it is important that individually certified canisters and a certified source air/nitrogen are used.

3.4 AMBIENT BLANK

An ambient blank is an ambient air sample collected in the field and represents the ambient levels of site contaminants. Ambient blanks are typically associated with active soil gas (i.e., indoor air, subslab, or soil vapor) or stationary source sampling. Analysis of the ambient blank can provide information on the ambient levels of site contaminants. Ambient air sample locations should be in an area near the sampling locations that has unobstructed airflow, especially in the direction of any recognized sources of the materials being sampled. Once a sample location is identified, the ambient

blank is collected in the same manner as the sample. For sorbent tubes and bags, this means actively drawing air through the tube or into the bag. For canisters, this means opening the valve and monitoring the vacuum gauge. When the sampling is complete, the valve is closed and the final vacuum is recorded. It is recommended that an individually certified canister be used to collect an ambient blank.

3.5 FIELD DUPLICATE

A field duplicate is a duplicate sample collected in the field simultaneously with the primary sample at a single sampling location. For sorbent tubes, duplicate samples are collocated samples collected from side-by-side locations at the same time. For canisters, duplicate samples are collected from a common sampling port where tubing from the inlet is connected to a sampling "T," and the outlets of the sampling "T" are connected to individual air sampling trains (typically consisting of a flow controller, pressure gauge, and collection vessel). If the collection flow rate is to be maintained, then the tubing from the sampling port is connected to a flow controller in series with the sampling "T." Analysis of field duplicates provides information about the precision of the field sampling procedures.

3.6 SPIKED SAMPLE

A spiked sample consists of a canister/cylinder or sorbent to which a known amount of the analyte(s) of interest has been added by the laboratory and is shipped to the field for use. A spike sample may be desirable in situations in which high concentrations of contaminants other than the target compounds are found to exist (e.g., landfills). The additional level of QA/QC attained by this practice can be useful in determining the effects of interferences caused by these non-target compounds. The spiked sample is kept with the air samples that have completed sample collection. Analysis of spiked samples provides information regarding the integrity of the canister/cylinder or sorbent to retain analyte(s) of interest during the air sampling process.

4. Responsibilities

The prime contractor CTO Manager is responsible for ensuring that the air monitoring and sampling QA/QC activities are followed during projects conducted under the NAVFAC Pacific ER Program. The prime contractor's QA Manager or Technical Director, as well as QC coordinators will be responsible for compliance with the procedure. These QA/QC activities will be implemented in accordance with the work plan (WP) for the respective CTO activity. The CTO Manager is responsible for determining which team members shall record information in the field logbook and for checking sample logbooks and chain-of-custody (COC) forms to ensure compliance with these procedures. The CTO Manager shall review COC forms on a monthly basis at a minimum. The CTO Manager is responsible for ensuring that the air monitoring and sampling QA/QC activities are followed during projects conducted under the NAVFAC Pacific ER Program. These QA/QC activities will be implemented in accordance with the work plan (WP) for the respective CTO activity. The CTO Manager is responsible for ensuring that all personnel involved in sampling and/or testing shall have the appropriate education, experience, and training to perform their assigned tasks as specified in Chief of Naval Operations Instruction 5090.1, under *Specific Training Requirements* (DON 2014).

The Laboratory Project Manager or Sample Control Department Manager is responsible for reporting any sample documentation or COC problems to the CTO Manager or CTO Laboratory

Coordinator within 24 hours of sample receipt. The Laboratory Manager is responsible for ensuring that field QC samples are analyzed according to the specifications of the project Statement of Work and the analytical methods used.

The Field Manager is responsible for ensuring that all field personnel follow these procedures. The CTO Laboratory Coordinator is responsible for verifying that the COC/analytical request forms have been completed properly and match the sampling and analytical plan. The CTO Manager or CTO Laboratory Coordinator is responsible for notifying the laboratory, data managers, and data validators in writing if analytical request changes are required as a corrective action. These small changes are different from change orders, which involve changes to the scope of the subcontract with the laboratory and must be made in accordance with a respective contract (e.g., Comprehensive Long-Term Environmental Action Navy, remedial action contract).

NAVFAC Pacific ER Program field personnel are responsible for following these procedures while conducting sampling activities. Field personnel are responsible for recording pertinent data into the logbook to satisfy project requirements and for attesting to the accuracy of the entries by dated signature.

5. Procedures

NAVFAC Pacific ER Program air monitoring program consists of complex activities using air sampling equipment and laboratory analysis techniques. This approach is necessary to accurately and consistently quantify concentrations of contaminants in ambient air, indoor air, subslab vapor, and soil gas. Therefore, it is critical to ensure and maintain a high-quality program by implementing the appropriate QA/QC program elements.

QA and QC activities are both concerned with maintaining consistent and verifiable quality in each element of the program. Strictly speaking, QC applies to measures taken, on an ongoing basis, by personnel involved in producing the primary output of the activity. These actions are taken to maintain performance parameters within acceptable levels. An example of a QC activity is a routine zero/span calibration check of a monitoring instrument by the responsible operating technician.

QA refers to checks or tests performed by personnel other than the primary operators to verify that the performance parameters have, in fact, been maintained within acceptable limits. Examples of QA activities include performing a quarterly audit of monitoring instruments and checking output data for “out-of-limits” values. In the discussion that follows, QA/QC is used as a general term to encompass both QA and QC activities.

To meet monitoring objectives, a rigorous QA/QC effort is necessary during the operation of an air monitoring program. Major QA/QC elements that shall be implemented during the operational phase of an air monitoring program include QA/QC management, sample QA/QC, analytical QA/QC, and data reduction QA/QC.

QA management involves implementing project-specific task order administrative procedures to control QA/QC functions. The potential for, and types of, quality problems vary depending on the activity: sampling, analysis, or data reduction. Therefore, individual QA/QC requirements must be developed for each of these activities. Summaries of typical sampling and analysis frequencies, QA/QC requirements, and calibration requirements for sampling and analysis instrumentation are presented in Table III-C-1 and Table III-C-2, respectively.

Data recording procedures to be specified in the air sampling activity include: (1) periodic readings of the temperature, flow, volumes, and other parameters; (2) documentation of meteorological conditions at appropriate time points; (3) documentation of instrument operating variables (i.e., resin cartridge number); (4) documentation of any upset conditions, such as sudden leakage or pressure surges; and (5) documentation of calibration or maintenance activities. Maintain a logbook for the overall field program that documents sampling descriptions, meteorological data, and upset conditions. In addition, prepare a sampling data sheet for each sample or set of samples in which the periodic readings and instrument parameters are recorded. Maintain separate maintenance and calibration logbooks for each sampling/monitoring instrument. In most cases, sampling data forms specific to a given CTO must be prepared because of differences in the sampling design between CTOs.

Table III-C-1: Typical Sampling/Analysis Frequencies for QC Samples (Air)

Type of Sample [a]	Description	Typical Frequency
Field blank (Sorbent tubes)	A sorbent tube field blank is a sample collected in the field by breaking the ends of the tubes at the beginning of the day, resealing the tube, and shipping with samples. Analysis of a field blank provides information about sample handling procedures.	If project objectives require field blanks, then at least one per day.
Field blank (Sample bags or canisters)	A sample bag or canister field blank is a sample collected in the field from a certified air or nitrogen source passed through non-disposable equipment. Analysis of a field blank provides information about the cleanliness of equipment and sample handling procedures.	If project objectives require field blanks, then at least one per sampling event.
Ambient Blank	An ambient blank is an ambient air sample collected in the field in the same manner as the samples and represents the ambient level of site contaminants.	At least one per distinct site location (i.e., for each building or area of concern).
Trip blanks (Sorbent tubes, filters or liquids)	For sorbent tubes, filters, or liquids, the trip blank is prepared and added to the site samples after sampling has been completed, but prior to shipment. The ends of the sorbent tube should be broken, but no air drawn through the tube when it is added to the group of samples. Analysis of a trip blank provides information about sample shipping procedures.	At least one per shipment media.
Trip blanks (Sample bags or canisters)	The trip blanks for bags and canisters are shipped to the field and remain unopened (i.e. are not used for sampling), and are treated in the same manner as project samples (i.e. the trip blank will accompany project samples throughout the sample collection and analysis process; including return to the laboratory for analysis). Analysis of a trip blank provides information about the storage, shipping and handling procedures and evaluates if contaminants were introduced into the sample or if the original packing material or laboratory equipment was potentially contaminated.	At least one per shipment media.
Spiked sample	Media to which a known amount of the analyte(s) of interest has been added by the laboratory and is shipped to the field for use.	At least once, to establish the integrity of the sample collection vessels provided by the laboratory.
Field duplicate	A field duplicate is a duplicate sample collected in the field simultaneously with the primary sample at a single sampling location.	One per distinct site location (i.e., for each building or area of concern or at minimum 10% of the sample total).
Instrument calibration standards	Calibration devices or material traceable to known certified standards.	Test at least twice daily at the beginning and end of the sampling period.

% percent
* Specify the manner in which these types of samples are collected within the specific CTO WP.

Table III-C-2: Calibration Requirements for Field Air Sampling and Analysis Instrumentation

Device	Parameter Calibrated	Method of Calibration	Approximate Frequency	Comments
Sampling Instrumentation				
Sampling flow rate measurement device/air flow calibrator	Flow rate	Flow calibration kit; primary standard film calibrator; calibration flow meter; dry test meter	Dependent on the sampler; generally immediately prior to and after the sampling event	None
Sample volume measurement device (usually a dry test meter)	Total volume	Wet test meter or any appropriate volume standard	Dependent on the sampler; generally immediately prior to and after the sampling event	Must be determined at known atmospheric pressure and temperature. Flow rate should be similar to that used for sampling.
Sample pump (used with sorbent tubes)	Flow rate	Flow calibration kit; calibration flow meter; bubble generator calibrator	Dependent on the sampler; generally immediately prior to and after the sampling event	Flow rate should be similar to that used for sampling.
Mass flow controller (used with canisters)	Flow rate	Flow calibration kit (with four times better collective accuracy than the mass flow controller recommended)	Before and after sampling	Calibrated in the laboratory.
Continuous monitors (i.e., FID, PID, FPD)	Response	Standard concentrations	Daily or more frequently, if required	Test atmosphere should be referenced to primary standard (i.e., NIST, SRM, or CRM). Flow/pressure conditions should duplicate sampling process.
Field gas chromatographic instruments	Column performance and response retention time for each analyte	Injection of standard using the same process as for sample injection	Daily or more frequently, if required	Standard composition should be checked against primary standards if available.

CRM Certified Reference Material
FID flame ionization device/detector
FPD flame photometric detector
NIST National Institute of Standards and Technology
PID photoionization detector
SRM Standard Reference Material

In addition to site-specific air sampler(s) and meteorological station parameters, note the monitored area or locale elevation (i.e., feet above mean sea level). Other types of readings that may be taken include the following: ambient air temperature, relative humidity, and barometric pressure. These values may be obtained from either a nearby National Weather Service station or an airport that measures/records these parameters.

Specify sample labeling, preservation, storage, and transport procedures, and carefully explain these procedures to field personnel prior to sampling to ensure proper implementation. Sample labels, prepared in advance, should include sufficient information to associate the sample with a particular data sheet, as well as the overall program record notebook. In general, give each sample a unique identification number with a prefix describing the type of sample.

Sample preservation, storage, and transport procedures must be appropriate for the type of analyses required. Generally, place particulate samples in airtight containers and store them in the dark to minimize analyte degradation. Resin cartridges, and sorbents generally require more attention because of analyte instability in the matrix, and should be shipped to the laboratory on the same day that the sample was collected for analysis. Place these sample types in airtight, glass containers and store them at subambient temperatures until analysis. Avoid exposure to solvents for resin cartridges during all stages of handling to avoid sample contamination. Place air samples collected in Tedlar bags within an opaque plastic bag (i.e., plastic garbage bag), and then place them in an appropriate opaque shipping container at room temperature for shipment to the laboratory on the same day that the sample was collected for analysis. It should be noted that the use of Tedlar bags for air sampling is not recommended if the sample needs to be shipped to the analytical laboratory via airplane due to the potential for the Tedlar bag to be compromised due to significant pressure/temperature differentials during transport.

COC forms are required. The objective of the COC procedure is to document the movement of a sample from collection until analysis to ensure its integrity.

5.1 ROUTINE QA/QC CHECKS

The field air monitoring program should incorporate the following four-component approach for routine QA/QC checks:

- Use field duplicate samples for precision checks.
- Use ambient blanks and field blanks for ambient locale, shipping container, and/or sampling train contamination checks, respectively.
- Use analytical standards and equipment calibrations for accuracy checks.
- Perform data review for internal consistency.

During each air monitoring sampling event, collect one field duplicate per area of concern or at least 10 percent of the total number samples collected. Use the analytical results from the field duplicate air samples to assess the precision and overall homogeneity of the samples, including the influence of the combined field and analytical procedures.

The purpose for collecting sample blanks (i.e., field blank) is to document that extraneous concentrations of the target analyte(s) are not introduced into the collection medium by handling or working with it in a normal, routine fashion. Generally, one field blank per collection medium per day is sufficient to demonstrate the levels of target analyte(s) found in the normal handling of the media in the field. In some instances, where the field environment is known to have high levels of contaminants, collecting more field blanks may be deemed appropriate. However, the WP should identify those requirements.

The exact procedure for collecting field blanks is specific to each type of medium. However, the general concept is to handle the field blank media in exactly the same fashion as the media used for actual sample collection except no sample volume of air is moved through the media. For example, glass sorbent tubes used for field blanks are shipped, labeled, have their ends broken open, are placed in the sampling mechanism, removed from the sampling mechanism, capped, logged, and packed for shipment in an identical manner to the sorbent tubes used to collect air samples. The difference is

that no air is pulled through the field blank sorbent tubes. Table III-C-3 lists general procedures for collecting field blanks for some of the collection media.

Table III-C-3: Common Field Blank Collection Procedures (Air)

Media	Field Blank Collection Procedure
Sorbent tubes	The tubes are removed from their shipping package and labeled as if they were to be used to collect samples. The tube ends are snapped off and the tubes are placed in the sampler mechanism (e.g., personal sampling pump or flow control device). Without turning on the sampler mechanism, the tubes are then removed, capped, logged, and placed in the shipping container along with regular samples for transport to the laboratory. Analysis of the field blank tubes is identical to the tubes used to collect air samples.
Tedlar bags	The empty bags intended for field blanks are removed from the shipping package and labeled as if they are to be used for normal sample collection. The field blank bags are then filled with ultra pure nitrogen, logged, and packaged for shipment to the laboratory along with normally collected air samples.
Summa canisters	The canisters intended for field blanks are removed from the shipping container and labeled as if they are to be used for normal sample collection. The field blank canisters are then filled with ultra-pure nitrogen, logged, and packaged for shipment to the laboratory along with canisters used for normally collected air samples.

Test canisters (Summa-stainless steel, SilcoCan-fused silica/stainless steel) used for ambient air sampling purposes to determine vacuum/pressure condition before and after sampling. Evacuated canisters should undergo two separate tests. First check the canister with a vacuum gauge to determine negative pressure, and then test the attached critical flow orifice that is used to control flow during the prescribed sampling interval with a rotometer. Test canisters used with a positive displacement sampling pump (i.e., canister at atmospheric pressure at the start of the sampling period and then pressurized under constant flow pump conditions to approximately two atmospheres) for pressure conditions with a pressure gauge. Additionally, determine the sample pump flow rate with either a film calibrator or a flowmeter kit and stopwatch.

Attach a vacuum/pressure gauge known to be free of contaminants to the fitting upstream of the canister's main valve. Then open the main valve and read the gauge to confirm that the evacuated canister has maintained the same vacuum reading, within ±5 percent, reported by the laboratory or provider of the canister. The laboratory uses the same method to confirm the fill of a pressurized canister. The canister should be within ±5 percent of the pressure valve reported by the field crew. Flag canisters outside the gauge error margin as suspect and qualify their data accordingly.

5.2 PERIODIC QA/QC CHECKS

Implement periodic field QA/QC checks to supplement the more frequent routine QC checks required by the project. These periodic checks will serve to determine compliance with siting and operating criteria and should be made after the specific CTO air monitoring plan is in full operation. Include air matrix spiked samples, instrument performance audits of the air monitoring and meteorological equipment, and system audits in the periodic QA/QC checks.

Routinely check the accuracy of sample analysis by submitting spiked and blank gas samples as part of the laboratory analysis package. Spiked samples should contain a known concentration of some of the same compounds for which the laboratory is performing analysis. Blank samples are collection media that have no measurable amounts of the substance(s) of interest. The analysis of spiked and blank samples should be reported along with the normal samples collected during the project.

Qualified air quality technicians who are not directly involved in the routine operation of the air monitoring activity are to conduct instrument performance audits on air sampling and meteorological measurement equipment. In addition, the auditing equipment used to conduct the tests must be independent and different from that used to calibrate or maintain the air monitoring instrumentation. The audited instruments are challenged with known input values (e.g., air flow rates, electrical signals, timing mechanisms, temperature environments) and the instrument's observed response to the known inputs is reported.

The system audit provides an onsite qualitative evaluation of the installation of air sampler array and the meteorological monitoring station. The system audit documents the following:

- General physical condition and operability of the air sampling equipment
- Operational QC procedures in use (calibrations, single-point checks, instrument operation check lists, documentation)
- Instrument siting and exposure criteria
- Data acquisition, validation, and reporting procedures

The frequency of periodic QA/QC checks depends on the duration of the project. Where a long-term (i.e., 6 to 12 months or more) project is in effect, perform the periodic QA/QC checks quarterly. For short-term projects lasting only a few weeks or less, an initial QA/QC check at the beginning followed by a final check at the project's end is sufficient.

Document problems or discrepancies discovered during performance and system audits in a report, and discuss them with the respective CTO Manager who will initiate the required corrective actions.

5.3 LABORATORY QA/QC PROGRAM

Laboratory analytical techniques must properly identify the sample components and accurately and precisely measure concentrations. This requires the pre-concentration and/or storage of air samples. Therefore, methods chosen for time-integrated monitoring usually involve a longer analytical time period, more sophisticated equipment, and more rigorous QA procedures. Canister sampling includes replicate analyses and duplicate canisters to assess analytical and sampling precision. Analysis of field duplicate samples with laboratories is desirable to check field precision and laboratory analytical performance.

Laboratory QC methods must include the requirements noted in the *Department of Defense Quality Systems Manual for Environmental Laboratories* (DoD 2013. For air monitoring projects, these requirements should address the following elements (as required by the test method): laboratory control samples, duplicates, blanks, surrogates, other laboratory QC samples, field QC samples, internal standards, calibration standards, and canister cleanup and certification. Inter-laboratory analysis of duplicate or co-located samples is desirable to check laboratory analytical performance.

6. Records

Records of QC samples analyzed during ER Program CTO activities will be maintained on laboratory bench sheets, raw data sheets, in the laboratory computerized data system, and on QC summary forms, as requested. Analytical laboratories maintain records in accordance with their quality assurance manual (QAM), as part of performing environmental analytical work under DoD.

Records shall be maintained in accordance with the analytical laboratory subcontract agreement specifications or the laboratory-specific QAM, whichever is more stringent. Document field QA/QC Samples (Air) as prescribed in the respective Air Monitoring Plan or WP.

7. Health and Safety

Field personnel shall perform work in accordance with the current (or as contractually obligated) U.S. Army Corps of Engineers Safety and Health Requirements Manual EM-385-1-1 (USACE 2008) and site-specific health and safety plan (HSP).

8. References

Department of Defense, United States (DoD). 2005a. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

———. 2005b. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2B: Quality Assurance/quality Control Compendium: Minimum QA/QC Activities*. Final Version 1. DoD: DTIC ADA 426957, EPA-505-B-04-900B. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/swerffrr/pdf/-qaqc_v1_0305.pdf.

———. 2012. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2A: Optimized UFP-QAPP Worksheets*. Revision 1. March.

———. 2013. *Department of Defense Quality Systems Manual for Environmental Laboratories.* Version 5.0. Draft Final. Prepared by DoD Environmental Data Quality Workgroup and Department of Energy Consolidated Audit Program Operations Team. July.

Department of the Navy (DON). 2014. *Environmental Readiness Program Manual*. OPNAV Instruction 5090.1D. 10 January.

United States Army Corps of Engineers (USACE). 2008. *Consolidated Safety and Health Requirements Manual.* EM-385-1-1. Includes Changes 1–7. 13 July 2012.

9. Attachments

None.

Level C and Level D Data Validation for GC/FID/ECD Volatile Organics and Fixed Gases in Soil Gas/Vapor by EPA Method TO-03 and ASTM D1946

1. Purpose

This data validation procedure sets forth the standard operating procedure for performance of Level C and Level D data validation of volatile organic and fixed gases data obtained under the United States (U.S.) Navy Environmental Restoration (ER) Program for Naval Facilities Engineering Command (NAVFAC), Pacific and is consistent with protocol in the *Department of Defense Quality Systems Manual (QSM) for Environmental Laboratories* (DoD QSM) (DoD 2013). Level B validation is addressed separately in Procedure II-A, *Data Validation*.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the most recent version of the Uniform Federal Policy-Quality Assurance Project Plan (UFP QAPP) Part 1 (DoD 2005a), 2A (DoD 2012), and 2B (2005b), as well as the DoD Quality Systems Manual (DoD 2013). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Responsibilities

The CTO Manager, the QA Manager or Technical Director, and the CTO QA Coordinator are responsible for ensuring that this procedure is implemented by data validation personnel.

Data validation personnel are responsible for implementing this procedure for validation of all gas chromatography with flame ionization detection or electron capture detection (GC/FID/ECD) volatile and fixed gases data.

4. Procedure

This procedure addresses the validation of volatile organic and fixed gases data obtained using U.S. Environmental Protection Agency (EPA) Method TO-03 (EPA 1999) and American Society for Testing and Materials (ASTM) D1946 (ASTM 2011). The quality control (QC) criteria identified in this procedure are those specified in the analytical method and the DoD QSM (DoD 2013). Where project specific criteria are identified in the CTO work plan, they will supersede the QC criteria identified in this procedure.

- Form I: Sample Results Summary Form

- Form II: Surrogate Recovery Summary Form
- Form III: Matrix Spike/Matrix Spike Duplicate or Blank Spike/Blank Spike Duplicate Recovery Summary Form
- Form IV: Method Blank Summary Form
- Form VI: Initial Calibration Summary Form
- Form VII: Continuing Calibration Summary Form
- Form VIII: Analytical Sequence Form

Level C data validation consists of review of summary forms only while Level D data validation requires review of both summary forms and all associated raw data. Data review guidelines and how they apply to the different validation levels are indicated in the following text.

4.1 SAMPLE MANAGEMENT

QA/QC criteria included under sample management are sample preservation, handling, and transport; chain of custody (COC); and holding times.

4.1.1 Sample Preservation, Handling, and Transport

Level C and Level D:

1. Evaluate sample collection, handling, transport, and laboratory receipt from chain of custody and laboratory receipt checklists to ensure that the samples have been properly preserved and handled.
2. The chain-of custody, laboratory traffic reports, and sample preparation logs will be reviewed to verify that tedlar bags and sorbent tubes were properly filled and canisters were properly pressurized and handled. Improper pressurization or analysis of an inappropriately pressurized sample by the laboratory may require that all results be reported as estimated (J) or unusable (R).

4.1.2 Chain of Custody

Level C and Level D:

Examine the COC form for legibility and check that all volatile and fixed gas analyses requested on the COC have been performed by the laboratory. Ensure that the COC Sample Number on the laboratory sample results form (Form I [or equivalent]) matches the Sample Identification on the COC. Read the laboratory case narrative for additional information.

1. Any samples received for analysis that were not analyzed shall be noted in the data validation report, along with the reason(s) for failure to analyze the samples, if the reason(s) can be determined. Conversely, samples that were analyzed for volatiles and fixed gases but were not requested should also be noted.
2. Any discrepancies in sample naming between the COC and Form I (or equivalent) form shall be noted in the data validation report with the correct sample name being identified if the correct sample name can be determined.

3. If the receiving laboratory transferred the samples to another laboratory for analysis, both the original COCs and transfer COCs shall be present. Document in the data validation report if the transfer COCs are not present.
4. Internal COC is required for all samples, extracts, and digestates from receipt to disposal. Verify the internal COC forms for completeness. Document in the data validation report if the internal COC forms are not present.

4.1.3 Holding Times

Level C and Level D:

Holding times for volatile organics and fixed gases are measured from the time of collection (as shown on the COC) to the time of sample analysis (as shown on the sample results form and instrument performance check summary form [Forms I and V (or equivalent)]). If canisters and sorbent tubes were used to collect the samples, all samples must be analyzed within 30 days of sample collection. If tedlar bags were used to collect the samples, all samples must be analyzed within 72 hours of sample collection.

1. If holding times are exceeded, flag positive results as estimated "J" and limits of detection (LODs) (nondetects) as estimated "UJ," and document that holding times were exceeded.
2. If holding times are exceeded by more than a factor of 2 (e.g., air sample in a canister has a holding time of more than 60 days), detects will be qualified as estimated "J" and nondetects as unusable "R."

4.2 INSTRUMENT PERFORMANCE

The objective is to ensure that the instrument condition is adequate for proper identification and quantification of the compounds of interest. The chromatographic resolution and the sensitivity should be evaluated from the chromatograms.

Level C:

Instrument performance is not evaluated for Level C validation.

Level D:

Evaluate blank, standard, sample, and QC chromatograms to ensure that the chromatographic resolution and the sensitivity are adequate. Any shift in baseline, negative peaks, or peak tailing/splitting shall be discussed in the data validation report. If the data quality has been affected by poor instrument performance, the data should be qualified using the reviewer's professional judgment.

4.3 CALIBRATION

Compliance requirements for satisfactory instrument calibration are established to ensure that an instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that an instrument is capable of acceptable performance at the beginning of a sequence, and continuing calibration checks document satisfactory maintenance and adjustment of the instrument on a day-to-day basis.

4.3.1 Initial Calibration

The GC system can be calibrated using the external standard technique or internal standard technique. Because of the difficulty in selecting suitable internal standards, the external standard technique will often be the method of choice.

At the beginning of the analysis sequence, calibration standards must be run at three concentration levels for each parameter of interest to establish the calibration curve and expected retention time windows for the compounds of interest. One of the standards should be at a concentration at or just above the limit of quantitation (LOQ), and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

Level C and Level D:

For the initial calibration (at least three-points), the relative standard deviation (RSD) of the calibration factor (CF) for each target compound must be less than or equal to 20 percent. Verify the RSDs from the initial calibration summary forms. Alternatively, a linear curve may be used with a coefficient of determination (r^2); r^2 equal to or greater than 0.990. A second order calibration curve may also be used after evaluating the laboratory's acceptance criteria. If the initial calibration criteria are not met, flag all associated quantitative results as estimated "J" for detects and estimated "UJ" for nondetects.

Level D:

Verify the percent RSDs (%RSDs), r^2, or laboratory established measure of linearity for the initial calibration from the raw data. Verify the CF for each target compound from the raw data on the low-point calibration standard and one additional calibration standard. If errors are discovered, request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.3.2 Initial Calibration Verification

The initial calibration curve must be verified with a standard that has been purchased or prepared from an independent source each time initial calibration is performed. A standard from the same manufacturer but independently prepared from different source materials may also be used as an independent source. This initial calibration verification (ICV) must contain all of the method target compounds.

Level C and Level D:

1. Verify the ICV was analyzed following the initial calibration and contained all method target compounds.
2. If any target analyte has a percent difference (%D) greater than 20 percent, flag detects for the affected compounds as estimated "J" and nondetects as estimated "UJ" in all samples associated with the initial calibration.

Level D:

Verify from the raw data that there were no calculation or transcription errors by recalculating a percentage of the ICV calculations.

4.3.3 Continuing Calibration

The working calibration curve or CF must be verified by the injection of a continuing calibration standard. A continuing calibration standard must also be analyzed after every 10 samples and at the end of the analysis sequence to ensure that system performance has not degraded. The initial calibration standard chosen for the continuing calibration standard shall be the mid-level standard or the standard with a contaminant concentration level that is potentially the most representative of contaminant concentrations in the next 10 samples.

Level C and Level D:

Verify the %Ds from the continuing calibration summary forms. The %D between the CF from the continuing calibration and the average CF from the initial calibration must be less than 20 percent. Alternatively, if a linear, (first-order) calibration curve is utilized in the initial calibration, the %D of the calculated amount and the true amount for each compound must be less than or equal to 20 percent. If the continuing calibration criteria are not met for both columns, qualify all associated results as estimated "J" for detects and estimated "UJ" for nondetects.

Level D:

Verify the %Ds from the raw data.

4.4 BLANKS

Method blank analytical results are assessed to determine the existence and magnitude of contamination problems. If problems with any method blank exist, all associated data must be carefully evaluated to determine whether there is any bias on the data, or if the problem is an isolated occurrence not affecting other data. No contaminants should be present in the method blank(s). The method blank should be analyzed on each GC system used to analyze site samples.

1. The reviewer should identify samples associated with each method blank using Form IV (or equivalent). Verify that method blank analysis has been reported per matrix and concentration level for each set of samples. Each sample must have an associated method blank. Qualify positive results in samples with no method blank as unusable "R." Nondetects do not require qualification.
2. If the method blank was not analyzed on a GC used to analyze site samples, note the deficiency in the data validation report. Professional judgment shall be used for subsequent qualification of the data.
3. Compare the results of each method blank with the associated sample results. The reviewer should note that the blank analyses may not involve the same volumes or dilution factors as the associated samples. These factors must be taken into consideration when applying the 5× criteria discussed below, such that a comparison of the total amount of contamination is actually made.
4. If a compound is found in the blank, but not in the associated sample, no action is taken.
5. Any compound, detected in both the sample and the associated blank shall be qualified when the sample concentration is less than the LOQ and the blank concentration is less than, greater than, or equal to the LOQ. Care should be taken to factor in the percent moisture

when comparing detects in the sample and the method blank. The applicable review qualifier(s) are summarized in Table II-W-1.

Table II-W-1: Blank Qualifications

Sample Result	Sample Value	Reviewer Qualifier(s)
Less than LOQ and blank result is <, > or = LOQ	Leave as reported	U
≥LOQ, blank result is <LOQ	Leave as reported	None
≥LOQ, blank result is >LOQ and sample result <blank result	Leave as reported	Use professional judgment
≥LOQ, blank result is >LOQ and sample result ≥blank result	Leave as reported	Use professional judgment
≥LOQ and blank result is = LOQ	Leave as reported	Use professional judgment

6. In the case wherein both the sample concentration and the blank concentration are greater than or equal to the LOQ, previously approved criteria as identified in the project planning documents may be applied to qualify associated sample results. Otherwise, qualify sample results as non-detect "U" when the sample concentration is less than or equal to 5 times the blank concentration (5× rule).
7. Instances of contamination can be attributable to the dilution process. These occurrences are difficult to determine; however, the reviewers should qualify the sample data as nondetects, "U," when the reviewer determines the contamination to be from a source other than the sample.
8. In the event of gross contamination (i.e., saturated peaks) in the blanks, the associated samples must be evaluated for gross contamination. If gross contamination exists in the samples, the affected compounds should be qualified as unusable, "R."

Level D:

1. Verify from the preparation log that the information recorded on Form IV (or equivalent) is correct.
2. Review the results of all blank raw data and Form I (or equivalent) to ensure that there were no false negatives or false positives.
3. Verify all target compound detects found in the method blanks against the raw data. Follow the guidelines specified in Sections 4.9 and 4.10 of this procedure. After the validity of the target compounds are verified, validate the corresponding data using the criteria outlined above for Level C and Level D validation.

4.5 BLANK SPIKES AND LABORATORY CONTROL SAMPLES

Blank spikes or laboratory control samples (LCSs) are not required by Method TO-03 and ASTM D1946. However, if the laboratory analyzes blank spikes or LCSs, these procedures shall be followed:

Level C and Level D:

1. Blank spike/LCS recoveries must be within project-specific control limits. Use in-house limits if there are no project-specific limits.
2. If the blank spike/LCS results are 0 percent, only the spiked compounds that showed low recovery in all associated samples shall be flagged as unusable "R" for nondetects and estimated "J" for detects.
3. If blank spike/LCS results are below the control limits (but above 0 percent), spiked compounds which showed low recovery in all associated samples shall be flagged as estimated "UJ" or "J."
4. If blank spike/LCS results are above the control limits, detects for only the spiked compounds which showed high recovery in all associated samples shall be flagged as estimated "J."
5. If the laboratory analyzes a blank spike duplicate/LCS duplicate (LCSD), evaluate and qualify the LCSD results using the criteria noted above.
6. If the relative percent differences (RPDs) between LCS and LCSD results are above the control limits (use the matrix spike [MS]/matrix spike duplicate [MSD] RPD control limits identified in DoD QSM Appendix B [DoD 2013], if none are available use laboratory in-house limits), spiked compounds which showed high RPD in all associated samples shall be flagged as estimated "UJ" or "J."

Level D:

To verify that the spike percent recovery was calculated and reported correctly using the following equation, recalculate one spike recovery per matrix (and any spike that would result in the qualification of a sample).

$$\%\text{Recovery} = \frac{Q_d}{Q_a} \times 100$$

Where:

Q_d = Quantity determined by analysis

Q_a = Quantity added to samples/blanks

If transcription errors are discovered on Form III (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.6 SURROGATE RECOVERY

Surrogates are not required by Method TO-03 and ASTM D1946. However, if the laboratory spiked samples with surrogate compounds, these procedures shall be followed:

Level C and Level D:

Sample and blank surrogate recoveries for herbicides must be within the QC limits specified in the DoD QSM Appendix C (DoD 2013) unless project-specific control limits are established. Use in-house limits if surrogates are not listed in Appendix C or project limits are not specified. Verify that no samples or blanks have surrogates outside the criteria from Form II (or equivalent).

1. Sample and blank surrogate recoveries must be within project-specific control limits. Use in-house limits if there are no project-specific limits. Verify that no samples or blanks have surrogates outside the criteria from Form II (or equivalent).
2. If any surrogate recovery is below the QC limits for either one of the surrogates, but above or equal to 10 percent, flag associated positive results as estimated "J" and nondetects as "UJ."
3. If any surrogate recovery is less than 10 percent, flag all nondetects as unusable "R" and detects as estimated "J." No qualification is done if surrogates are diluted beyond detection but note in the data validation report that surrogate evaluation could not be performed due to the high dilution factor.
4. If any surrogate recovery is above the upper QC limit, flag associated positive results as estimated "J." No qualification of nondetects is necessary in the case of high recoveries.
5. Surrogates may be reported as "diluted out" (D); if dilution is such that the surrogate can no longer be detected. If this is the case, note in the data validation report that surrogate evaluation could not be performed due to a high dilution factor. A full evaluation of the sample chromatogram may be necessary to determine that surrogates are truly "diluted out."

Level D:

The reported surrogate recoveries on Form II should be verified from the raw data for a representative number of samples.

4.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATE AND MATRIX DUPLICATE

MS/MSDs are not required by Method TO-03 and ASTM D1946.

Matrix duplicate (MD) data are used to determine the effect of the matrix on a method's recovery efficiency and precision for a specific sample matrix. MD analyses are also performed to demonstrate acceptable method precision by the laboratory at the time of analysis.

Level C and Level D:

The laboratory must spike and analyze a MS/MSD from the specific project site as required for each matrix type and analytical batch.

1. MD data should be reported on a summary form similar to Form III (or equivalent).
2. Compare the RPD for each spiked compound with project-specific control limits. Use in-house limits if there are no project-specific limits.

3. If the sample results are greater than 5× the LOQ and the RPDs between sample and duplicate results are greater than the control limits, detects for only the spiked compounds which showed high RPD in the parent sample shall be flagged as estimated "J."

Level D:

Check the raw data and recalculate one or more RPDs, especially RPDs that resulted in the qualification of data, using the following equations to verify that results on Form III (or equivalent) are correct.

$$RPD = \frac{ABS|SR - DR|}{(SR + DR)/2} \times 100$$

Where:

SR	=	sample result
DR	=	duplicate result
ABS	=	absolute value

If transcription errors are discovered on Form III (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.8 FIELD QC SAMPLES

Field QC samples discussed in this section of the procedures are ambient blanks, field blanks, and field duplicates.

4.8.1 Ambient Blanks and Field Blanks

An ambient blank is collected in the same type of container used for an environmental sample, kept with the sample containers before sample collection and opened at the site and exposed to the ambient conditions. Ambient levels of site contaminants are determined by the analysis of ambient blanks.

A field blank is a sample collected in the field from a certified air source. Compounds detected in field blanks indicate the possibility of cross-contamination between samples due to improper equipment decontamination.

If target compounds are detected in the ambient blanks and/or field blanks, the procedure for the qualification of associated sample results is identical to the criteria outlined in Section 4.4 of this procedure.

Level C and Level D:

1. Determine which field QC samples apply to samples in the sample delivery group.
2. Because of the way in which the field blanks and equipment blanks are sampled, equipment blanks are not qualified because of field blank contamination. The affected samples are qualified, however, by either the field blank or equipment blank results, whichever has the higher contaminant concentration.

3. Ambient blanks and field blanks are only qualified with method blank results in order to account for laboratory contamination.

Level D:

Compound identification and quantification of field blank and equipment blank samples must be verified. Follow the guidelines specified in Sections 4.9 and 4.10 of this procedure.

4.8.2 Field Duplicates

Field duplicates are samples collected in the field simultaneously. Field duplicates should be collected in separate sample containers at the same location and depth. Field duplicate results are an indication of both field and laboratory precision; the results may be used to evaluate the consistency of sampling practices.

Level C and Level D:

1. Check to ensure that field duplicates were collected and analyzed as specified in the project planning documents. If the sampling frequency is less than the frequency stated in the planning documents, no qualification of the associated sample results is necessary but the incident shall be discussed in the data validation report.
2. For field duplicate results, if the RPDs are greater than 100 percent or as stated in the planning document if more conservative, no qualification of the associated sample results is necessary, but the differences should be noted in the data validation summary.

Level D:

Before comparison of duplicates, the compound identification and quantification must be verified. Follow the guidelines specified in Sections 4.9 and 4.10 of this procedure.

4.9 TARGET COMPOUND IDENTIFICATION

Qualitative criteria for compound identification have been established to minimize the number of erroneous identifications of compounds. An erroneous identification can be either a false positive (reporting a compound present when it is not) or a false negative (not reporting a compound that is present).

The laboratory must report retention (RT) time window data for each compound on each column used to analyze the samples. The RT windows are used for qualitative identification. RTs of reported compounds must fall within the calculated window for both chromatographic columns. Second column confirmation must be performed for all GC work. Sample chromatograms for both columns must be provided.

Level C:

Target compound identification is not evaluated for Level C validation since it requires the interpretation of raw data.

Level D:

1. Verify from the raw data that the RT of the detected compound and the RT windows are correct.
2. Evaluate all sample chromatograms to ensure that there were no peaks present which were not reported (false negatives) or the reported detects did not meet identification criteria (false positives). Presence of a large interfering peak may result in false positives or false negatives. The reviewer should use professional judgment in evaluating the effect of interference.

4.10 COMPOUND QUANTITATION AND REPORTING LIMITS

The objective is to ensure that the reported quantitation results and reporting limits (i.e., LOQ, LOD, detection limit [DL]) are accurate. All soil sample results are reported on a dry weight basis.

Level C:

Specific compound quantitation is not verified for Level C validation.

Level C and Level D:

1. Verify that the RLs for nondetects are equal to the LODs. Verify that an annual DL study was performed or quarterly LOD/LOQ verification checks were performed in accordance with the DoD QSM (DoD 2013). The LOD/LOQ verification check must be evaluated to determine whether the laboratory can reliably detect and identify all target analytes at a spike concentration of approximately two times but not more than four times the current reported DL. Qualify nondetects as unusable "R."
2. Check that reported nondetects and positive values have been adjusted to reflect sample dilutions. When a sample is analyzed at more than one dilution, the lowest LODs are used unless a QC criterion has been exceeded. In this case, the higher LODs from the diluted analysis are used. The least technically sound data will be flagged "R" with a qualification code "D."
3. Verify that no results exceed the highest calibration standard without being diluted. If a result has exceeded the highest calibration standard, verify that a dilution was performed. If not, qualify the detected compound that required dilution as "J" and document the event in the data validation report.

Level D:

Compound quantification should be verified by recalculation from the raw data for a representative number of samples.

5. Records

A Form I that has been validated and verified, and has been determined by the data validator to accurately represent the appropriate sample results to be utilized, shall be stamped "NAVFAC PACIFIC VALIDATED." Additionally, sample result forms for which the data has been validated at the Level D validation level shall be stamped or noted "Level D."

Copies of all documents generated by the data validation personnel will be stored for no less than 10 years. The original validated laboratory data shall be archived to the Federal Records Center at project completion.

6. References

ASTM International (ASTM). 2011. *Standard Practice for Analysis of Reformed Gas by Gas Chromatography*. D1946-90(2011). West Conshohocken, PA.

Department of Defense, United States (DoD). 2005a. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

———. 2005b. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2B: Quality Assurance/quality Control Compendium: Minimum QA/QC Activities*. Final Version 1. DoD: DTIC ADA 426957, EPA-505-B-04-900B. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www2.epa.gov/sites/production/files/documents/qaqc_v1_0305.pdf.

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———. 2013. *Department of Defense Quality Systems Manual for Environmental Laboratories.* Version 5.0. Draft Final. Prepared by DoD Environmental Data Quality Workgroup and Department of Energy Consolidated Audit Program Operations Team. July.

Environmental Protection Agency, United States (EPA). 1999. *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*. 2nd ed. EPA-625/R-96-010b. Center for Environmental Research Information. January.

———. 2007. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846.* 3rd ed., Revision 6. Office of Solid Waste. November. On-line updates at: http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm.

Procedure II-A, *Data Validation*.

7. Attachments

None.

Level C and Level D Data Validation for GC/MS Volatile Organics and Fixed Gases in Soil Gas/Vapor by EPA Methods TO-14, TO-15, and TO-17

1. Purpose

This data validation procedure sets forth the standard operating procedure for performance of Level C and Level D data validation of volatile organic and fixed gases data obtained under the United States (U.S.) Navy Environmental Restoration (ER) Program for Naval Facilities Engineering Command (NAVFAC), Pacific and is consistent with protocol in the *Department of Defense Quality Systems Manual (QSM) for Environmental Laboratories* (DoD QSM) (DoD 2013). Cursory validation is addressed separately in Procedure II-A, *Data Validation*.

2. Scope

This procedure applies to all Navy ER projects performed in the NAVFAC Pacific Area of Responsibility.

This procedure shall serve as management-approved professional guidance for the ER Program and is consistent with protocol in the most recent version of the Uniform Federal Policy-Quality Assurance Project Plan (UFP QAPP) Part 1 (DoD 2005a), 2A (DoD 2012), and 2B (2005b), as well as the DoD Quality Systems Manual (DoD 2013). As professional guidance for specific activities, this procedure is not intended to obviate the need for professional judgment during unforeseen circumstances. Deviations from this procedure while planning or executing planned activities must be approved and documented by the following prime contractor representatives: the CTO Manager and the Quality Assurance (QA) Manager or Technical Director. A Navy project representative (i.e., Remedial Project Manager or QA Manager) shall also concur with any deviations.

3. Responsibilities

The CTO Manager, the QA Manager or Technical Director, and the CTO QA Coordinator are responsible for ensuring that this procedure is implemented by data validation personnel.

Data validation personnel are responsible for implementing this procedure for validation of all gas chromatography/mass spectrometry (GC/MS) volatile and fixed gases data.

4. Procedure

This procedure addresses the validation of volatile organic and fixed gases data obtained using U.S. Environmental Protection Agency (EPA) Methods TO-15, TO-16, and TO-17 (EPA 1999). The quality control (QC) criteria identified in this procedure are those specified in the analytical method and the DoD QSM (DoD 2013). Where project specific criteria are identified in the CTO work plan, they will supersede the QC criteria identified in this procedure.

- Form I: Sample Results Summary Form
- Form II: Surrogate Recovery Summary Form

- Form III: Matrix Spike/Matrix Spike Duplicate or Blank Spike/Blank Spike Duplicate Recovery Summary Form
- Form IV: Method Blank Summary Form
- Form V: Instrument Performance Check Summary Form
- Form VI: Initial Calibration Summary Form
- Form VII: Continuing Calibration Summary Form
- Form VIII: Internal Standard Summary Form

Level C data validation consists of review of summary forms only, whereas Level D data validation requires review of both summary forms and all associated raw data. Data review guidelines and how they apply to the different validation levels are indicated in the following text.

4.1 SAMPLE MANAGEMENT

QA/QC criteria included under sample management are sample preservation, handling, and transport, chain of custody (COC), and holding times.

4.1.1 Sample Preservation, Handling, and Transport

Level C and Level D:

Evaluate sample collection, handling, transport, and laboratory receipt from chain of custody and laboratory receipt checklists to ensure that the samples have been properly preserved and handled.

1. The COC, laboratory traffic reports, and sample preparation logs will be reviewed to verify that tedlar bags and sorbent tubes were properly filled and canisters were properly pressurized and handled. Improper pressurization or analysis of an inappropriately pressurized sample by the laboratory may require that all results be reported as estimated (J) or unusable (R).
2. Sorbent tubes should be properly stored at <4 degrees Celsius in the field prior to use and in the laboratory prior to analysis. Document in the data validation report if storage temperature was not met.

4.1.2 Chain of Custody

Level C and Level D:

Examine the COC form for legibility and check that all volatile and fixed gas analyses requested on the COC have been performed by the laboratory. Ensure that the COC Sample Number on the laboratory sample results form (Form I [or equivalent]) matches the Sample Identification on the COC. Read the laboratory case narrative for additional information.

1. Any samples received for analysis that were not analyzed shall be noted in the data validation report, along with the reason(s) for failure to analyze the samples, if the reason(s) can be determined. Conversely, samples that were analyzed for volatiles and fixed gases but were not requested should also be noted.

2. Any discrepancies in sample naming between the COC and sample results form shall be noted in the data validation report with the correct sample name being identified if the correct sample name can be determined.
3. If the receiving laboratory transferred the samples to another laboratory for analysis, both the original COCs and transfer COCs shall be present. Document in the data validation report if the transfer COCs are not present.
4. Internal COC is required for all samples, extracts, and digestates from receipt to disposal. Verify the internal COC forms for completeness. Document in the data validation report if the internal COC forms are not present.

4.1.3 Holding Times

Level C and Level D:

Holding times for volatile organics and fixed gases are measured from the time of collection (as shown on the COC) to the time of sample analysis (as shown on the sample results form and instrument performance check summary form [Forms I and V (or equivalent)]). If canisters and sorbent tubes were used to collect the samples, all samples must be analyzed within 30 days of sample collection. If tedlar bags were used to collect the samples, all samples must be analyzed within 72 hours of sample collection.

1. If the holding time is exceeded, flag all associated positive results as estimated "J" and all associated limits of detection (LODs) (nondetects) as estimated "UJ," and document that holding times were exceeded.
2. If holding times are grossly exceeded by greater than a factor of 2.0 (e.g., air sample in a canister has a holding time of more than 60 days), detects will be qualified as estimated "J" and nondetects as unusable "R."

4.2 GC/MS INSTRUMENT PERFORMANCE CHECK

Level C and Level D:

GC/MS instrument performance checks (formerly referred to as tuning) are performed to ensure mass resolution, identification, and to some degree, sensitivity. These criteria are not sample specific. Conformance is determined using standard reference materials; therefore, these criteria should be met in all circumstances.

The analysis of the instrument performance check solution must be performed at the beginning of each 12-hour (24-hour for TO-15 and TO-17) period during which samples or standards are analyzed. The instrument performance check, bromofluorobenzene (BFB) for volatile analysis, must meet the ion abundance criteria given in Table II-X-1 and Table II-X-2.

Table II-X-1: Ion Abundance Criteria – BFB (TO-14)

m/z	Ion Abundance Criteria
50	15.0–40.0% of m/z 95
75	30.0–60.0% of m/z 95
95	Base peak, 100% relative abundance
96	5.0–9.0% of m/z 95

m/z	Ion Abundance Criteria
173	Less than 2.0% of m/z 174
174	Greater than 50.0% of m/z 95
175	5.0–9.0% of m/z 174
176	Greater than 95.0% but less than 101.0% of m/z 174
177	5.0–9.0% of m/z 176

% percent
m/z mass-to-charge ratio

Table II-X-2: Ion Abundance Criteria – BFB (TO-15 and TO-17)

m/z	Ion Abundance Criteria
50	8.0–40.0% of m/z 95
75	30.0–66.0% of m/z 95
95	Base peak, 100% relative abundance
96	5.0–9.0% of m/z 95
173	Less than 2.0% of m/z 174
174	50.0–120% of m/z 95
175	4.0–9.0% of m/z 174
176	93.0–101% of m/z 174
177	5.0–9.0% of m/z 176

Check that all sample runs are associated with an injection. Make certain that a BFB performance check is present for each 12-hour or 24-hour period samples are analyzed (Form V [or equivalent]). Verify that all samples were analyzed within 12 hours or 24 hours of BFB injection.

If ion abundance criteria are not met, professional judgment may be applied to determine to what extent the data may be utilized. The most important factors to consider are the empirical results that are relatively insensitive to location on the chromatographic profile and type of instrumentation; therefore, the critical ion abundance criteria for BFB are the mass-to-charge ratio (m/z) 95/96, 174/175, 174/176, and 176/177 ratios. The relative abundance of m/z 50 and 75 are of lesser importance. Use professional judgment when samples are analyzed beyond the 12-hour or 24-hour time limit.

Decisions to use analytical data associated with BFB instrument performance checks not meeting requirements should be noted in the data validation report.

Level D:

Verify by recalculating from the quantitation reports, mass spectra, and chromatograms that the mass assignment is correct and that the mass listing is normalized to the specified m/z. If transcription errors are discovered on the Form V (or equivalent), request a resubmittal from the laboratory. Validate the data using the criteria outlined above.

4.3 CALIBRATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data for compounds on the target compound list.

4.3.1 Initial Calibration

Initial calibration demonstrates that the instrument is capable of acceptable performance in the beginning of the analytical run and of producing an acceptable calibration curve.

Level C and Level D:

1. Evaluate the average relative response factors (RRFs) for all target compounds by checking Form VI (or equivalent).
2. Check Form VI (or equivalent) and evaluate the percent relative standard deviation (%RSD) for all target compounds. If any target compound has a %RSD of greater than 30 percent, flag detects for the affected compounds as "J" and nondetects as "UJ" in the associated samples that correspond to that initial calibration.

Level D:

1. Verify the files reported on Form VI (or equivalent) against the quantitation reports, mass spectra, and chromatograms. If the files do not match, the RRFs reported are likely to be from another initial calibration and will have to be changed. Request a resubmittal from the laboratory.
2. Recalculate the average RRFs and %RSDs reported on Form VI (or equivalent) for one compound per internal standard, (preferably compounds which were identified in the samples) on the low-point calibration standard and one additional calibration standard. If errors are discovered, request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.3.2 Initial Calibration Verification

The initial calibration curve must be verified with a standard that has been purchased or prepared from an independent source each time initial calibration is performed. A standard from the same manufacturer but independently prepared from different source materials may also be used as an independent source. This initial calibration verification (ICV) must contain all of the method target compounds.

Level C and Level D:

1. Verify the ICV was analyzed following the initial calibration and contained all method target compounds.
2. If any target compound has a percent difference (%D) greater than 30 percent, flag detects for the affected compounds as estimated "J" and nondetects as estimated "UJ" in all samples associated with the initial calibration.

Level D:

Verify from the raw data that there were no calculation or transcription errors by recalculating a percentage of the ICV calculations.

4.3.3 Continuing Calibration

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Continuing calibration establishes the 12-hour or 24-hour relative response factors on which the quantitations are based and checks satisfactory performance of the instrument on a day-to-day basis.

Level C and Level D:

1. Continuing calibration standards containing both target compounds and system monitoring compounds must be analyzed every 12 hours or 24 hours during operation. Evaluate the continuing RRFs on Form VII (or equivalent).
2. Ensure that the average RRFs reported on Form VII (or equivalent) correspond to the average RRFs reported on Form VI (or equivalent) for the corresponding initial calibration.
3. If any target compound has a %D between the initial calibration average RRF and continuing calibration RRFs outside 30 percent, flag all detects as "J" and all nondetects as "UJ" in all associated samples that correspond to that continuing calibration.

Level D:

1. Verify the file reported on Form VII (or equivalent) against the raw data for the continuing calibration. If the file does not match, the RRFs reported are likely to be from another continuing calibration and will have to be changed. Request a resubmittal from the laboratory.
2. Recalculate the reported RRFs and %Ds reported on Form VII (or equivalent) for one compound per internal standard. If errors are discovered, request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.4 BLANKS

4.4.1 Method Blanks

Method blank analytical results are assessed to determine the existence and magnitude of contamination problems. If problems with any method blank exist, all associated data must be carefully evaluated to determine whether there is any bias associated with the data, or if the problem is an isolated occurrence not affecting other data. Results may not be corrected by subtracting any blank values.

Level C and Level D:

1. The reviewer should identify samples associated with each method blank using Form IV (or equivalent). Verify that method blank analysis has been reported per matrix and concentration level for each 12-hour or 24-hour time period on each GC/MS system used to analyze volatile and fixed gas samples. Each sample must have an associated method blank. Qualify positive results in samples with no method blank as unusable "R." Nondetects do not require qualification.

2. Compare the results of each method blank with the associated sample results. The reviewer should note that the blank analyses may not involve the same volumes or dilution factors as the associated samples. These factors must be taken into consideration when applying the criteria discussed below, such that a comparison of the total amount of contamination is actually made.
3. If a compound is found in the blank, but not in the associated sample, no action is taken.
4. Any compound, other than those listed in Table II-X-3, detected in both the sample and the associated blank shall be qualified when the sample concentration is less than the limit of quantitation (LOQ) and the blank concentration is less than, greater than, or equal to the LOQ. Compounds listed in Table II-X-3 shall be qualified when the sample concentration is less than two times (2×) the LOQ and the blank concentration is less than, greater than, or equal to 2× LOQ. Care should be taken to factor in the percent moisture when comparing detects in the sample and the method blank. The applicable review qualifier(s) are summarized in Table II-X-4.

Table II-X-3: Common Laboratory Contaminants

1. Methylene chloride
2. Acetone
3. 2-Butanone

Table II-X-4: Blank Qualifications

Sample Result	Sample Value	Reviewer Qualifier(s)
Less than LOQ* and blank result is <, > or = LOQ*	Leave as reported	U
≥LOQ*, blank result is <LOQ*	Leave as reported	None
≥LOQ*, blank result is >LOQ* and sample result <blank result	Leave as reported	Use professional judgment
≥LOQ*, blank result is >LOQ* and sample result ≥blank result	Leave as reported	Use professional judgment
≥LOQ* and blank result is = LOQ*	Leave as reported	Use professional judgment

*2× LOQ for common laboratory contaminants

5. In the case wherein both the sample concentration and the blank concentration are greater than or equal to the LOQ, previously approved criteria as identified in the project planning documents may be applied to qualify associated sample results. Otherwise, qualify sample results as non-detect "U" when the sample concentration is less than or equal to 10 times the blank concentration (10× rule) for the compounds listed in Table II-X-3 and tentatively identified compounds (TICs). For all other compounds, qualify sample results as non-detect "U" when the sample concentration is less than or equal to 5 times the blank concentration (5× rule).
6. If gross contamination exists in the blanks (i.e., saturated peaks by GC/MS), all compounds affected shall be flagged as unusable "R" due to interference in all samples affected and this shall be noted in the data validation comments.
7. If target compounds other than common laboratory contaminants are found at low levels in the blank(s), it may be indicative of a problem at the laboratory and shall be noted in the data validation report.

8. Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary. Contamination introduced through dilution is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. It may be impossible to verify this source of contamination; however, if the reviewer determines that the contamination is from a source other than the sample, the data should be qualified. The sample value shall be reported as a nondetect and the reason shall be documented in the data validation report. Qualification of the data will be performed as given in Table II-X-4.

Level D:

1. Verify all target compound and TIC detects found in the method blanks against the raw data.
2. Verify that the target compound detects have valid spectra, as defined in Section 4.10 and the tentative identity of any TICs against the raw data, as defined in Section 4.12. If the spectra are not valid or the tentative identity is in error, request for a corrected Form I for the method blank from the laboratory.
3. Verify detected concentrations of target compounds and TICs from the raw data, as defined in Section 4.11. After the validity of the target compounds and TICs is verified, validate the corresponding data using the criteria outlined above.

4.4.2 Canister Blanks

All canisters must be clean and free of any contaminants before sample collection. Each sample must have an associated canister blank. Verify that canister blank analysis has been reported per canister. Canister blank analysis results are assessed to determine the existence and magnitude of contamination problems. The reviewer should refer to the COC to identify samples associated with each canister blank.

1. If target compounds are detected in the canister blanks, the procedure for the qualification of associated sample results is identical to the criteria outlined in Section 4.4.1 of this procedure.
4. The reviewer, using professional judgment, may qualify detected result in samples with no associated canister blank data as estimated (J).

4.5 BLANK SPIKES AND LABORATORY CONTROL SAMPLES

Blank spikes or laboratory control samples (LCSs) are not required by Methods TO-14, TO-15, and TO-17 (EPA 1999). However, if the laboratory analyzes blank spikes or LCSs, these procedures shall be followed:

Level C and Level D:

1. Blank spike/LCS recoveries must be within project-specific control limits. Use in-house limits if there are no project-specific limits.
2. If the blank spike/LCS results are 0 percent, only the spiked compounds that showed low recovery in all associated samples shall be flagged as unusable “R” for nondetects and estimated “J” for detects.

3. If blank spike/LCS results are below the control limits (but above 0 percent), spiked compounds which showed low recovery in all associated samples shall be flagged as estimated "UJ" or "J."
4. If blank spike/LCS results are above the control limits, detects for only the spiked compounds which showed high recovery in all associated samples shall be flagged as estimated "J."
5. If the laboratory analyzes a blank spike duplicate/LCS duplicate (LCSD), evaluate and qualify the LCSD results using the criteria noted above.
6. If the relative percent differences (RPDs) between LCS and LCSD results are above the control limits (use the matrix spike [MS]/matrix spike duplicate [MSD] RPD control limits identified in DoD QSM Appendix B [DoD 2013], if none are available use laboratory in-house limits), spiked compounds which showed high RPD in all associated samples shall be flagged as estimated "UJ" or "J."

Level D:

To check that the spike percent recovery was calculated and reported correctly using the following equation, recalculate one or more spike recoveries per matrix (and any spike that would result in the qualification of a sample).

$$\%\text{Recovery} = \frac{Q_D}{Q_A} \times 100$$

Where:

Q_D = Quantity determined by analysis

Q_A = Quantity added to samples/blanks

If transcription errors are discovered on Form III (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.6 SYSTEM MONITORING COMPOUNDS (SURROGATE SPIKES)

Surrogates are not required by Methods TO-14, TO-15, and TO-17 (EPA 1999). However, if the laboratory spiked samples with surrogate compounds, these procedures shall be followed:

Level C and Level D:

1. Sample and blank surrogate recoveries must be within project-specific control limits. Use in-house limits if there are no project-specific limits. Verify that no samples or blanks have surrogates outside the criteria from Form II (or equivalent).
2. If any surrogate recovery is below the QC limits for either one of the surrogates, but above or equal to 10 percent, flag associated positive results as estimated "J" and nondetects as "UJ."
3. If any surrogate recovery is less than 10 percent, flag all nondetects as unusable "R" and detects as estimated "J." No qualification is done if surrogates are diluted beyond detection

but note in the data validation report that surrogate evaluation could not be performed due to the high dilution factor.

4. If any surrogate recovery is above the upper QC limit, flag associated positive results as estimated "J." No qualification of nondetects is necessary in the case of high recoveries.
5. Surrogates may be reported as "diluted out" (D), if dilution is such that the surrogate can no longer be detected. If this is the case, note in the data validation report that surrogate evaluation could not be performed due to a high dilution factor. A full evaluation of the sample chromatogram and quantitation report may be necessary to determine that surrogates are truly "diluted out."

Level D:

To verify that the surrogate percent recovery was calculated and reported correctly using the following equation, recalculate all surrogate recoveries per matrix (and any surrogate that would result in the qualification of a sample).

$$\%\text{Recovery} = \frac{Q_D}{Q_A} \times 100$$

Where:

Q_D = Quantity determined by analysis

Q_A = Quantity added to samples/blanks

If transcription errors are discovered on Form II (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATE AND MATRIX DUPLICATE

MS/MSDs are not required by Methods TO-14, TO-15, and TO-17.

Matrix duplicate (MD) data are used to determine the effect of the matrix on a method's recovery efficiency and precision for a specific sample matrix. MD analyses are also performed to demonstrate acceptable method precision by the laboratory at the time of analysis.

Level C and Level D:

1. MD data should be reported on a summary form similar to Form III (or equivalent).
2. Compare the RPD for each spiked compound with project-specific control limits. Use in-house limits if there are no project-specific limits.
3. If the sample results are greater than 5× the LOQ and the RPDs between sample and duplicate results are greater than the control limits, detects for only the spiked compounds which showed high RPD in the parent sample shall be flagged as estimated "J."

Level D:

Check the raw data and recalculate one or more RPDs, especially RPDs that resulted in the qualification of data, using the following equations to verify that results on Form III (or equivalent) are correct.

$$RPD = \frac{ABS|SR - DR|}{(SR + DR)/2} \times 100$$

Where:

SR	=	sample result
DR	=	spiked duplicate result
ABS	=	absolute value

If transcription errors are discovered on Form III (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.8 FIELD QC SAMPLES

Field QC samples discussed in this section of this procedure are ambient blanks, field blanks, and field duplicates.

4.8.1 Ambient Blanks and Field Blanks

An ambient blank is collected in the same type of container used for an environmental sample, kept with the sample containers before sample collection and opened at the site and exposed to the ambient conditions. Ambient levels of site contaminants are determined by the analysis of ambient blanks.

A field blank is a sample collected in the field from a certified air source. Compounds detected in field blanks indicate the possibility of cross-contamination between samples due to improper equipment decontamination.

If target compounds are detected in the ambient blanks and/or field blanks, the procedure for the qualification of associated sample results is identical to the criteria outlined in Section 4.4.1 of this procedure.

Level C and Level D:

1. Determine which field QC samples apply to samples in the sample delivery group (SDG).
2. Because of the way in which the field blanks and equipment blanks are sampled, equipment blanks are not qualified because of field blank contamination. The affected samples are qualified, however, by either the field blank or equipment blank results, whichever has the higher contaminant concentration.
3. Ambient blanks and field blanks are only qualified with method blank results in order to account for laboratory contamination.

Level D:

1. Verify all target compound and TIC detects found in the ambient blanks and field blanks against the raw data.
2. Verify that the target compound detects have valid spectra, as defined in Section 4.10 and the tentative identity of any TICs against the raw data, as defined in Section 4.12. If the spectra are not valid, or if the tentative identity is in error, request for a corrected Form I (or equivalent) for the equipment blank or field blank from the laboratory.
3. Verify detected concentrations of target compounds and TICs from the raw data, as defined in Section 4.11. After the validity of the target compounds and TICs is verified, validate the corresponding data using the criteria outlined above.

4.8.2 Field Duplicates

Field duplicates are samples collected in the field simultaneously. Field duplicates should be collected in separate sample containers at the same location and depth. Field duplicate results are an indication of both field and laboratory precision; the results may be used to evaluate the consistency of sampling practices.

Level C and Level D:

1. Check to ensure that field duplicates were collected and analyzed as specified in the project planning documents. If the sampling frequency is less than the frequency stated in the planning documents, no qualification of the associated sample results is necessary but the incident shall be discussed in the data validation report.
2. For field duplicate results, if the RPDs are greater than 100 percent or as stated in the planning document if more conservative, no qualification of the associated sample results is necessary, but the differences should be noted in the data validation summary.

Level D:

1. Verify all target compound and TIC detects found in the field duplicates against the raw data.
2. Verify that the target compound detects have valid spectra, as defined in Section 4.10 and the tentative identity of any TICs against the raw data, as defined in Section 4.12. If the spectra are not valid, or if the tentative identity is in error, request for a corrected Form I for the field duplicates from the laboratory.
3. Verify detected concentrations of target compounds and TICs from the raw data, as defined in Section 4.11. After the validity of the target compounds and TICs is verified, validate the corresponding data using the criteria outlined above.

4.9 INTERNAL STANDARDS PERFORMANCE

Internal standards performance criteria ensure that GC/MS sensitivity and response are stable during every analytical run.

Level C and Level D:

1. If an internal standards area count for a sample is outside –50 percent or +100 percent of the area for the initial calibration midpoint standard:
    a. Positive results for compounds quantitated using an internal standards area count greater than 100 percent should be qualified as estimated "J." Nondetected compounds should not be qualified.
    b. Compounds quantitated using an internal standards area count less than 50 percent should be qualified as estimated "J" for detects and estimated "UJ" for nondetects.
    c. If extremely low area counts are reported (less than 20 percent of the area for associated standards), detected compounds should be qualified as estimated "J" and nondetected target compounds should then be qualified as unusable "R."
2. If an internal standards retention time varies by more than 20 seconds from the retention time of the initial calibration midpoint standard, the nondetected target compounds should be qualified as unusable "R" at Level C validation. A Level D validation examination of the raw data should be recommended to the CTO Manager. The chromatographic profile for that sample must be examined to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Positive results should be qualified as "NJ" if the mass spectral criteria are met.

Level D:

Verify the internal standard areas reported on Form VIII (or equivalent) from the raw data for at least one sample per SDG, and verify internal standard areas for samples that were qualified due to out-of-control internal standard areas. If errors are discovered between the raw data and the Form VIII (or equivalent), request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.10 TARGET COMPOUND IDENTIFICATION

The objective of the criteria for GC/MS qualitative analysis is to minimize the number of erroneous identifications of target compounds. An erroneous identification can either be false positive (reporting a compound present when it is not) or a false negative (not reporting a compound that is present).

The identification criteria can be applied more easily in detecting false positives than false negatives. More information is available for false positives due to the requirement for submittal of data supporting positive identifications. However, negatives, or nondetected compounds represent an absence of data and are therefore more difficult to assess. One example of detecting false negatives is the not reporting of a target compound that is reported as a TIC.

Level C:

Target compound identification is not evaluated for Level C validation since it requires the interpretation of mass spectral raw data.

Level D:

The following criteria should be followed when evaluating raw data.

1. The relative retention times (RRTs) must be within ± 0.06 RRT units of the standard RRT.
2. Mass spectra of the sample compound and a current laboratory-generated standard (i.e., the mass spectrum from the associated calibration standard) must match according to the following criteria:
    a. All ions present in the standard mass spectrum at a relative intensity greater than 10 percent must be present in the sample spectrum.
    b. The relative intensities of these ions must agree within ± 20 percent between the standard and sample spectra. (Example: For an ion with an abundance of 50 percent in the standard spectrum, the corresponding sample ion abundance must be between 30 percent and 70 percent.)
    c. Ions present at greater than 10 percent in the sample mass spectrum, but not present in the standard spectrum, must be considered and accounted for.
    d. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgment. It is up to the reviewer's discretion to obtain additional information from the laboratory and CTO Manager. If it is determined that incorrect identifications were made, all such data should be qualified as not detected "U" or unusable "R."
    e. Professional judgment must be used to qualify the data if it is determined that cross-contamination has occurred. Any changes made to the reported compounds or concerns regarding target compound identifications should be clearly indicated in the data validation report.

4.11 COMPOUND QUANTITATION AND REPORTING LIMITS

The objective is to ensure that the reported quantitation results and reporting limits (i.e., LOQ, LOD, decision level [DL]) are accurate. All soil sample results are reported on a dry weight basis.

Level C and Level D:

1. Verify that the reporting limits for nondetects are equal to the LODs. Verify that an annual DL study was performed or quarterly LOD/LOQ verification checks were performed in accordance with the DoD QSM (DoD 2013). The LOD verification check must be evaluated to determine whether the laboratory can reliably detect and identify all target analytes at a spike concentration of approximately two times but not more than four times the current reported DL. Qualify nondetects as unusable "R."
2. Check that reported nondetects and positive values have been adjusted to reflect sample dilutions. When a sample is analyzed at more than one dilution, the lowest LODs are used unless a QC criterion has been exceeded. In this case, the higher LODs from the diluted analysis are used. The least technically sound data will be flagged "R" with a qualification code "D."
3. Verify that no results exceed the highest calibration standard without being diluted. If a result has exceeded the highest calibration standard, verify that a dilution was performed. If

not, qualify the detected compound that required dilution as "J" and document the event in the data validation report.

Level D:

The compound quantitation must be evaluated for all detects by evaluating the raw data. Compound concentrations must be calculated based on the internal standards associated with that compound, as listed in the following equation. Quantitation must be based on the quantitation ion (m/z) specified in the analytical method for both the internal standards and target compounds. The compound quantitation must be based on the RRF from the appropriate ICAL standard.

Air

$$\mu g/m^3 = \frac{A_x \times I_s \times D_f \times MW \times Dw}{A_{is} \times ARRF \times Gas}$$

Where:

$\mu g/m^3$	=	microgram per cubic meter
A_x	=	area of characteristic ion for compound being measured
I_s	=	amount of internal standard added (parts per billion)
D_f	=	dilution factor
MW	=	molecular weight of compound
Dw	=	density of water (1.44 gram/milliliter)
A_{is}	=	area of characteristic ion for the internal standard
Gas	=	gas constant at 25^oC (24.45 mole/liter)
ARRF	=	average relative response factor for compound being measured

If discrepancies are discovered in the quantitation, request a resubmittal from the laboratory. Validate the data according to the criteria outlined above.

4.12 TENTATIVELY IDENTIFIED COMPOUNDS

For each sample, the laboratory must conduct a mass spectral search of the spectral library and report the possible identity for up to 30 of the largest volatile fraction peaks that are not system monitoring compounds (surrogates), internal standards, or target compounds, but which have area or height greater than 10 percent of the area or height of the nearest internal standard. TIC results are reported for each sample on the Form I or equivalent.

Level C and Level D:

1. All TIC results should be qualified "NJ," tentatively identified with approximated concentrations.
2. The reviewer should be aware of common laboratory artifacts and their sources such as siloxane compounds, which indicate capillary column degradation, and CO2 which indicates a possible air leak in the system. These may be qualified as unusable "R."

3. If a target compound is identified as a TIC by non-target library search procedures, the reviewer should request that the laboratory recalculate the result using the proper quantitation ion.
4. TIC results that are not above the 10× level in the blank should be qualified as unusable, "R." (Dilutions and sample size must be taken into account when comparing the amounts present in blanks and samples.)
5. The reviewer may elect to report all similar compounds as a total (e.g., all alkanes may be summarized and reported as total hydrocarbons).

Level D:

Check each TIC for each sample using the following criteria.

1. Major ions (greater than 10 percent relative intensity) in the reference spectrum should be present in the sample spectrum.
2. The relative intensities of the major ions should agree within ± 20 percent between the sample and the reference spectra.
3. Molecular ions present in the reference spectrum should be present in the sample spectrum.
4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination, interference, or co-elution of additional TIC or target compounds.
5. When the above criteria are not met, but in the technical judgment of the data reviewer or mass spectral interpretation specialist, the identification is correct, the data validator may report the identification.
6. Since TIC library searches often yield several candidate compounds having a close matching score, all reasonable choices must be considered. The reviewer may use judgment to change the reported tentative identity.

5. Records

A Form I or equivalent that has been validated and verified, and has been determined by the data validator to accurately represent the appropriate sample results to be utilized, shall be stamped "NAVFAC PACIFIC VALIDATED." Additionally, sample result forms for which the data has been validated at the Level D validation level shall be stamped or noted "Level D."

Copies of all documents generated by the data validation personnel will be stored for no less than 10 years. The original validated laboratory data shall be archived to the Federal Records Center at project completion.

6. References

Department of Defense, United States (DoD). 2005a. *Uniform Federal Policy for Quality Assurance Project Plans, Part 1: UFP-QAPP Manual*. Final Version 1. DoD: DTIC ADA 427785, EPA-505-B-04-900A. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www.epa.gov/fedfac/pdf/ufp_qapp_v1_0305.pdf.

———. 2005b. *Uniform Federal Policy for Quality Assurance Project Plans, Part 2B: Quality Assurance/quality Control Compendium: Minimum QA/QC Activities*. Final Version 1. DoD: DTIC ADA 426957, EPA-505-B-04-900B. In conjunction with the U. S. Environmental Protection Agency and the Department of Energy. Washington: Intergovernmental Data Quality Task Force. March. On-line updates available at: http://www2.epa.gov/sites/production/files/documents/qaqc_v1_0305.pdf.

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———. 2013. *Department of Defense Quality Systems Manual for Environmental Laboratories.* Version 5.0. Draft Final. Prepared by DoD Environmental Data Quality Workgroup and Department of Energy Consolidated Audit Program Operations Team. July.

Environmental Protection Agency, United States (EPA). 1999. *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air.* 2nd ed. EPA-625/R-96-010b. Center for Environmental Research Information. January.

———. 2007. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846.* 3rd ed., Revision 6. Office of Solid Waste. November. On-line updates at: http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm.

Procedure II-A, *Data Validation*.

7. Attachments

None.

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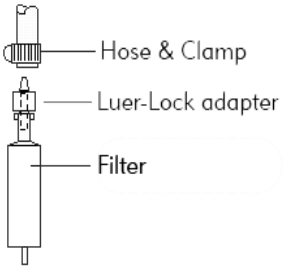
Bio-Flo – DNA Sampling Protocol

Sampling Instructions



1. Purge the well.
2. Prepare the pump (Peristaltic preferred, Grundfos, or air bladder) as normal. Use the clamp provided to ensure a leak-proof connection.
3. Remove the filter from the Falcon tube.
4. Attach the inlet of the filter with a 1/4" - 5/16" inner diameter (I.D.) tubing using the clamp to secure.
5. Place the filter within a receiving container so that the amount of water filtered can be measured accurately.
6. The amount of water filtered will vary depending upon the turbidity of the water. We recommend filtering 1-2 L.
7. Record the volume of water that passed through the filter, and then submit the filter for analysis. The water may then be discarded. Please cap the filter on both ends. The thinner end should be closed with the red rubber cap and the thicker end should be closed with the clear luer plug.

Note: If the filter clogs before 1L has been filtered, record how much water was passed through the first filter, and then collect an additional filter, also recording the volume of water that went through the second filter. In this case, both filters are then submitted for testing. For each location there should be **no more than 2 filters** used and there is no need to filter more than 2L of water.

To Submit Sample:

1. Place the filter in the Falcon tube provided.
2. Affix the label to the Falcon tube and note the amount of water that passed through the filter, the well location, sampling date, and the analyses requested.

Shipping Instructions

Packaging Samples:

1. Samples should be shipped in a cooler with ice or blue ice for next day delivery. If regular ice is used, the ice should be double bagged.
2. A chain of custody form must be included with each shipment of samples. Access our chain of custody at www.microbe.com

Shipment for Weekday Delivery:

Samples for weekday delivery should be shipped to:

Sample Custodian
Microbial Insights, Inc.
10515 Research Drive
Knoxville, TN 37932
(865) 573-8188

Shipment for Saturday Delivery:

Coolers to be delivered on Saturday must be sent to our . To ensure proper handling the following steps must be taken:

1. FedEx shipping label should be marked under (6) Special Handling, check Hold Saturday.
2. The cooler must be taped with FedEx SATURDAY tape.
3. The shipping label must be filled out with the Drop Location address below. Our laboratory name must be on the address label.
4. You MUST ***notify by email*** customerservice@microbe.com with the tracking number of the package on Friday (prior to 4pm Eastern Time) to arrange for Saturday pickup. Please make sure you write "Saturday Delivery" in the subject line of the message.

Samples for _____ should be shipped to:

Microbial Insights, Inc.
FedEx Drop Location
10601 Murdock Drive
Knoxville, TN 37932
(865) 300-8053

10515 Research Drive
Knoxville, TN 37932
Phone: 865.573.8188
Fax: 865.573.8133
www.microbe.com

Groundwater – DNA Sampling Protocol

Sampling Instructions

The following sampling instructions are used for collecting water or groundwater samples for DNA analysis by DGGE and/or CENSUS. The recommended sampling container is a 1L Poly bottle with a screw cap. Amber glass bottles can be used but are not recommended due to the likelihood of breakage during shipment. Microbial Insights, Inc. can provide the proper sampling supplies upon request.

Once the proper sampling bottle is obtained be sure not to contaminate the inside of the sample bottle with skin, dirt or any form of debris (this helps to ensure the accuracy of the data results). Wearing latex gloves (or similar) is recommended when sampling.

The required volume of water to conduct DNA based analyses from groundwater samples is 1L.

* Note: It is important to collect as close to the required amounts as possible to ensure the ability to properly conduct the analysis requested.

To Submit Sample:

1. Once the required amount of groundwater has been collected into the proper sampling container, seal the container tightly with a screw cap lid.
2. Properly affix a label with the sample name, date and analysis.
3. Be sure to fill out the Chain of Custody (COC) form correctly and accurately and ship it along with the samples. A COC form is required for QA/QC purposes.
4. Once the bottles have been correctly labeled, place them in the designated cooler. Be sure to fill the remaining space in the cooler with blue ice or regular ice that has been double bagged in Ziploc bags. Use sufficient ice to keep the entire shipment around 4°C, especially during the summer months.
5. All paperwork to be sent with the samples should be placed within a waterproof pouch or Ziploc bag and placed on top of the samples or affixed to the inside lid of the cooler.
6. Seal the cooler lid with a strong packaging tape.

Shipping Instructions

Packaging Samples:

1. Samples should be shipped in a cooler with ice or blue ice for next day delivery. If regular ice is used, the ice should be double bagged.
2. A chain of custody form must be included with each shipment of samples. Access our chain of custody at www.microbe.com .

Shipment for Weekday Delivery:

Samples for weekday delivery should be shipped to:

Sample Custodian
Microbial Insights, Inc.
10515 Research Drive
Knoxville, TN 37932
(865) 573-8188

Shipment for Saturday Delivery:

Coolers to be delivered on Saturday must be sent to our _____. To ensure proper handling the following steps must be taken:

1. FedEx shipping label should be marked under (6) Special Handling, check Hold Saturday,
2. The cooler must be taped with FedEx SATURDAY tape.
3. The shipping label must be filled out with the Drop Location address below. Our laboratory name must be on the address label.
4. You MUST ***notify by email*** customerservice@microbe.com with the tracking number of the package on Friday (prior to 4pm Eastern Time) to arrange for Saturday pickup. Please make sure you write "Saturday Delivery" in the subject line of the message.

Samples for _____ should be shipped to:

Microbial Insights, Inc.
FedEx Drop Location
10601 Murdock Drive
Knoxville, TN 37932
(865) 300-8053

Bio-Trap – Stable Isotope Probing Protocol

Sampling Instructions

Handling:

- Bio-Trap Samplers used for Stable Isotope Probing (SIP) are baited with ^{13}C-labeled contaminant of interest (e.g. benzene, MTBE, chlorobenzene) adsorbed onto the powder activated carbon (PAC). Controlled laboratory conditions show only minimal loss of contaminant due to volatilization. However, special considerations must be taken into account when handling SIP Bio-Trap Samplers in order to reduce the risk of volatilization.
- SIP Bio-Trap Samplers are shipped out chilled, on blue ice, and it is essential that they should be kept cool (not frozen) until deployment.
- When retrieving the Bio-Trap Samplers that have been deployed in the field, they should immediately be placed on ice and shipped on ice for next day delivery. These steps will ensure the most accurate results.
- Although the contaminant is absorbed onto the beads, caution should be used in handling these Bio-Trap Samplers because the contaminant compounds are associated with possible health and safety risks.

Note: Clean latex gloves (or similar) should be used at all times when handling the Bio-Trap Samplers.

Storage:

It is important to minimize the amount of time that Bio-Trap Samplers are stored prior to being installed in the field. The physical properties of the Bio-Trap Samplers that make them an ideal medium for collecting microbes also increase the chances of microbial or chemical contamination. Bio-Trap Samplers need to remain sealed and refrigerated (not frozen) until they can be installed in the field.

Installation:

- Prior to installing Bio-Trap Sampler, the monitoring well may need to be purged if it has not been sampled in a while. If purging is necessary, MI recommends that three well volumes be removed to ensure contact with formation water and reduce well bore effect.
- Attach the Bio-Trap Sampler's nylon loop (provided) to a nylon line (not provided) and suspend Bio-Trap Sampler at a depth where significant contaminant concentrations exist. If no data are available on the vertical distribution of contaminants, then suspend the Bio-Trap Sampler in the middle of the saturated screened interval.
- If large fluctuations in the water level are anticipated during the period of incubation, the Bio-Trap Sampler should be suspended from a float (contact MI for further details). Be sure not to suspend the bio-trap in the NAPL zone.
- Once installed, incubation times can vary depending upon the scope of the project. A typical Stable Isotope Probing (SIP) study incubation period is 30 days but is project dependant. Please contact us if you have questions regarding the optimum deployment period for your samples.

Retrieval:

- Open the monitoring well and pull up the Bio-Trap Sampler. Cut and remove the braided nylon line used to suspend the Bio-Trap Sampler.
- Transfer the recovered Bio-Trap Sampler to labeled (well number and date) zippered bags, seal and then double bag in a larger (one-gallon) zippered bag, immediately place on blue ice in a cooler.
- Repeat above for all the Bio-Trap Samplers from the site.
- A chain of custody (COC) form must be included with each shipment of samples.
- In order to minimize the potential effect of these samplers on the monitoring well, MI recommends purging three well volumes from the test well following the retrieval of the SIP Bio-Trap Samplers.

Shipping Instructions

Packaging Samples:

1. Samples should be shipped in a cooler with ice or blue ice for next day delivery. If regular ice is used, the ice should be double bagged.
2. A chain of custody form must be included with each shipment of samples. Access our chain of custody at www.microbe.com.

Shipment for Weekday Delivery:

Samples for weekday delivery should be shipped to:

Sample Custodian
Microbial Insights, Inc.
10515 Research Drive
Knoxville, TN 37932
(865) 573-8188

Shipment for Saturday Delivery:

Note: Microbial Insights, Inc is **closed** on Sunday, however we can receive samples on Saturday. Please contact us prior to shipping if the delivery of the samples is going to be on a Saturday.

Samples for Saturday delivery should be shipped to:

Microbial Insights, Inc.
FedEx Drop Location
10601 Murdock Drive
Knoxville, TN 37932
(865) 300-8053

Notes:

- **Stable Isotope Probing (SIP) may preclude subsequent Compound Specific Isotope Analysis (CSIA) in the study well for a period of time. CSIA can be performed prior to SIP or at another location.**

ISOTECH®
ISOTECH LABORATORIES INC
1308 Parkland Court Champaign, IL 61821 • (877) 362-4190 • www.isotechlabs.com

Collection of Ground Water Samples from Domestic and Municipal Water Wells for Dissolved Gas Analysis Using IsoFlasks®

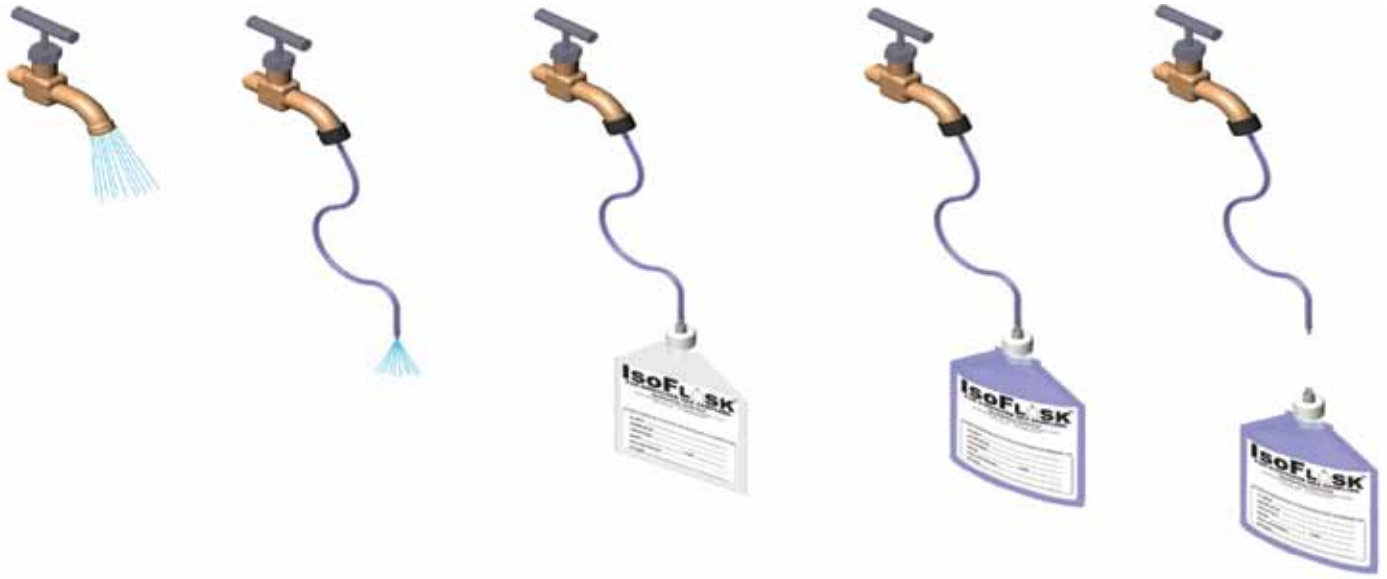
1. Sampling source: Water samples should either be collected from a pressurized water system or by using a suitable water pump. When sampling from a pressurized water system, it is recommended to use an outdoor spigot or other source which bypasses any water treatment systems (i.e. water softeners, etc.). When using a pump, it should be capable of maintaining a constant pressure at or above that which exists within the aquifer. This is to ensure that gases dissolved in the water within the aquifer remain dissolved until the water is transferred into an IsoFlask.
2. Record sample information onto the IsoFlask using the provided soft-tip, permanent pen.
3. Purge the well (see reverse side).
4. Attach the fill tube and purge with the source water. A control valve is included on the fill tube to assist in sampling. Use the control valve to stop/start flow into the IsoFlask (after purging).
5. The IsoFlasks have been evacuated in advance. A capsule filled with bactericide has also been inserted. A properly evacuated IsoFlask will be tightly held against the bactericide capsule. **There is no need to break the capsule even if you don't see it dissolving.** If the IsoFlask appears to have lost vacuum, do not use and contact Isotech for further instruction and/or replacement.
6. ***While the water is flowing*** attach the fill tube to an evacuated IsoFlask.
7. The IsoFlask should be filled with 600-700 cc of water (i.e. to a thickness of about 2 inches). When sufficient sample has been collected, close the sampling valve and quickly disconnect the fitting from the IsoFlask. The water flow can now be turned off and the hose disconnected. NOTE: Do not overfill the IsoFlask (the IsoFlask should not be pressurized).
8. Submission of samples: Place the IsoFlask into its protective box lying flat. Complete a Chain-of-Custody/Analysis Request Form and include it with the sample(s). Please note Isotech's receiving hours of Monday through Friday 8:00 a.m. to 4:30 p.m.

Ship samples to:
Isotech Laboratories, Inc.
1308 Parkland Court
Champaign, IL 61821

These instructions have been provided to simplify the collection of samples for dissolved gas analysis. Although we try to foresee and avoid problems in the field, it is never possible to predict every situation. If you encounter any difficulties, or if any additions or changes in these instructions would be beneficial, please let us know.

ISOTECH®
ISOTECH LABORATORIES INC
1308 Parkland Court Champaign, IL 61821 • (877) 362-4190 • www.isotechlabs.com

How to properly fill an IsoFlask



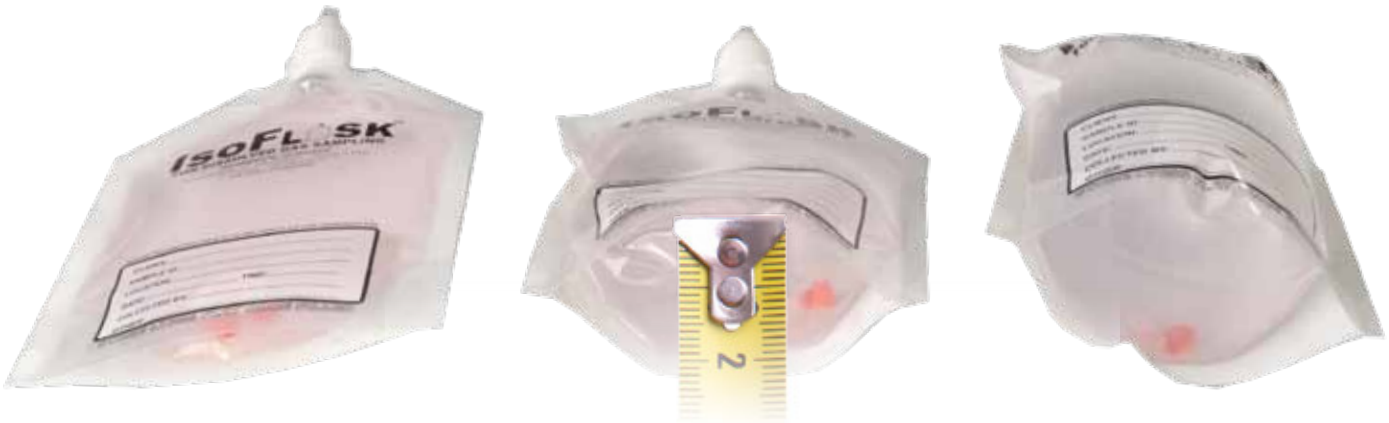
1
Purge line

2
Attach fill tube & purge

3
Attach evacuated IsoFlask (***while water is still flowing***)

4
Fill IsoFlask 2/3 full (***approximatley 2 inches thick, see below***)

5
Detach IsoFlask from fill tube



Not full enough | Correct | Too full

Do Not Try to Break the Capsule.
It does not need to be broken to release the bactericide.

Weatherford LABORATORIES

Appendix B:
Westbay Documentation

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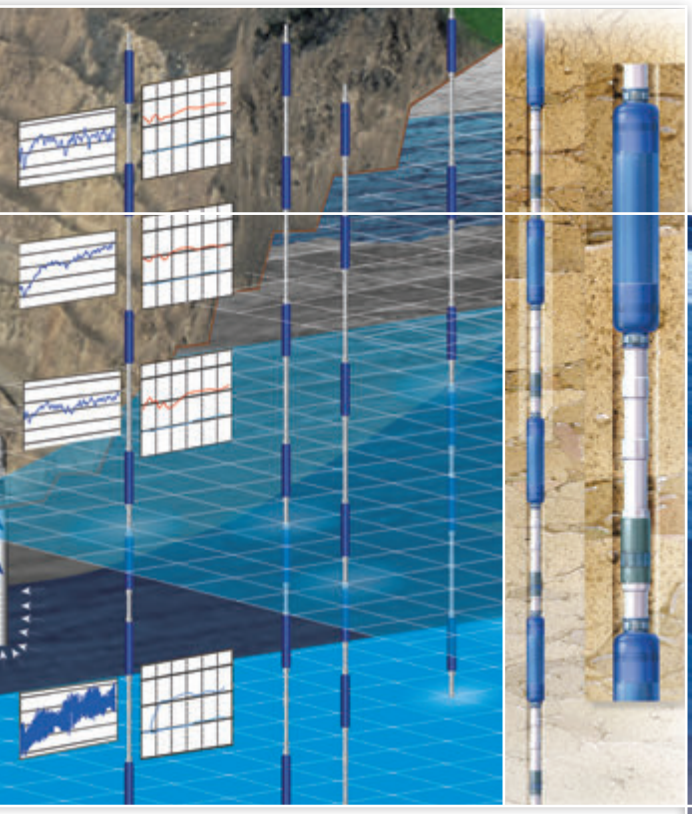
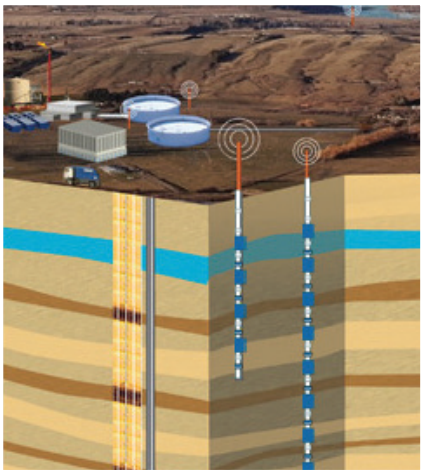
Westbay Instruments



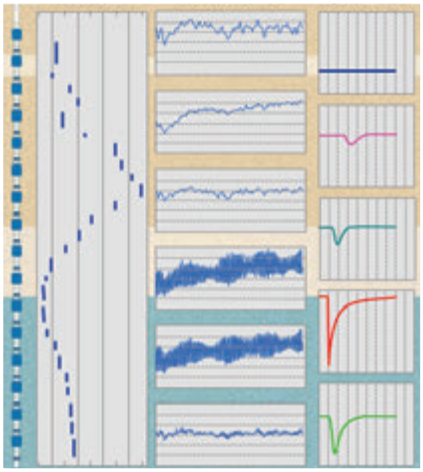
Westbay System

Multilevel Technology for Subsurface Characterization and Monitoring

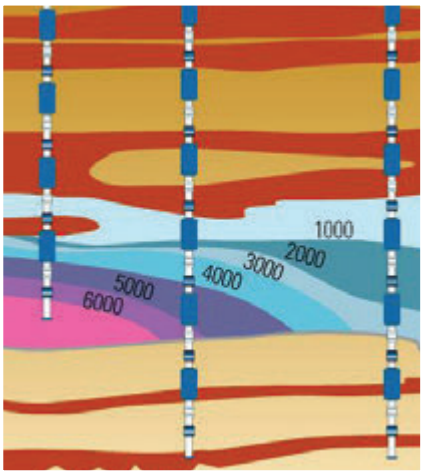
Is Groundwater Monitoring Important?



Environmental monitoring for unconventional oil and gas



4D subsurface characterization using Westbay technology



Characterization of contamination plume using Westbay System

WHY GROUNDWATER MONITORING?

Groundwater is an essential resource of great social, environmental and economic importance. With continuous population growth and industrial expansion impacting the state of groundwater around the world, implementing comprehensive groundwater management strategies is critical.

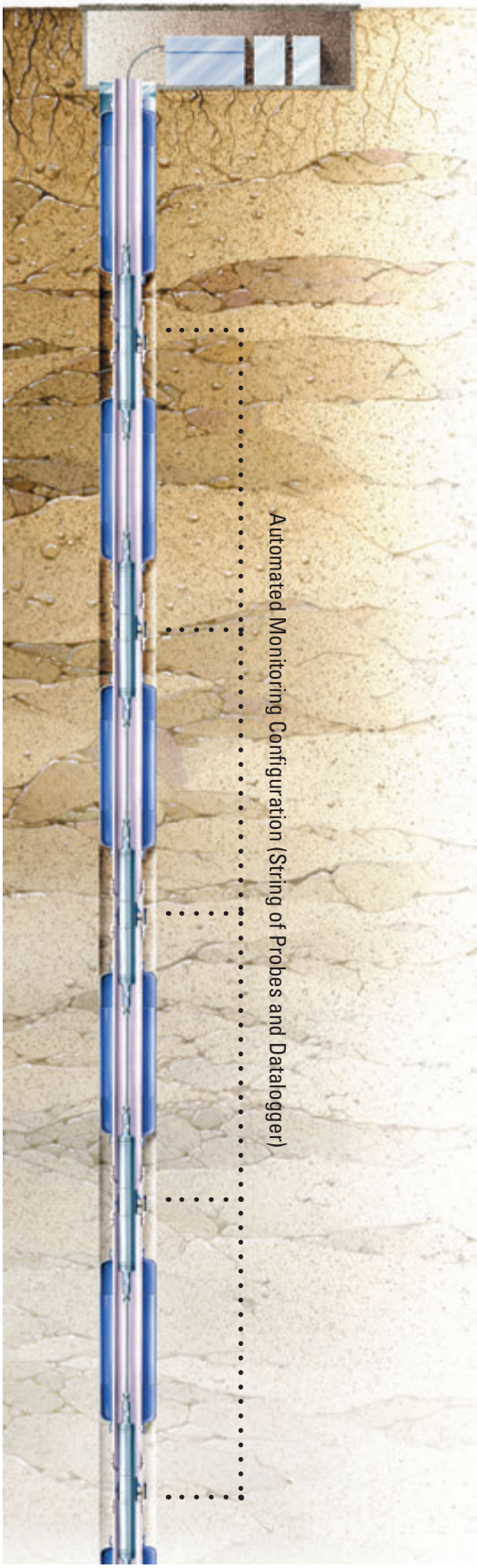
As an essential component of water management, groundwater monitoring networks are designed to optimize the collection of vast amounts of field data during the life of a project. Collection, analysis, and management of water levels and water quality parameters provide fundamental baseline information necessary for identifying potential risks and managing groundwater as a sustainable resource.

Groundwater monitoring networks:

- provide baseline data to map the spatial and temporal distribution of water quality
- identify short-term changes to groundwater flow from pumping, natural recharge and discharge, agricultural and industry use
- isolate impacts to groundwater from contaminant spills and releases
- present early warning of potential risks and the need for mitigation measures
- offer real-time accounting of water use and compliance with regulatory guidelines

OUR SOLUTION

Since 1978, the Westbay* System has provided its clients with a cost-effective, multilevel monitoring technology designed for long-term groundwater monitoring and data acquisition. The Westbay System is designed for collecting subsurface data at any number of discrete positions within a single well. Under even the most complex hydrogeologic conditions, this completely customizable system is a cost-effective, reliable solution that surpasses traditional monitoring methods.



Westbay System

Flexible, industry-tested design offers *Superior Performance*

OVERVIEW

The Westbay System is a completely versatile, multilevel monitoring technology that allows testing of hydraulic conductivity, monitoring of fluid pressure and collection of fluid samples from multiple zones within a single borehole. Designed for reliability and defensibility, the Westbay System can accommodate a wide variety of borehole conditions including diameter, depth, temperature and chemistry considerations.

Westbay System advantages:

- obtain measurements and samples at any number of discrete locations along a single borehole
- collect samples without purging
- designed for long-term monitoring
- engineered to operate at great depths
- reduced drilling and installation costs, with minimal site disturbance
- removable probes allow for convenient calibration and servicing
- built-in defensible QA/QC procedures

WELL COMPLETIONS

Westbay Systems are engineered with a unique, customizable casing system. The casing system is available in two sizes (MP38 and MP55) and manufactured from plastic or stainless steel to fit various borehole dimensions and operational requirements. Hydraulically-inflated packers and/or backfill provide engineered seals between monitoring zones, preventing unnatural flow and cross-contamination. Valved ports in the zones provide access for monitoring, sampling and hydraulic testing.

Westbay Systems can be installed in a number of different ways to suit geologic conditions, drilling methods, and project objectives.

Completion methods include:

- packers in open borehole
- packers through temporary casing
- packers in a cased well
- packers in cemented and perforated well
- direct backfill

WESTBAY SYSTEM PROBES

A variety of probes are available for use with the Westbay System. Reliable, accurate, and portable wireline-operated probes can be lowered into the casing system and used to:

- measure groundwater pressure
- test hydraulic parameters
- collect samples in-situ
- perform system specific tests

COLLECTING GROUNDWATER SAMPLES

Westbay Systems offer the unique ability to collect discrete fluid samples at formation pressure. For sample collection the probe and sample container are lowered to the desired depth, where the sample is collected into the container. The probe and container are then retrieved to the surface for further analysis.

Westbay System sampling allows you to:

- collect samples with minimal disturbance and without repeated purging
- maintain samples at formation pressure
- monitor pressure during sampling
- document quality assurance

1 PACKERS

- Engineered seal in a range of borehole sizes
- No dedicated inflation lines
- Controlled hydraulic inflation with record of pressure and volume
- Quality control tests to confirm performance at any time after installation

2 MEASUREMENT PORT

- For fluid pressure measurements, fluid sampling and low-k testing

3 PUMPING PORT

- For purging, hydraulic conductivity testing, and quality control testing.

Accurate, reliable long-term monitoring delivers *Definitive Results*

MEASURING GROUNDWATER PRESSURE

Westbay pressure probes can be used to take periodic, manual measurements of in-situ fluid pressures or to automatically monitor pressures using telemetry.

With a single probe, pressures are measured one port at a time. The output from the probes is digitized and transmitted through a rugged but lightweight wireline to a control unit at the surface. By attaching a standard laptop to the interface, data can easily be downloaded and stored for interpretation and analysis.

For automated multilevel measurements of fluid pressures, a string of pressure probes can be distributed down the well with each probe located at a selected measurement port. Each probe has a unique identity, allowing them to be polled individually or simultaneously by the datalogger.

Westbay Systems allow you to:

- measure pressure at multiple locations in a single well
- measure manually or automatically
- redeploy probes in alternate locations
- select from a variety of logging modes
- perform in-situ calibration checks
- document quality assurance

TESTING HYDRAULIC PARAMETERS

Westbay technology provides many effective methods for evaluating and testing the hydraulic characteristics of a site.

Discrete monitoring ports offer the unique ability to observe and record details within a single well.

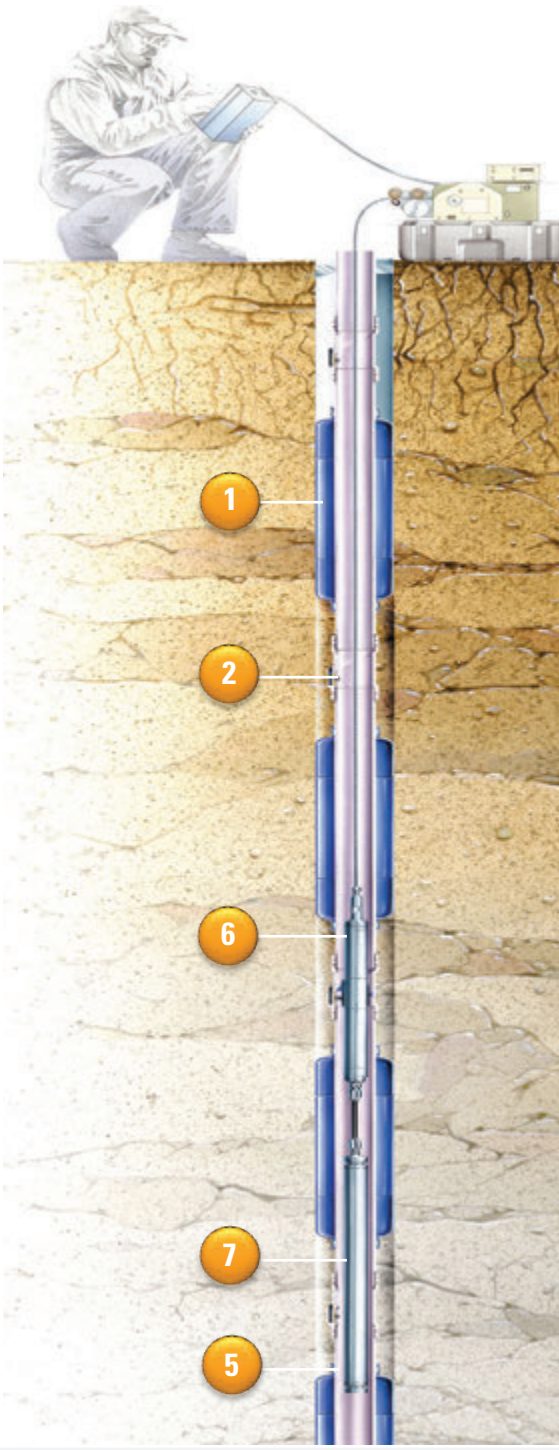
Westbay Systems allow you to:

- observe detailed distributions of groundwater pressures
- observe the effects of pumping tests or changes in barometric pressures
- gain insight into permability variations
- generate a stress in a monitoring zone and observe responses of neighbouring zones and wells

A number of qualitative and quantitative tests can be performed to determine the hydraulic parameters of formation materials or to verify the operation of the system.

- single-zone tests
- slug tests
- pulse-interference tests
- constant-head tests
- vertical interference tests
- cross hole tests
- tracer tests

As part of a complete environmental monitoring project, Westbay Systems are engineered to meet the rigorous demands of a wide range of operations. Westbay Systems provide the highest level data quality necessary to support critical decisions.



4 CENTRAL ACCESS CASING

- Made of plastic (PVC) or stainless steel
- Two sizes: 38 mm [1.5 in], 55 mm [2.2 in]
- Operational capability to depths of 1,200m [4,000 ft]

5 SEALED CONNECTIONS

- All casing connections sealed by o-rings

6 SAMPLER PROBE

- Independently controlled sampling valve
- Silicon strain-guage pressure transducer
- Location/activation mechanism compatible with Westbay System

7 SAMPLE CONTAINER

- Maintains sample pressure during recovery
- Easy to clean

Applications

Groundwater Resource Management	■ Groundwater basin management ■ Manage aquifer recharge operations ■ Seawater intrusion ■ Detailed long-term monitoring
Contaminant Site Investigations	■ Site characterization ■ Plume delineation ■ Remediation design and performance monitoring
Geologic Repositories	■ Site characterization ■ Determine feasibility of underground disposal site
Geotechnical Projects	■ Monitoring of pore pressure, slope stability for tunnels, subsidence and drainage ■ Groundwater pressure monitoring at large dams
Mining	■ Pre-feasibility planning and support ■ Subsurface characterization and monitoring ■ Acid rock drainage assessment and control ■ Monitoring of leach operations ■ Environmental impact assessment and site closure ■ Sub-permafrost groundwater monitoring
Unconventional Oil and Gas	■ Site characterization to reduce risk and minimize regulatory pushback ■ Evaluation of water management alternatives ■ Optimum placement, design and construction of injection wells ■ Compliance monitoring and minimization of cross-contamination ■ Closure design and performance monitoring

Features and Benefits

Features	■ Unlimited number of monitoring zones in a single well ■ Additional data at small incremental cost ■ Sealed monitoring zones ■ Collect water samples without repeated purging ■ Automated pressure monitoring at multiple depths	■ Wide suite of hydraulic test methods ■ Removable and upgradeable probes ■ Improved security ■ Excellent field quality control procedures ■ Custom components available to meet operational requirements
Benefits	■ Improve understanding of hydrogeological conditions and contaminant transport ■ Minimize drilling cost and time ■ Reduce site disturbances	■ Minimize wellbore storage effects ■ Minimize cross-contamination ■ Increase confidence in data ■ Reduce health, safety and environmental risks

Westbay Instruments

Operating worldwide since 1978

Over 2000 wells installed

www.westbay.com

WB-15-09-25 Printed in Canada.

NOVA METRIX

Multi-Level Groundwater Monitoring with the MP System®

Abstract

Defining the extent of a groundwater contaminant plume in geologic materials requires a three-dimensional array of sampling points. Such an array is commonly installed by placing a single access tube and inlet screen in each of a series of boreholes. With this method, the number of sampling points at a given site is generally limited by the high cost of drilling. An alternative is to install monitoring points at many levels in each borehole. Multi-level monitoring can provide increased data density and therefore an improved understanding of site conditions. This paper describes how the MP System®, one type of multi-level monitoring well, is installed and operated. Field quality control procedures, 1) to verify the integrity of the access tube, inlet valves, and borehole seals, and 2) to confirm the operation of measuring and sampling equipment, are also discussed.

Introduction

When groundwater contaminant plumes are suspected of having significant depth as well as lateral distribution, a three-dimensional array of monitoring points is needed to identify and characterize such plumes. Thus, groundwater data must be obtained from a number of different locations and from a number of different depths at each location. As a result, either a large number of boreholes are required, each with a separate instrument installed, or instruments must be combined and installed at multiple levels in each of a smaller number of boreholes.

Multi-level groundwater monitoring devices have been described by many writers, some discussing the technical benefits and others the advantages to schedules and costs which can result when multi-level monitoring devices are used to reduce the number of boreholes required. Most important, however, are the advantages that accrue from the increased data density and from the field verification procedures that are available. The very fact that one is capable of accessing several different discrete zones in one monitoring well provides a testing and verification capability that is simply not possible in a single-level device such as a standpipe monitor well.

The basic requirements of any groundwater monitoring system are that it provide the user with the ability to measure fluid pressure, purge the monitoring zone, collect fluid samples, and undertake standard hydrogeologic tests, such as permeability tests and tracer tests. In addition, quality assurance plans for groundwater monitoring programs have led to a requirement for periodic testing and calibration of all aspects of groundwater monitoring devices.

Quality assurance plans normally require field verification tests immediately following installation and again at periodic intervals during the operating lifetime of the installation. In fact, few groundwater monitoring devices are designed to allow extensive field verification tests to be carried out. However, some types of multi-level monitoring instruments, such as the MP System®, developed by Westbay Instruments Inc., were designed with field verification tests in mind (Patton and Smith, 1986). With such systems, questions of data quality can be readily addressed.

General Description of the MP System

The MP System is a modular multi-level groundwater monitoring device employing a single, closed access tube with valved ports. The valved ports are used to provide access to several different levels of a borehole through a single well casing. The modular design permits as many

monitoring zones as desired to be established in a borehole. Furthermore, at the time of installation, zones may be added or modified without affecting other zones or significantly complicating the installation. As a result, the number and location of monitoring zones can be decided based on the information obtained during drilling. Only a broad scope of requirements need be defined in advance of drilling.

The MP System consists of casing components, which are permanently installed in the borehole, portable pressure measurement and sampling probes, and specialized tools. The casing components include casing sections of various lengths, regular couplings, two types of valved port couplings with different capabilities, and packers, which seal the annulus between the monitoring zones. The MP System has been used in many different geologic and climatic environments in boreholes ranging from a few feet to over 4,000 ft (1,200 m) in length. The 1.5-inch (38 mm) I.D. MP38 System has been used in the field since 1978, while the 2.25-inch (55 mm) I.D. MP55 System was developed in 1990-91.

Casing Components

The casing components of the MP System are made in either plastic or stainless steel. While the illustrations are of plastic components, the descriptions of operating principles that follow apply to both types of materials. Most of the components referred to are shown in Figures 1 and 2.

Casing

MP casing is supplied in a number of different lengths to provide flexibility in establishing the position of monitoring zones and associated seals in the borehole. Common nominal casing lengths are 2 ft (0.5 m), 5 ft (1.5 m) and 10 ft (3.0 m). Actual casing lengths are less than the nominal lengths to account for the lengths of the couplings. The casing ends are machined to mate with MP System couplings.

Telescoping casing sections are used to protect the casing string from damage when ground movements are anticipated or where measurements of vertical displacements are desired.

Regular Couplings and End Caps

MP regular couplings are used to connect casing lengths where valved couplings are not required. The couplings incorporate O-rings for a positive hydraulic seal. A flexible shear rod provides a tensile connection. No adhesives are used when joining casings and couplings. MP38 regular couplings incorporate an internal, helical shoulder for the accurate location of probes and tools in the well. MP55 regular couplings do not incorporate a helical shoulder.

End caps are placed on the bottom of a casing string. They also incorporate an O-ring seal so that the entire casing string is hydraulically sealed during installation. End caps are frequently used to seal the top of the casing between monitoring periods.

Valved Couplings

There are two types of valved couplings, measurement port couplings and pumping port couplings. Measurement port couplings (or measurement ports) are used where pressure measurements and fluid samples are required. In addition to the features of a regular coupling (including the helical shoulder in the case of MP55), measurement ports incorporate a valve in the wall of the coupling, a leaf spring which normally holds the valve closed, and a cover plate or screen which holds the spring in place. When the valve is opened, an access port is provided for the groundwater to enter the coupling.

Pumping port couplings (or pumping ports) are used where the injection or withdrawal of larger volumes of fluid is desired than would be reasonable through the relatively small measurement port valve (such as for purging or hydraulic conductivity testing). Pumping ports incorporate a sleeve valve, sealed by O-rings, which can be moved to expose or cover slots that allow groundwater to pass through the wall of the coupling. A screen is normally fastened around the coupling outside the slots.

Annulus Seals

When there are many monitoring zones in a single borehole, multiple seals are required to prevent fluid migration from one zone to another along the annular opening between the borehole wall and the casing. Placement of these seals can be difficult with any groundwater monitoring device. However, considerable success has been achieved with three types of well completion used with the MP System, provided each is combined with appropriate drilling and placement methods.

With the MP System, seals can be obtained by: a) backfilling with alternating layers of sand and bentonite or grout, b) using hydraulic (water) inflated packers or c) using packers inside a cased well with multiple screens. Figure 1 illustrates a borehole containing the MP System with packers. Figure 2 illustrates a single measurement zone where the MP System is completed by each of the three common methods. Each sealing method is possible in most environments, but in many situations one method will stand out as the most advantageous.

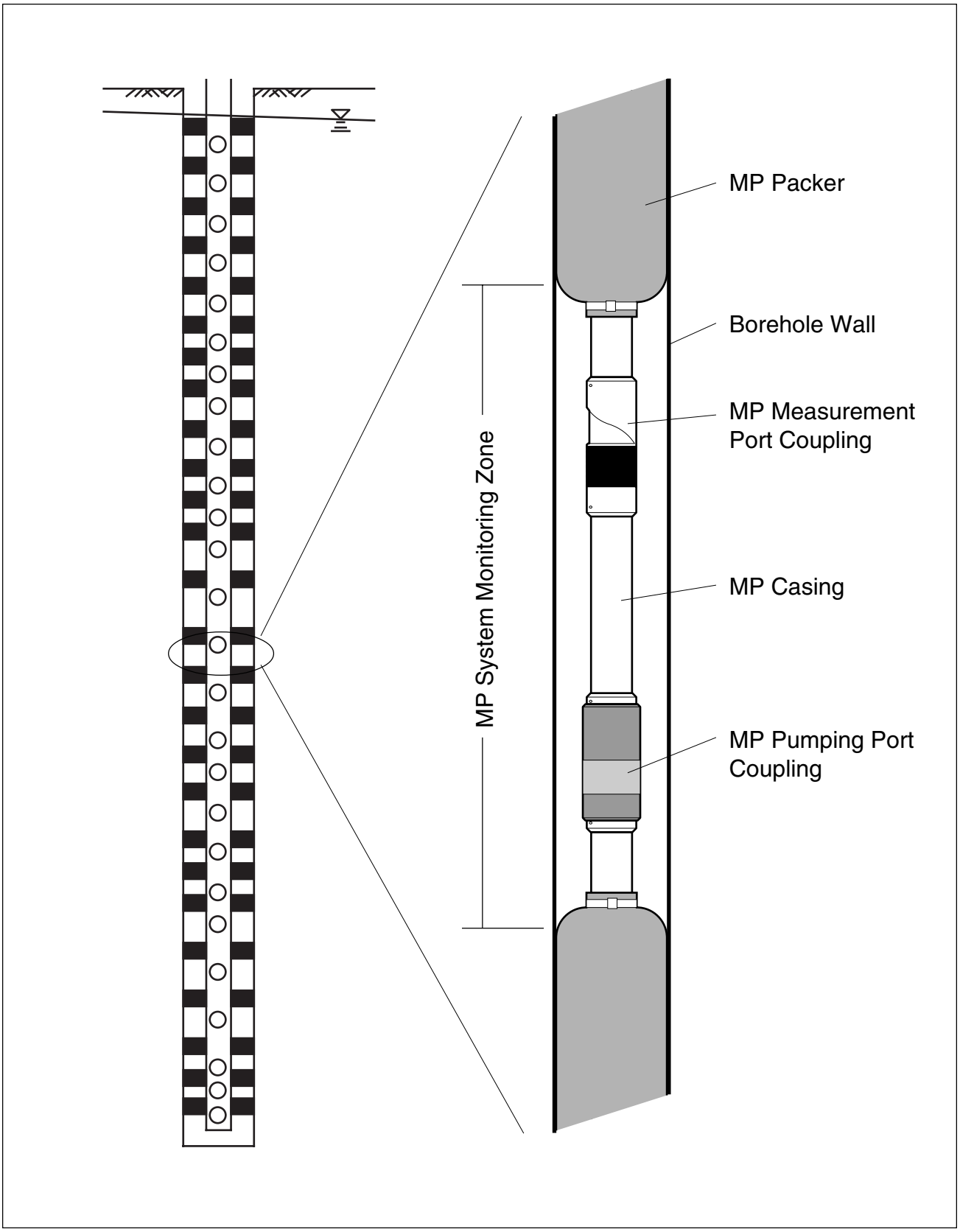


Figure 1. MP System installation with monitoring zones isolated by packers.

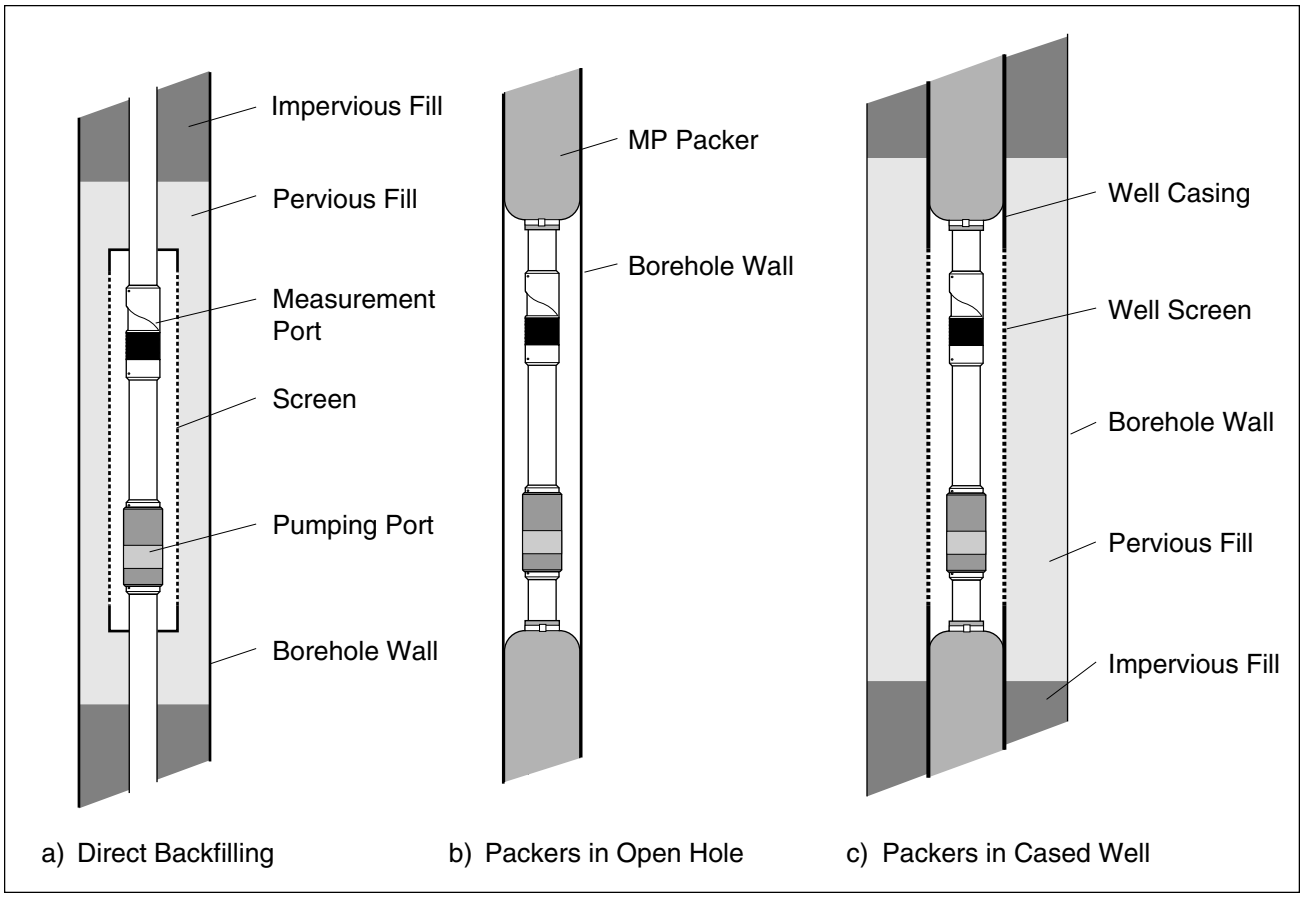


Figure 2. Common completion methods for the MP System.

Direct backfilling (Figure 2a) is recommended for: 1) large diameter boreholes, 2) shallow boreholes, 3) boreholes where little or no fluid circulation is anticipated in the hole during installation (i.e., when near-hydrostatic fluid pressures or low hydraulic conductivity is present over the length of the borehole), and d) where packer gland materials are incompatible with the chemistry of the fluids present.

When direct backfilling is considered and fluid sampling is required, a very clean drilling method must be employed. While the MP System does permit purging of monitoring zones, the small size of the casing (particularly MP38) prevents sufficient energy being generated to develop the monitoring zone.

Backfill seals may include bentonite and / or grout slurries, bentonite chips or pellets or other materials with a relatively low hydraulic conductivity in comparison to that of the natural formations present.

MP casing packers incorporate an expandable gland mounted over a standard length of MP casing. The casing incorporates a one-way valve that allows fluid to travel through the wall of the casing into the packer and prevents this fluid from flowing back out of the packer. Gland lengths are typically 3 ft (~1 m).

Packers in an open borehole (Figure 2b) are typically recommended for: 1) small diameter boreholes (those too small for good quality backfilling to be achieved), 2) deep boreholes, and 3) sealing against significant flows (e.g., flowing artesian conditions) in the borehole. When packers are used, field labour is reduced since packer inflation is generally much faster than backfilling. When using packers, additional measurement ports are installed between monitoring zones. Such additional ports provide additional fluid pressure data for quality assurance (QA) purposes.

Packers in a cased well (Figure 2c) is a completion method that has proven very successful, particularly for environments where available hole sizes are too large for packers and / or where drilling additives, such as mud, must be used. This completion method involves drilling a large diameter hole, typically 12-inch (300 mm) and installing a 4-inch (100 mm) (for MP38) nominal diameter well casing with multiple screens. The well screens are located at all of the desired monitoring levels, based on information gathered during and following

drilling. Layers of backfill are placed to provide filters around the well screens and annular seals between. Each monitoring zone is then developed through the well casing. Following development, MP casing, ports and packers are installed inside the well casing. The MP packers are inflated against the inside of the well casing, providing interior annular seals between the monitoring zones. This completion method provides the ability to properly develop mud from deep mud-rotary boreholes, as well as to service the MP System during the operating life of the monitoring well.

Whenever casing packers are used, whether in open boreholes or cased wells, additional measurement ports are installed between monitoring zones for QA purposes. Measurements and tests carried out through these additional "QA ports" enable an evaluation of the effectiveness of each annulus seal. In open hole installations, such additional ports also provide added information on piezometric pressures in the portions of the borehole between primary monitoring zones.

Screens and Filters

Where both pumping ports and measurement ports are being used and the ports are likely to be surrounded by sand fill or collapsed geologic material, a single well screen is generally placed over both the measurement port coupling and pumping port coupling in each monitoring zone as shown in Figure 2a. The screen helps ensure that the zone influenced by pumping through a pumping port coupling will extend to and include the region surrounding the adjacent measurement port coupling. Screen slot size and length should be chosen with a knowledge of local site conditions. If only fluid pressure measurements are required, a simpler fabric filter tube can be placed over the measurement port coupling and clamped at either end. This filter will help maintain the length of the monitoring zone and protect the measurement port valve from fine particles. The filter material should be compatible with the chemistry of fluids present.

Installation Procedures

Selection of Casing Components

The valved couplings (measurement port couplings and pumping port couplings) allow many monitoring zones to be established in a single borehole. Horizons of hydrogeological interest are targeted on the basis of the best borehole geologic and geophysical logs available. An installation log is prepared showing the locations of the casing components. If only fluid pressures are needed, only a measurement port coupling is required in each monitoring zone. If sampling, fluid withdrawal or fluid injection is anticipated, both a pumping port coupling and a measurement port coupling are recommended in each monitoring zone. This is the case illustrated in Figures 1 and 2.

The casing lengths are chosen based on the desired locations of the monitoring zones and sealing elements. This requires an interpretation of the hydrogeologic conditions anticipated in each borehole. Caliper logs and borehole video can be useful in selecting packer locations.

If consolidation or heave is expected along the borehole axis, telescoping casing sections may be used to minimize the opportunity for compressional or tensile forces to damage the casing.

MP Casing Installation

The downhole MP System components - casing, couplings and packers - are laid out at the site of the proposed monitoring well in accordance with the casing installation log. At that time, any last minute adjustments required to make the positions of the monitoring zones and seals match hydrogeologic details of the borehole are completed and the appropriate revisions made to the installation log.

Next, the required coupling is attached to the top of each length of casing. The casing layout is checked again for compliance with the installation log. Serial numbers of measurement ports, pumping ports and packers are recorded, indicating their position on the installation log. The length of all casing sections is measured and recorded on the log.

The casing string is then assembled by lowering the casing segments into the borehole and attaching each successive segment to the adjacent coupling one at a time. As each successive MP casing section is attached to the string in the well, the section number is checked and recorded on the installation log. The coupling joint is then subjected to an internal hydraulic pressure to verify its hydraulic integrity and the test result is recorded on the log. At intervals during placement of the MP System casing clean water is added to the inside of the MP casing to reduce its buoyancy.

In collapsing soil and poor quality rock, MP casing with packers and screens may be installed through flush-jointed guide tube such as drill rods or casing. Table 1 provides ranges of borehole, casing and guide tube sizes for the MP38 and MP55 Systems. Figure 3 illustrates the major stages of installing through a guide tube:
A) Following completion of drilling, the guide tube is positioned in the hole. All parts of the guide tube, including any shoe attached to the bottom, must be flush on the interior and of sufficient inside diameter to permit the MP components to pass through; B) The MP components are assembled and lowered into the guide

System	I.D.		Max. Depth		Borehole/Casing Size		Min. Guide Tube Size	
	in.	mm	ft	m	in.	mm	in.	mm
Plastic MP38	1.5	38	1,500	450	3-4 1/2	75-115	3	75
Steel MP38	1.5	38	5,000	1,500	4-4 1/2	100-115	4	100
Plastic MP55	2.25	55	4,000	1,200	4 3/4-6 1/4	121-159	4 3/4	121
Steel MP55	2.25	55	6,600	2,000	4 3/4-6 1/4	121-159	4 3/4	121

Table 1. Important dimensions for the MP System.

tube in such a fashion that the packers and ports will be correctly positioned in the hole when the bottom of the MP is resting on the bottom of the borehole; C) The guide tube is pulled back to expose a packer and that packer is inflated. The pulling / inflating sequence is repeated until all of the packers have been inflated. More than one packer may be exposed during each pull of the guide tube, depending upon the stability of the borehole walls.

Casing without packers can be placed in various sizes of boreholes, with or without protective casing, as long as the borehole diameter (and casing) is compatible with the backfilling method. Good backfilling techniques involve the use of one or more tremie pipes.

Once the MP casing has been placed in the borehole, the packers are inflated (see Figure 3) or backfill is placed. If the MP casing was lowered inside a guide tube, the guide tube may be withdrawn all at once or in steps as the packer inflation or backfilling operation proceeds. An incremental casing withdrawal can reduce the opportunity for the borehole wall to loosen and cave prior to the placement of seals.

Packer Inflation

Figure 4a shows the appearance of a casing packer when it has been placed in a borehole before inflation. Figure 4b shows how the MP System casing packers are individually inflated using a packer inflation tool. This tool is lowered down the inside of the MP casing and is located in the correct position by the location arm seating in a coupling adjacent to the packer.

Two small packers (tool packers) are inflated, isolating the short segment of the casing containing the valve for the casing packer. At a pre-set pressure, the tool injection valve opens and water is injected into the casing packer. During inflation the vent-head mechanism on the tool holds open the measurement port beneath the packer. This vents the pressure in the zone below the packer, allowing the packer to square-off without generating unnatural squeeze pressures. Figure 4c shows the inflated MP packer after the inflation tool has been removed. At increments of volume during the inflation process, pumping is stopped and the fluid pressure of the inflation system is measured and recorded. The pressure / volume data is plotted and kept for quality assurance purposes.

Packer inflation proceeds from the bottom of the hole to the top. There are no permanent inflation lines leading to each packer. As a result, there is no limit to the number of packers that can be placed in a borehole apart from the finite limitations of packer length and borehole length.

Purging Monitoring Zones

The strategy for purging the monitoring zones may vary depending on site conditions. Figure 5 shows a typical sequence of events in drilling and completing a monitoring well. Figure 5a shows a typical borehole environment where the invasion of drilling fluids and / or the unnatural circulation of formation fluids has caused groundwater adjacent to the borehole to be nonrepresentative of the formation fluid. Once the casing and annular seals (packer seals are shown in Figure 5b) have been installed, it is usually desirable to remove the nonrepresentative fluid. This removal, or purging, can be done in one of two basic ways: 1) Purging by natural groundwater flow, or 2) Pumping to purge monitoring zones.

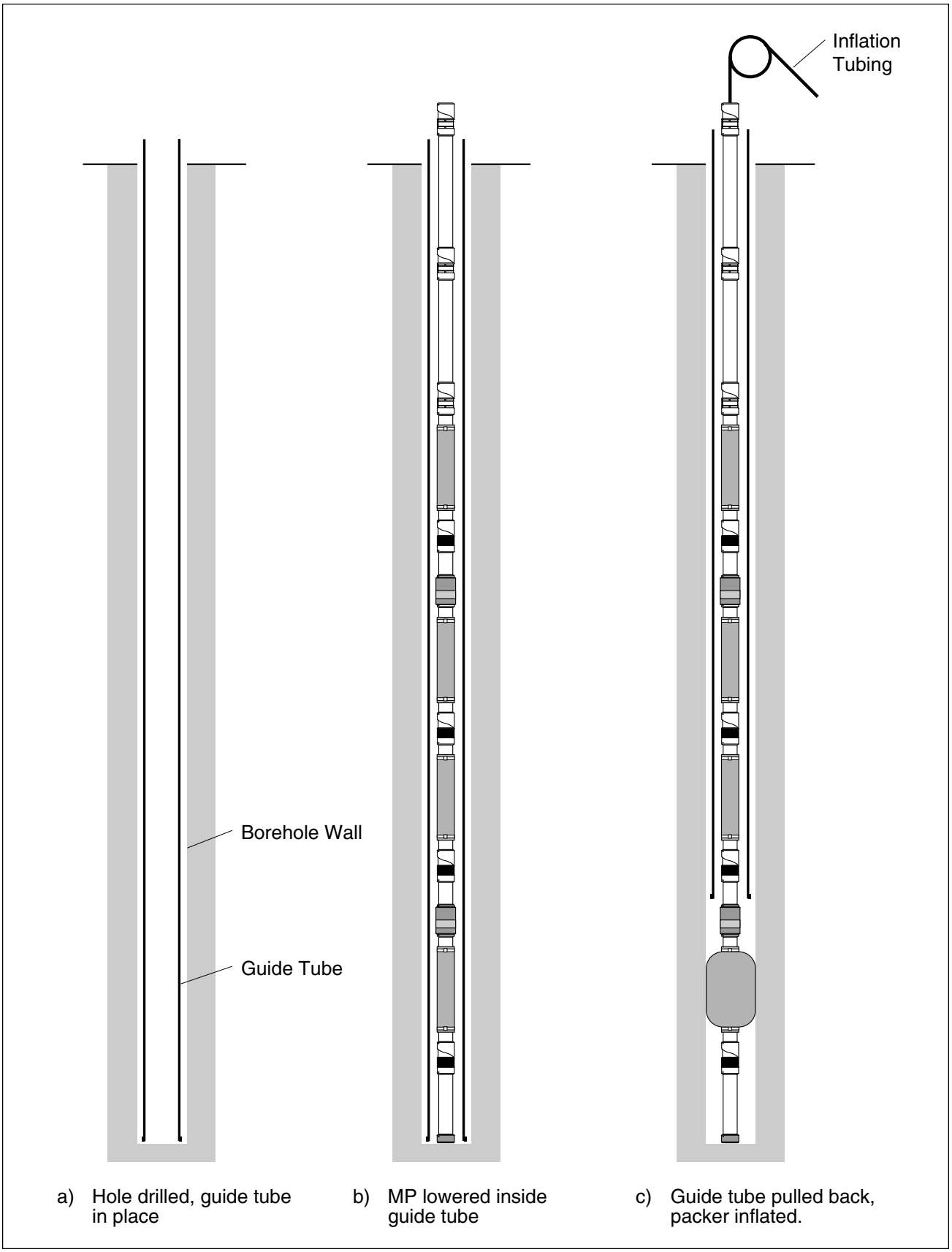


Figure 3. Installation of MP casing through a guide tube.

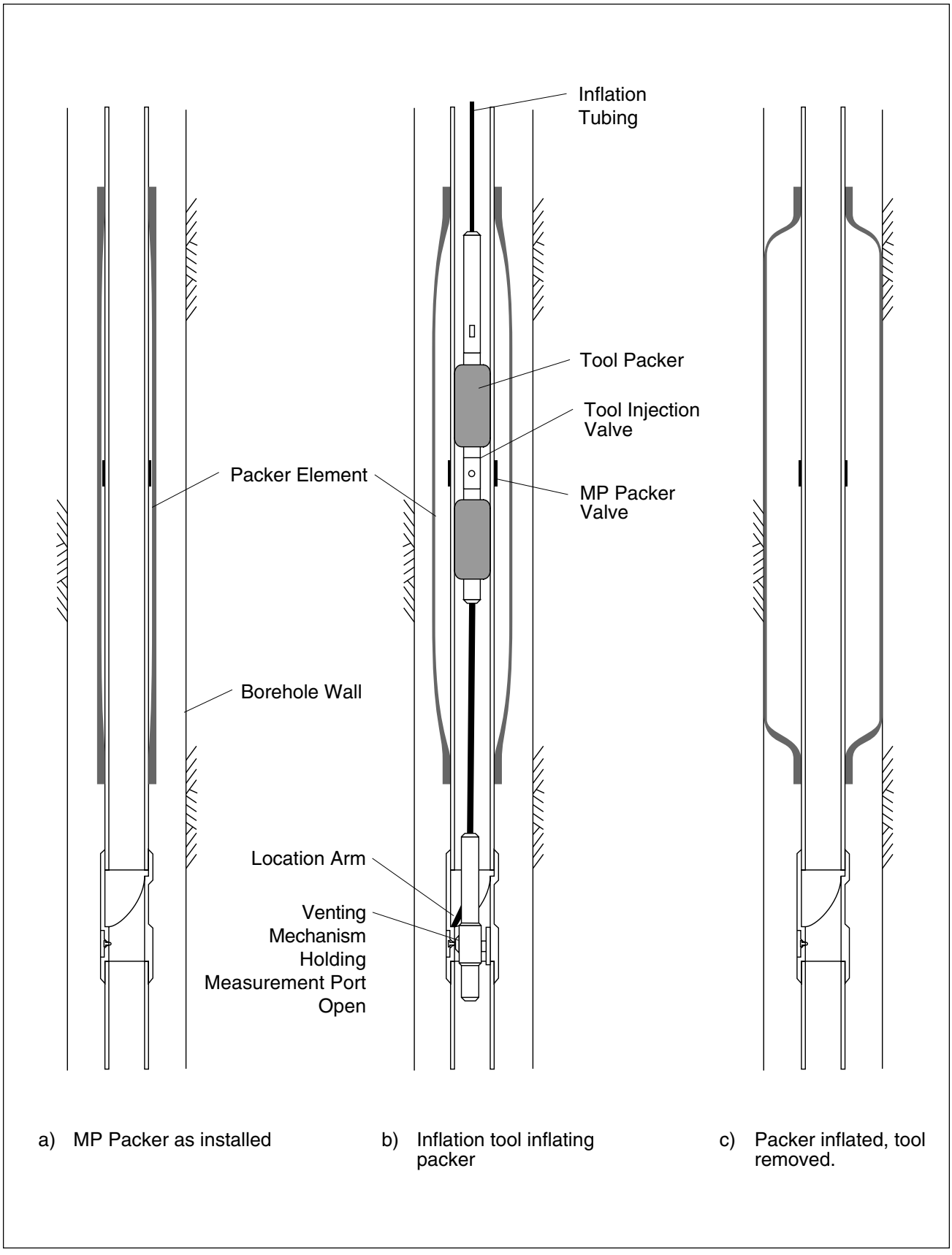


Figure 4. Steps in the inflation of an MP System packer.

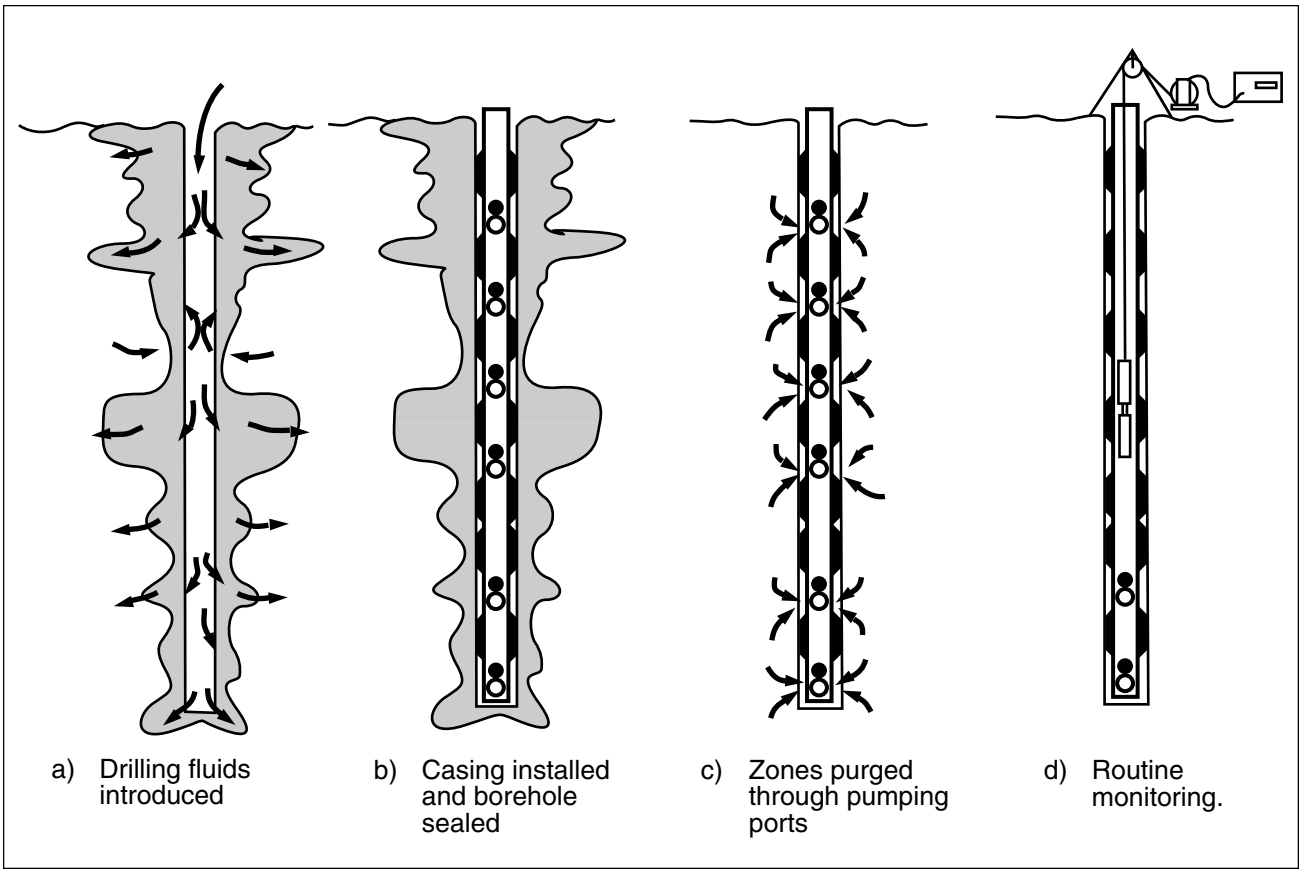


Figure 5. Typical sequence of events in purging monitoring zones.

Purging by natural groundwater flow is attractive, particularly in environments where groundwater flow is understood to be relatively rapid. In such an environment, unnatural fluids introduced during drilling may no longer be adjacent to the borehole by the time the monitoring system has been installed. In such a case, there may be little to be gained from the investment of time and resources to pumping an arbitrary volume of water from each monitoring zone. Rather, fluid samples might be collected over a period of time and analytical results compared in order to evaluate the stabilization of conditions in the monitoring zone.

When purging by natural flow is not acceptable, monitoring zones can be purged by pumping. Zones may be pumped individually or several at a time (as shown in Figure 5c). Individual hydrogeologists and hydrochemists may prefer different purging techniques depending upon local conditions. However, the purging procedures are essentially the same as would be used for a single standpipe piezometer. One procedure which has been successfully used is described below.

1) An acceptable and convenient tracer is added to the drill fluid during drilling.

2) After the casing has been installed and the packers have been inflated, the pumping ports in all or a portion of the monitoring zones are opened with the use of an open / close tool.

3) Fluid from the inside of the MP casing is pumped out of the well. The volume of fluid removed and the pumping time will depend on many factors including: the drilling method, the length of time the hole was left open prior to completion, the hydrogeological conditions in the borehole, and the accuracy required. The use of a tracer can be helpful in determining when the pumping is completed.

4) Once pumping has been completed, all the pumping ports except one are closed with the use of the open / close tool. With one pumping port open, the MP casing is hydraulically identical to a standpipe piezometer. A quantity of fluid may be pumped from inside the MP casing to complete the development of this monitoring zone. Hydrogeologic testing of this zone and its adjacent casing seals can be done at this time. For example, slug tests can be undertaken to obtain transmissivity and storativity values. This

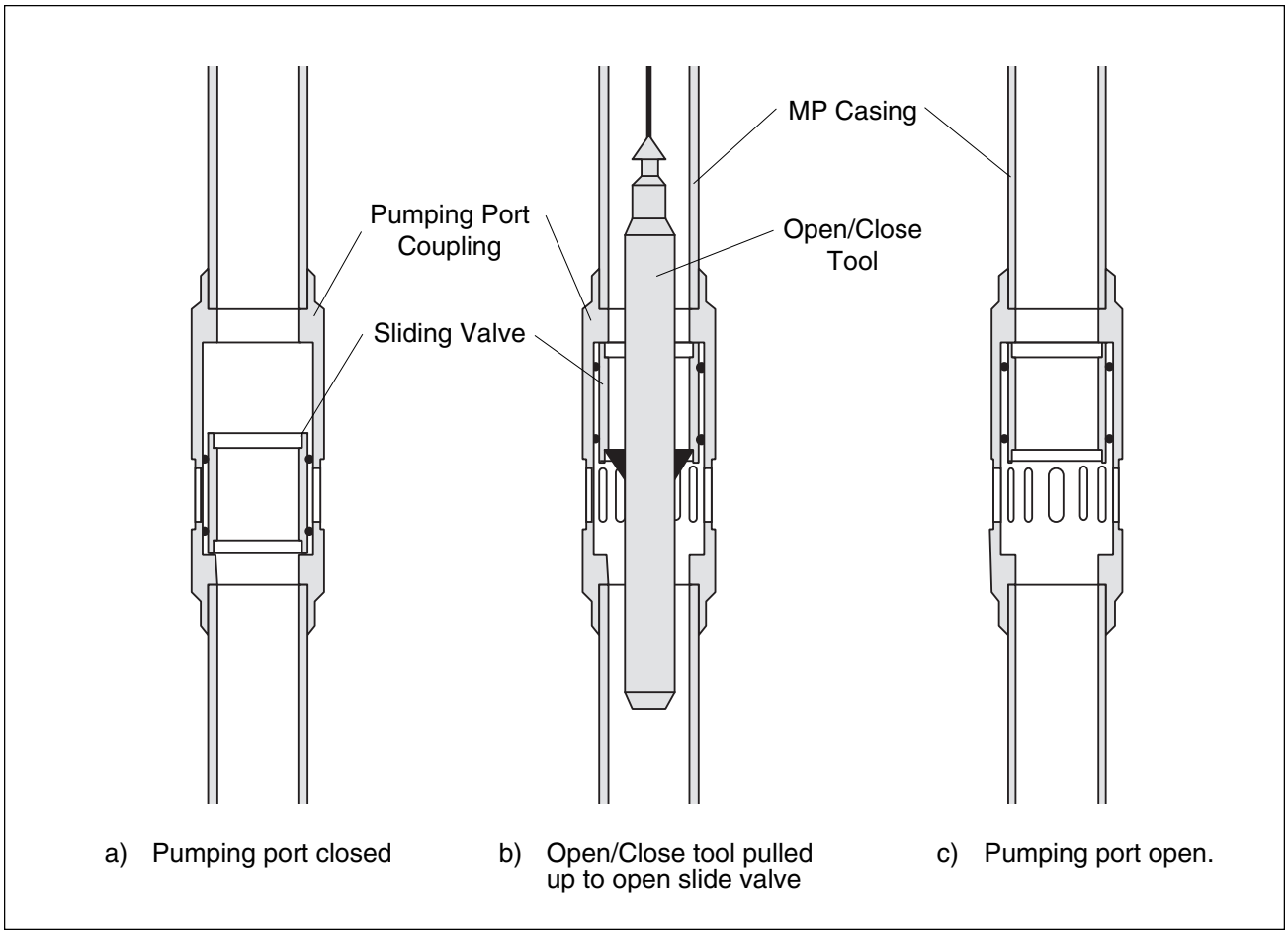


Figure 6. Operation of an MP pumping port.

pumping port can then be closed, the next one opened and the process repeated.

Following purging, the MP System is ready for sampling and for pressure measurements as indicated in Figure 5d.

Operation of the Pumping Ports

To operate the pumping port valve, an open / close tool is used as illustrated in Figure 6. This tool has spring-loaded "jaws" which can be mechanically activated from the surface. The pumping port is shown closed in Figure 6a. To open the valve, the tool is lowered on a wireline with the jaws extended and pointing upward (i.e., so that they will catch on shoulders when the tool is raised). In this condition, the jaws will spring through the couplings as the tool is lowered to just below the desired pumping port coupling. The tool is then pulled up so that the jaws engage the bottom shoulder of the sliding valve. By continuing to pull up on the wireline, the valve can be opened, as in Figure 6b. Once the valve is opened, the jaws can be collapsed into the housing and the tool recovered. With this one valve opened, fluids can be added to or removed from the monitoring interval by injecting or pumping from the MP casing. Other zones may still be monitored in the normal manner using a pressure probe or sampling probe as they will not be hydraulically connected to the interior of the casing.

To close the pumping port coupling, the open / close tool is brought to the surface and the housing is reversed so that the jaws point downward (i.e., the tool will stop on exposed shoulders when the tool is lowered). The tool is lowered to the open pumping port with the jaws collapsed into the housing. Once the tool is located near the pumping port, the jaws are released and the valve is closed by tapping on the top shoulder of the sliding valve with the tool.

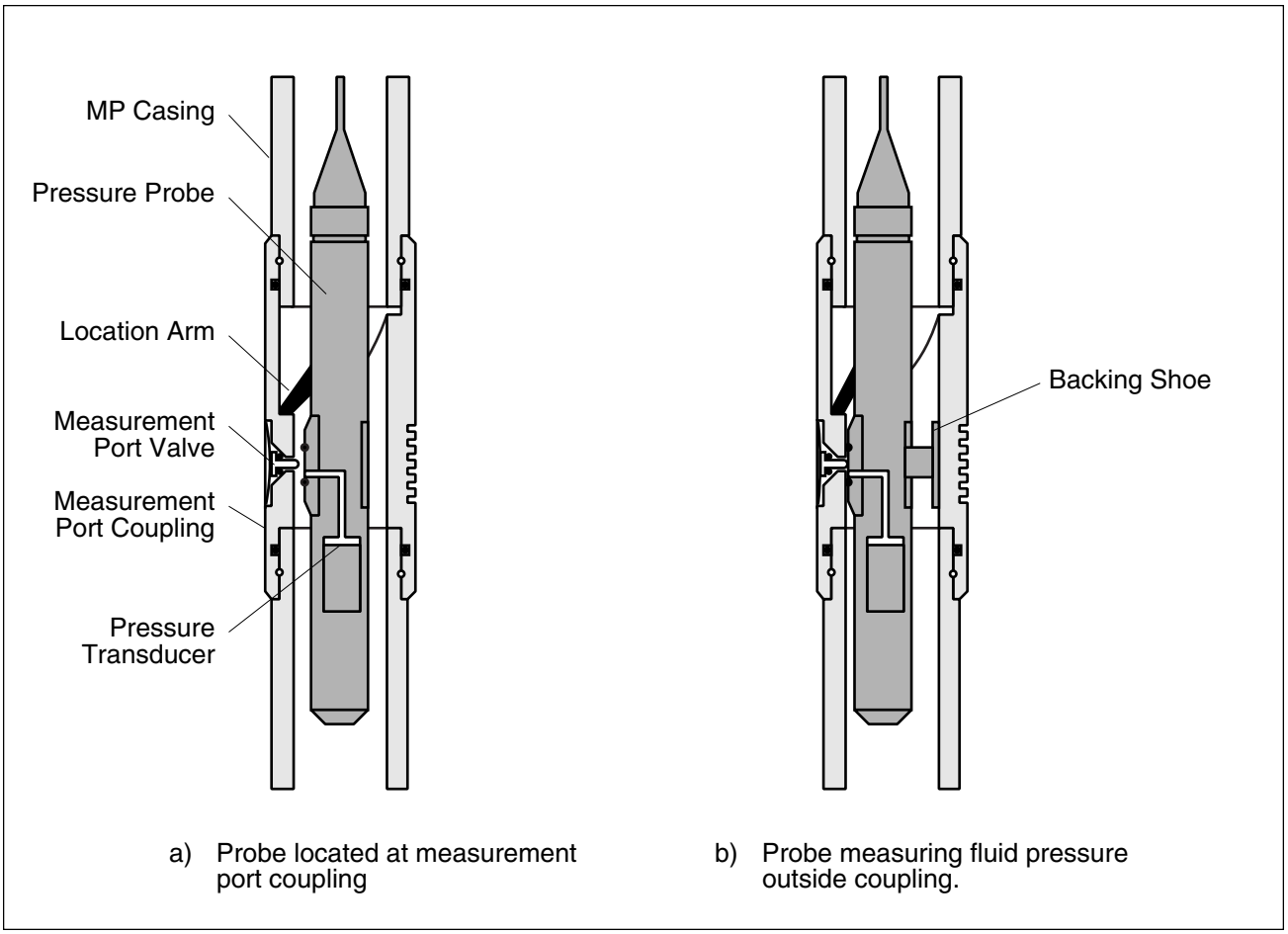


Figure 7. Operation of a pressure probe.

Testing and Monitoring

Fluid Pressure Measurements

Fluid pressure measurements can be made at each location in a borehole where an MP measurement port coupling has been installed. The measurement coupling includes a helical landing ring and a leaf spring valve which is normally closed. The fluid pressure is measured using a MOSDAX® pressure probe which incorporates a location arm, a backing shoe, a face seal, and a fluid pressure transducer. These features are shown on Figure 7. The probe is operated on a cable connected to an interface and portable computer at the top of the monitoring well. Using MProfile™ software, the computer displays the pressure both graphically and digitally, along with transducer temperature, well information and probe status (see Figure 8).

The following procedure is used to make fluid pressure measurements. The probe is lowered to a point below the first measurement port to be accessed (usually the deepest). The location arm is released from within the probe body. The probe is raised to just above the measurement port coupling and then lowered until the location arm rests on the helical landing ring in the coupling. The weight of the probe causes it to rotate into position at the correct depth and orientation to operate the valve (Fig. 7a). At this point the pressure transducer is measuring the fluid pressure inside the MP casing at that depth. This reading will be displayed on the surface computer and is recorded. If convenient, the depth to water inside the MP casing is also measured and recorded at this time as a check on the pressure transducer.

The backing shoe is then activated. It pushes the probe to the wall of the coupling so that the face seal on the probe seals around the measurement port valve at the same time as the face of the probe pushes the valve open. The transducer is now hydraulically connected to the fluid outside the coupling and isolated from the fluid inside the casing (Fig. 7b). The reading displayed on the surface computer will be the fluid pressure in the formation outside the measurement port. The pressure outside the port can be observed as long as desired and recorded as often as desired. After the reading has been recorded, the probe backing shoe is deactivated (retracted) and the valve in the coupling reseals. The probe will again be

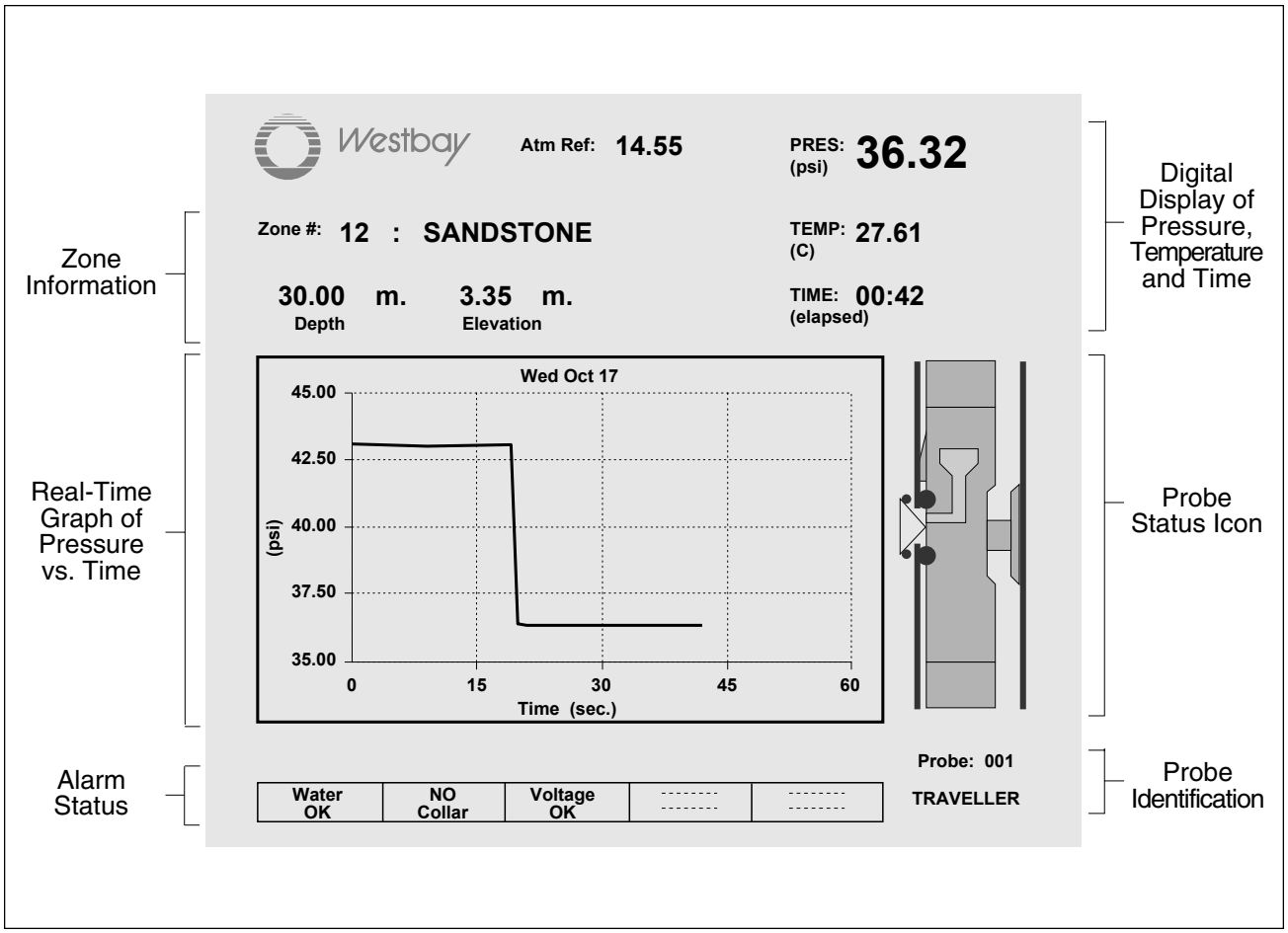


Figure 8. Data display on surface computer when using MProfile software to operate a MOSDAX pressure probe.

measuring the fluid pressure inside the MP casing (Fig. 7a). The pressure in the casing is again recorded, for quality assurance purposes.

Measuring Pressure in Low Permeability Environments

Very low permeability environments present a special challenge for measuring fluid pressures. When the routine profiling procedures described above are followed, a stable pressure may be observed through the measurement port. However, the act of opening the port may have been sufficient to change the pressure in the monitoring zone, and if the zone is very tight, that pressure change may not dissipate quickly enough to be observed. In such an environment it is always difficult to determine the validity of a static measurement unless some form of dynamic test is carried out as well. In the case of the MP System, this is done throught the use of a MOSDAX sampler probe. As illustrated in Figure 9a), the MOSDAX sampler incorporates all of the features of a pressure probe, plus a valved passage which is controlled via the surface computer. With the sampling valve closed the probe acts identically to a pressure probe and thus may be used for single-probe profiling. The difference is that once the probe is located and activated (Fig. 9b), the fluid level inside the MP casing may be adjusted to a level slightly higher or lower than the piezometric level in the monitoring zone. The sampling valve is then opened (Fig. 9c), exposing the monitoring zone to the fluid pressure in the MP casing. In very low permeability environments, no water will flow during this time. The sampling valve may be kept open for a specified period of time (such as one minute). The sampling valve is then closed (Fig. 9d) and the pressure recovery in the monitoring zone is recorded vs. time (Fig. 10). Standard analytical methods can be applied to the pressure recovery data in order to determine the apparent pressure in the monitoring zone. The same procedure can be used for testing hydraulic conductivity in low-k zones.

Pressure Monitoring Methods

The two principle methods of monitoring fluid pressure with the MP System are illustrated in Figure 11. Single probe profiling (Fig. 11a) involves an operator

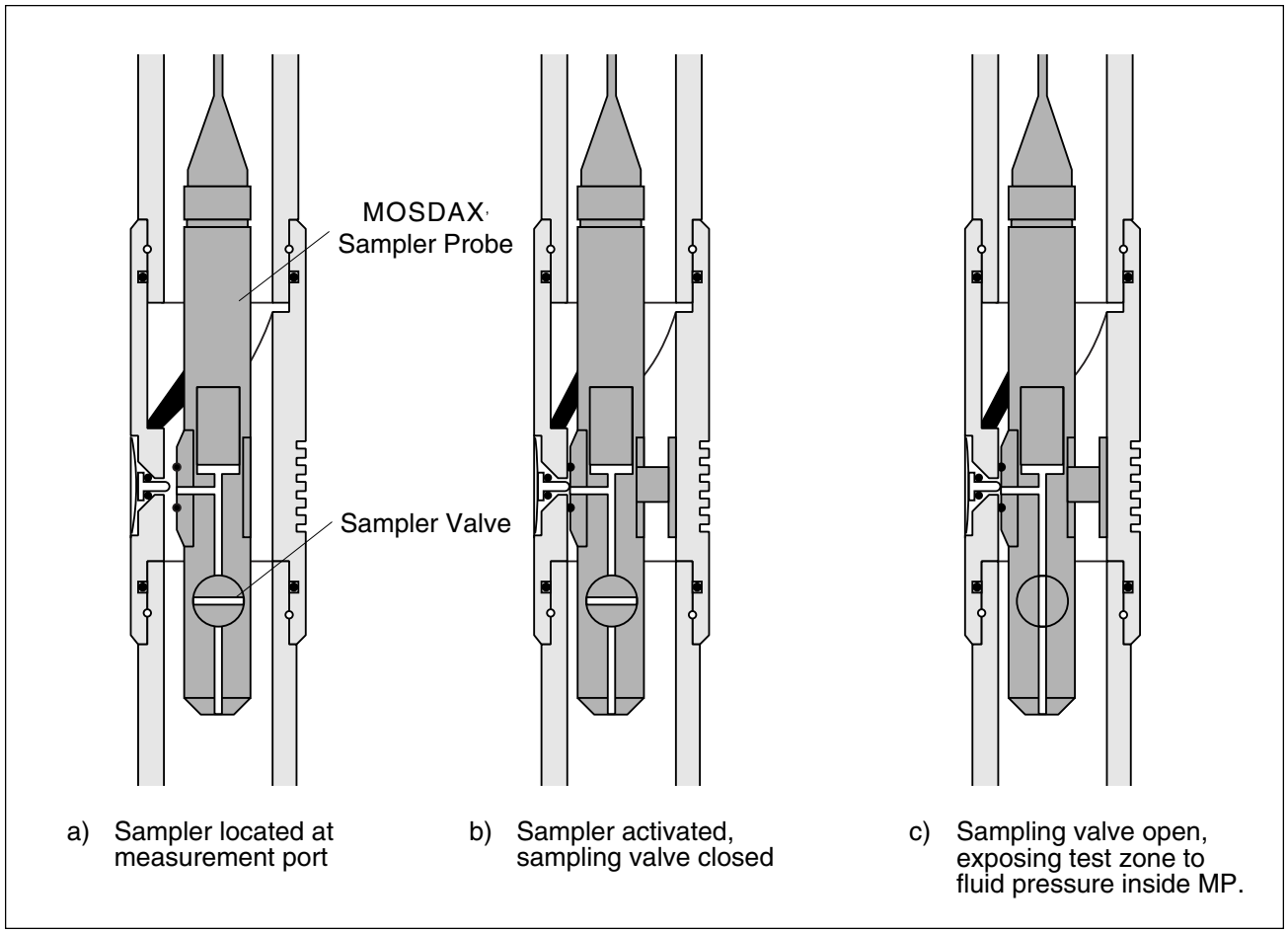


Figure 9. Using a sampler probe for testing hydraulic conductivity and verifying fluid pressure measurements in low permeability environments.

travelling to each well with a set of portable equipment including a pressure probe, cable and reel, interface and computer. The operator manually locates the probe at each measurement port and carries out fluid pressure measurements one at a time. MProfile stores the data on disk with each record tagged as to the location of the probe in the well, date, time, and probe status. Single probe profiling is generally adequate for monitoring fluid pressure up to a frequency of once per month.

When pressure measurements are desired more frequently than is reasonable for single-probe profiling, or when continual observation and recording of unanticipated events is required, the monitoring well can be configured for automated datalogging (Fig. 11b). Any or all of the measurement ports in a well may be selected for automated monitoring. Lengths of cable are made up to span the distance between each probe and the next. The string of probes and cable is assembled and lowered into the well. The datalogger and a computer are attached at the surface and the lowermost probe is located and activated in the appropriate measurement port. The

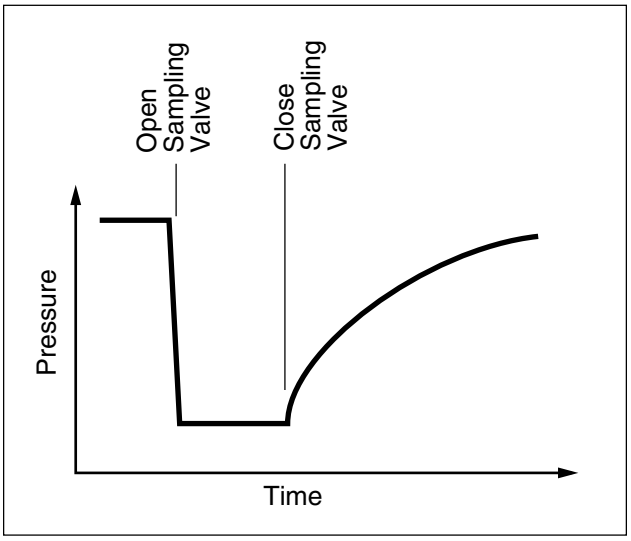


Figure 10. Typical data record from a test in a low permeability zone using sampler.

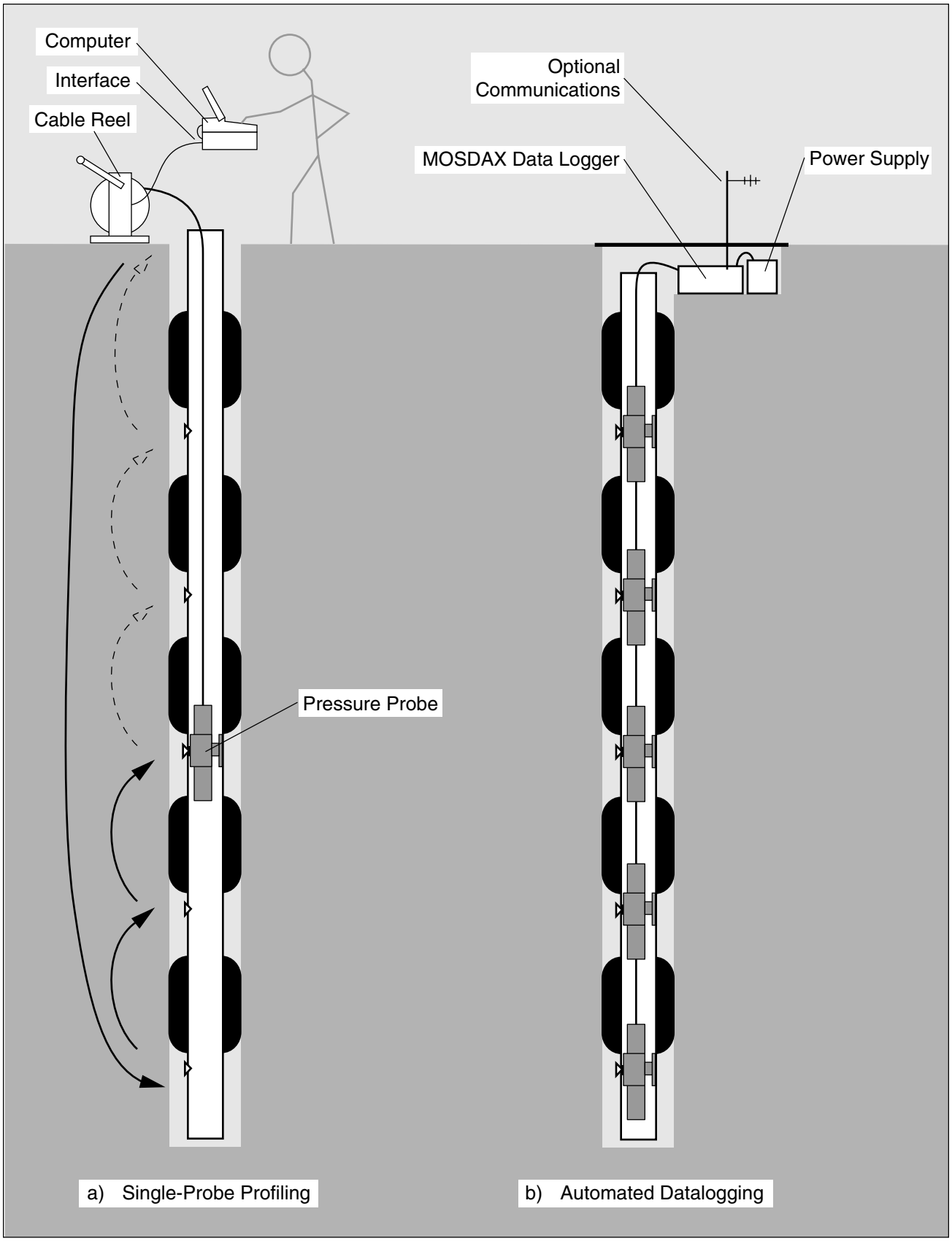


Figure 11. Methods of monitoring fluid pressure with the MP System.

remaining probes are located and activated sequentially from the bottom of the well to the top. Once all of the probes are activated, the computer is used to program the datalogger.

Recording of pressure measurements may be carried out on a simple time basis (e.g., one reading per hour or one per day), or the logger may be programmed to continually scan each probe and record pressures if a specific threshold value is exceeded. Each probe may be assigned an independent threshold (i.e., record data if probe 1's reading changes by 1 ft of water, probe 2 by 3 ft, etc.).

The datalogger may stand unattended, in which case an operator would periodically visit the site to download the stored data, or the datalogger may be connected to a telemetry system such as an RF modem, cell phone system, or landline. When connected to a communication device, a second threshold can be designated for each probe which will cause the logger to transmit an alarm signal to the host computer.

A unique aspect of monitoring in the MP System is that unusual pressure readings can often be verified by means of an in-situ calibration check. When an alarm condition is received, a natural first reaction is to question the validity of the measurement ("is it real, or is it the instrument?"). When datalogging with the MP System, if an alarm were received, the operator can log onto the well via remote communications, deactivate two or more probes including the one causing the alarm and compare their measurements of the fluid pressure within the MP casing. The column of fluid inside the MP casing is independent of all of the monitoring zones and thus serves as a reference pressure source. If the deactivated probes agree on the internal pressure, the alarm condition can be taken to be valid and the probes can be reactivated to resume monitoring. If the probe cauaing the alarm did not agree with the others, instrument error might be suspected. In such a case, an operator could visit the well, remove the string of probes, replace the offending probe and reinstall the string to resume monitoring. The offending probe could then be calibrated and serviced in a laboratory.

Fluid Sampling

Fluid samples are obtained by lowering a sampling probe and sample container to the desired measurement port coupling. As shown on Figure 12, the sampling probe operates in similar fashion to the pressure probe except that a groundwater sample is drawn through the measurement port coupling. Whenever the sampling probe is operated with the sampling valve closed, it is identical to a pressure probe, supplying the same data.

The procedure for taking a groundwater sample is as follows. A clean, empty sample container is attached to the sampling probe. The probe and container are prepared (e.g., evacuated) in a manner suited to the specific project and the sampling valve is closed to prevent the fluid inside the MP casing from entering the sample container. The probe and container are lowered to below the selected measurement port coupling. The location arm is released and the probe is positioned in the measurement port coupling (Fig. 12a). The fluid pressure inside the MP casing is recorded.

The backing shoe is activated and pushes the probe to the wall of the coupling so that the face seal on the probe seals around the measurement port valve at the same time as the face of the probe pushes the valve open. The interior passage of the probe is now hydraulically connected to water outside the coupling (Fig. 12b), but no fluid movement takes place. During this operation the change in fluid pressure is observed at the surface and may be recorded.

The sampling valve in the probe is opened, allowing fluid from outside the measurement port to flow through the probe and enter the sample container (Fig. 12c). The fluid displayed at ground surface drops and then recovers as the fluid in the container builds to formation pressure. Once the container is full, the sampling valve is closed (Fig. 12b), the backing shoe is deactivated (retracted) (Fig. 12a) and the fluid pressure inside the MP casing is once again recorded. The sampling probe and sample container are then pulled to the surface. The sampling probe can then be cleaned, a clean container attached and the procedure repeated.

When using a non-vented sample container, the fluid sample is maintained at formation pressure while the probe and container are returned to the top of the well. Once recovered, there are a variety of methods of handling the sample:

- the sample may be depressurized and decanted into alternate containers for storage and transport,
- the sample container may be sealed and transported to a laboratory with the fluid maintained at formation pressure,
- the sample may be transferred under pressure into alternate pressure containers for storage and transport.

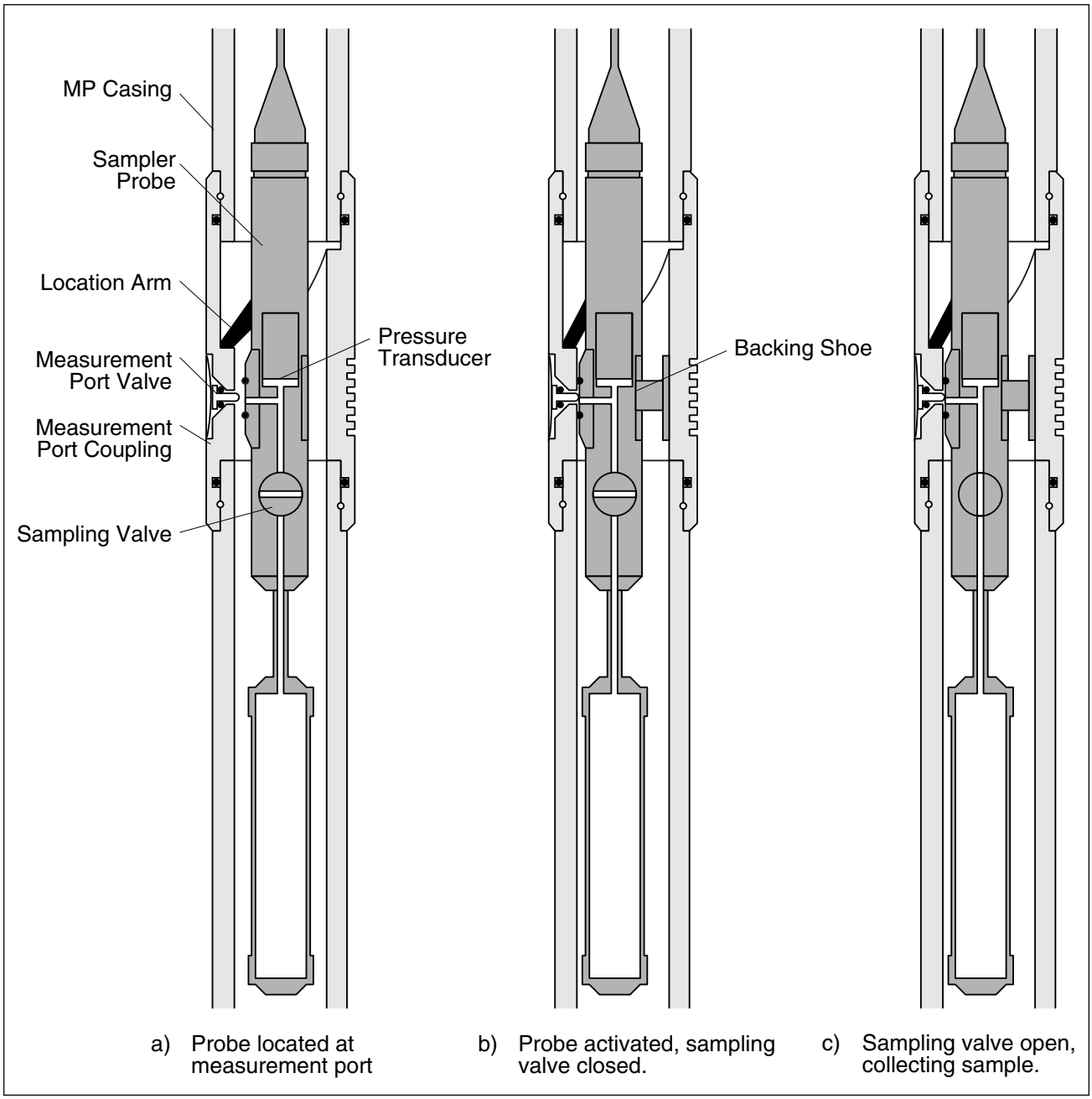


Figure 12. Operation of a sampler probe.

The advantages of this discrete sampling method can be summarized as follows:

1) The sample is drawn directly from formation fluids outside the measurement port. Therefore, there is no need for pumping a number of well volumes prior to each sampling event. Because there is no pumping prior to sampling, the sample is obtained with minimal distortion of the natural groundwater flow regime, the storage and disposal of large volumes of hazardous purge fluids is eliminated, and operator exposure to hazardous fluids is reduced.
2) The lack of pumping means samples can be obtained quickly, even in relatively low permeability environments.
3) The sample travels a short distance into the sample container, typically from 1 to 2 ft (0.3 to 0.6 m), regardless of depth.
4) The risk and cost of storing and disposing of hazardous purge fluids is virtually eliminated.

Hydraulic Conductivity Testing

A variety of different test methods can be employed to measure the hydraulic conductivity of formation materials with the MP System. These include variable head, constant head and pressure-pulse tests.

Variable head tests are the single well test method most commonly used with the MP System. Using these types of tests in the MP System, hydraulic conductivities between 10^{-2} and 10^{-8} cm / sec can be measured.

For variable head tests the valved pumping port couplings are used to provide the hydraulic connection between the interior of the MP riser tube and the test zone. In cases where monitoring zones are to be purged, it is convenient to carry out hydraulic conductivity testing just prior to or following purging. The head (fluid level) inside the MP casing can be adjusted while all port valves are closed, then the selected pumping port can be opened in a controlled manner (pumping port operation is described in the discussion of purging). This allows accurate measurement of both the initial head change and the time at which the head change is applied (t_0). The pumping port valve is opened rapidly (in less than one second), which satisfies the theoretical requirement that an instantaneous head change be applied to the tested zone.

For rising head tests the water level inside the MP casing is bailed or pumped down to a pre-determined level below the static water level in the test zone. For falling head tests the water level is raised to a level above the static water level in the zone to be tested. Measurement equipment is set in place and the pumping port valve is opened. Recovery of the water level in the MP casing is measured and recorded vs. time. A water level tape or pressure transducer is commonly used to record the water level changes. Figure 13 shows a typical record of water levels during a rising head hydraulic conductivity test.

Slug tests are carried out by opening the pumping port coupling at the zone to be tested and allowing the water level in the MP casing to equilibrate to the static water level for that zone with measurement equipment in place. The initial head change is then applied by rapidly lowering a displacement slug (a length of solid rod or sealed pipe) into the water. The recovery of the water level is measured and recorded vs. time. The slug test can be repeated and recorded again when the slug is removed from the water. Figure 14 shows a typical record of water levels during a slug test of hydraulic conductivity.

Data from variable head hydraulic conductivity tests may be analysed using any preferred calculation method. The most commonly used methods are those of Hvorslev (1951), Cooper et al. (1967) and Bouwer and Rice (1976). Selection of these or any other analytical method should be based upon an assessment of how well the test conditions comply with the simplifying assumptions inherent in the analytical method.

In very low permeability environments (hydraulic conductivity less than 10^{-7} or 10^{-8} cm / sec) the formation fluid pressure can be changed with very little fluid movement. As a result, tests can be carried out through the measurement port valve rather than the pumping port valve. Using a sampler probe with a transducer the zone to be tested may be exposed to the fluid pressure inside the MP casing for a period of time (see Fig. 9 and discussion of measuring fluid pressure in low-k environments). The zone may then be shut-in and the recovery of fluid pressure over time measured and recorded. Figure 10 shows a data record from such a test.

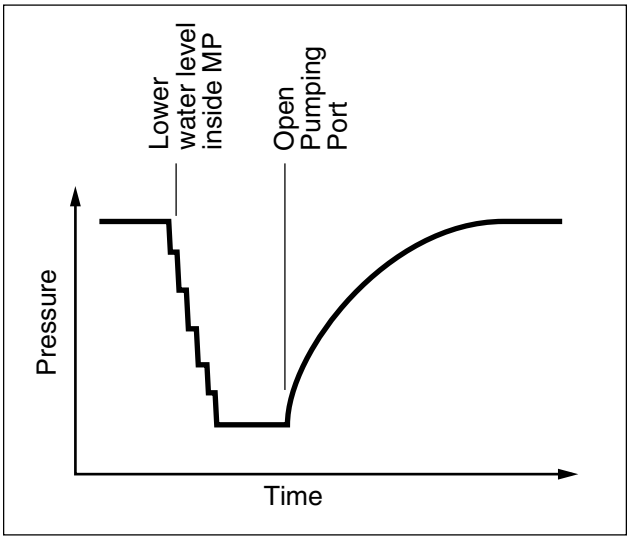


Figure 13. Typical data record from a rising head test.

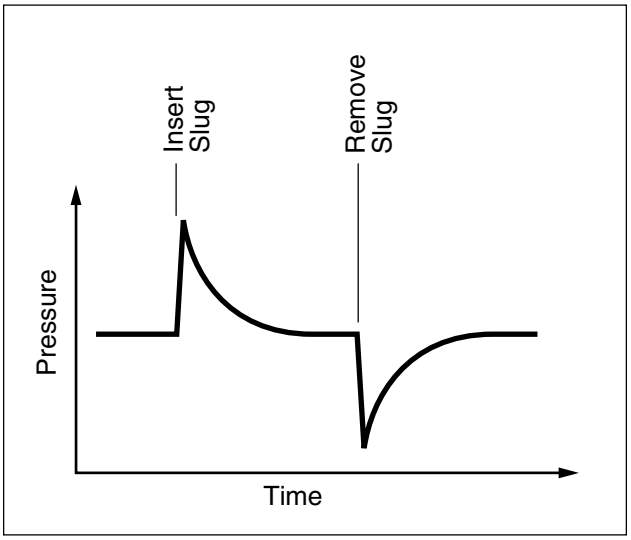


Figure 14. Typical data record from a slug test.

Field Quality Control

There are two distinctive parts to any quality assurance program. The first involves manufacturing and testing procedures which avoid the production or installation of equipment that may result in the collection of erroneous data. The second involves field operational procedures which will ensure that erroneous data are not generated as a result of the failure of any component to function as intended. Although the first part is necessary to allow the installation of useful monitoring wells, the second must also be rigorously applied to identify sources of erroneous and misleading results.

The MP System has many unique features for field quality control which clearly separate it from other types of groundwater monitoring instrumentation. These features are the result of designing components in response to the stringent requirements of users in the fields of nuclear and hazardous waste management.

Quality control tests are carried out at various points during the field use of the MP System and tend to be grouped into three periods: during installation, following installation, and during routine monitoring.

During Installation

During installation of the MP System the following operations form part of the quality control procedures:

Drill core or cuttings and geophysical logs are carefully checked to see that monitoring zones and annular seals are placed at the optimum positions. In cased wells, the well casing is inspected to verify that the interior surfaces are suitable for establishing good quality packer seals and backfill is placed under carefully controlled conditions with frequent measurements of material depths.

Westbay casing components are carefully inspected to see that critical surfaces are undamaged, sealing O-rings are clean and in place, and components are correctly oriented. Serial numbers are recorded along with component position in the installation. These operations link the field quality control to production test results.

As each section of MP casing is attached, the connection is pressurized with water and observed for any signs of leakage. Test results are recorded on the installation log.

During inflation of each MP packer, incremental volumes and pressures are recorded and plotted. These data allow an evaluation of borehole conditions and provide the first indication of the quality of the annular seal obtained.

Following Installation

Immediately following installation further checks are carried out to verify the operation of the system. These include the initial pressure profile which serves to confirm the operation of the inlet valves of the measurement port couplings. Observed head differences across exterior casing seals directly indicate the seal effectiveness. Where such head differences are not observed, the annular seals can be artificially stressed by opening a pumping port in one monitoring zone and withdrawing or adding a slug of water from inside the casing while using the pressure probe to observe the pressure response in the monitoring zone on the other side of the seal. In cased wells and wells in low permeability environments, stresses can be applied through measurement ports in order to evaluate seal integrity.

Additional measurement ports are routinely installed between monitoring zones, further enhancing the ability to carry out thorough quality control tests.

Fluid can be added to packers at any time following installation and the pressure at which further fluid injection occurs can be compared with the injection pressures recorded during the initial inflation.

During Routine Monitoring

A number of quality control checks are built into the routine monitoring procedures.

When measuring fluid pressures, the pressures measured inside the MP casing at each measurement port are recorded immediately before and after the measurement made through the port. These inside casing values serve a number of purposes: 1) comparison of the two values confirms that the transducer was operating the same way after the reading as before, 2) comparison of the inside values from one set of measurements to the next confirms transducer stability over the intervening time period (assuming the water level inside the casing is the same), and 3) if the head of fluid inside the MP casing is known, an in-situ calibration check of head of water versus transducer output is obtained. Any unacceptable changes which show up during monitoring can be checked and corrected through laboratory calibration of the instrument.

Water sampling procedures with the MP System improve quality control because: 1) the short flow path between the formation and the container greatly reduces the surface area contacted by the sample, 2) the contacts between the water sample and the atmosphere are eliminated, 3) observing and recording the water level inside the MP casing during sampling confirms that the sample obtained is from outside the casing, and 4) sampling without purging reduces the disturbance of the

natural system, minimizing unnatural changes in chemistry. Sampling methods can be varied to compare the effects of atmospheric contact versus no atmospheric contact and maintaining the sample under pressure versus allowing depressurization of the sample.

During water sampling, sample blanks and spikes may be collected using identical procedures for sampling, preservation, handling and shipping. Travel blanks and spikes may also be collected using identical procedures for handling, preservation and shipping. The chemical analyses of samples obtained using the MP System may be compared with those of samples collected from the same zone by alternate means.

Finally, the pumping port may be reopened should further purging appear to be desirable.

For both fluid pressure and water quality data, the MP System can provide corroborative data. That is, a high density of data can be obtained in a single installation so that significant changes in piezometric pressure and / or water quality can appear as transitions along a depth profile. Thus, neighboring values will corroborate one another rather than indicating abrupt changes which would cause one to question anomalous values.

Serviceability

In the event that quality control testing should reveal a component which is not operating properly, various steps can be taken to remedy the problem including, if certain cases, removing the MP casing string, replacing faulty components and reinstalling the string.

Table 2. Summary of major quality control aspects of the MP System.

Provides the Ability to Verify	
Well Integrity	✓
Individual Seals	✓
Sample Origin	✓
Fluid Pressures	✓
Well is Serviceable	✓

Summary

The modular nature of the MP System permits a large number of monitoring zones to be accessed through valves placed inside a single closed tube or casing installed in a single borehole. Such a monitoring system can provide a detailed view of the variation of piezometric pressure and water quality with depth. The valved couplings permit purging of the well following installation and allow all standard hydrogeologic tests to be carried out in each zone. Routine sampling is carried out without repeated purging, eliminating the need to store and dispose of large volumes of purge fluid and reducing operator exposure to hazardous fluids. The valves also permit an evaluation of the condition of exterior casing seals at any time after installation. Casing packers allow multiple seals to be established easily and quickly, providing the required hydraulic isolation of each monitoring zone. The modular design of the downhole components means the number and location of monitoring zones and seals can be modified on the basis of the best information available in the field at the time of installation. The exact depth of monitoring zones need not be known when equipment is purchased.

Field quality control procedures have been established which permit the quality of a well installation and the proper operation of testing and sampling procedures and equipment to be routinely verified. Thus, groundwater data and the additional data required to define the quality of the field data can be routinely collected. Furthermore, when a high density of groundwater monitoring zones are installed by using multi-level monitoring wells, the redundant monitoring points can provide important corroborative field data to an extent which is not available with single level monitoring wells. The result is a monitoring system which provides data with a degree of defensibility unattainable with any other monitoring method, single or multi-level.

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Appendix C:
Carbon Trap Documentation

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C.1 CARBON TRAP INSTALLATION

Carbon traps are relatively easily installed, minimally invasive, and can be deployed at multiple locations to identify areas of high activity and obtain a site-wide estimate of the light non-aqueous-phase liquid (LNAPL) loss rate. Since the carbon traps measure flux of carbon dioxide (CO_2) released from the soil, the traps need to be installed in the ground via a receiver pipe with an airtight seal (see the *E-Flux Receiver Pipe Installation Guide Standard Operating Procedure* [SOP] included in this appendix).

Up to three carbon traps will be installed below the concrete subgrade (into bedrock) of the lower access tunnel floor near Tank 5. These will be installed using the "At Grade" installation method (see SOP), where the carbon trap will protected by a traffic-rated 10- to 12-inch diameter housing set in the tunnel floor. Concrete coring or similar method will be used to cut a hole in the concrete tunnel floor to accommodate the traffic housing. A hole approximately 10 inches deep or deep enough to accommodate the carbon trap will be excavated below the grade of the concrete tunnel floor. The receiver pipe will be set in contact with the underlying bedrock, backfilled with sand and gravel, and then secured with concrete after backfilling.

Four carbon traps will be installed above ground in the greater vicinity of Tank 5, based on surface geologic conditions. These will be installed using the "Direct Push Method" installation method (see SOP), which involves hammering a beveled receiver pipe approximately 2 inches into the soil. This method is good for temporary monitoring in soils that, once disturbed, might not be recompacted back to their original condition. Soil disturbance is minimized with this method, reducing the possibility of interfering with soil gas transport by receiver pipe installation.

This method offers minimal stability to the receiver pipe, so the seal achieved is adequate for only a short term (no more than four sampling events). A rubber connector with hose clamps is used to attach the receiver pipe to the carbon trap so that disturbance to the seal between the receiver pipe and soil is minimized. A knife can be used to cut a ring into the soil that the beveled receiver is placed into. This groove eases installation and can help create a deeper seal without completely digging out and disturbing the soil.

General installation and placement guidelines are:

- Trap locations are recommended to be placed near existing groundwater monitoring wells. This is important for data discussion and potential correlation of CO_2 fluxes to known geologic, hydrogeologic, and hydrocarbon distribution conditions.
- Suitable trap locations require soils that are permeable to gas transport. Pavement or low-permeability surface covers (including free standing water, ice, or extremely compacted soils) should be avoided. In addition, areas close to impermeable surfaces should be avoided due to associated changes in gas transport pathways.
- Excess surface vegetation should be cleared from directly beneath the proposed trap location prior to installation of the in-ground receiver.
- Several background locations (unimpacted) should be chosen where soils, vegetation, and general site conditions are similar to the LNAPL monitoring locations.
- Additional site information useful for data interpretation includes:

- Collection of groundwater temperatures, if feasible. Due to the exothermic nature of biodegradation, in some cases groundwater temperatures have correlated to biodegradation rates estimated based on soil gas fluxes (McCoy 2012).
- Groundwater and LNAPL levels in wells (if applicable).
- Occurrence and extent of vadose zone impacts.

Additional installation guidelines to be considered during high wind conditions include:

- Consult weather data and estimate the bulk wind velocity (at a height of 10 meters) and, using the power law, estimate the velocity applicable to the traps (currently 5.5 inches tall).
- Estimate the bias (see Application Note 1504.2: *Wind Effects on Soil Gas Flux Measurements at Ground Level* [Zimbron 2015]) that such winds could generate.
- If the bias results in unacceptable data quality, consider modifying the design to achieve a lower profile (i.e., installation closer to ground cover or even with finish at grade). Alternatively, reconsider the deployment period to avoid unusual high wind periods.
- Record winds during the actual period of deployment. This can be done from the nearest weather station or by actually measuring it at the site (after the appropriate correction using the power law to estimate the wind velocity at the height of the trap top).
- Using the recorded wind velocity at the trap height, estimate the potential bias of the measurement and consider correcting the measurement.

C.2 LNAPL LOSS RATE ANALYSIS USING CARBON TRAPS

The carbon trap is a passive adsorption device that collects CO_2 emitted from the subsurface. E-Flux is a private company that leases the traps to users and also analyzes the data collected. The traps are deployed within shallow monitoring points (up to 6 inches below grade) and are a direct method to collect CO_2 released from the subsurface.

The carbon trap captures soil CO_2 coming out of the ground with CO_2 sorbent (E-Flux 2015). The sorbent is made of proprietary non-hazardous material consisting of a mixture of calcium and sodium hydroxides (strong bases). The top of the trap is open to the atmosphere so that gas flow is not disturbed. The trap contains two layers of sorbent; the top layer traps ambient CO_2 while the bottom layer catches CO_2 originating from the soil. Each sample will be assigned a unique identifier (see *SAP Addendum 01*, Table 3-5) and sent to E-Flux for analysis.

The sorbent is recovered from the trap housing, dried, and homogenized. The processed sorbent is then shipped to a third party lab for carbonate and fossil fuel carbon content analysis. E-Flux uses industry accepted practices and methodologies, including Quality Assurance and Quality Control protocols. In combination with proprietary technology, the analyses are based on ASTM International methods:

- D4373-02, *Standard Test Method for Rapid Determination of Carbonate Content of Soils*
- D6866-12, *Standard Test Methods for Determining the Biobased Content of Solid, Liquid, and Gaseous Samples Using Radiocarbon Analysis*.

Using travel-blank corrected CO_2 fluxes and assumptions about the LNAPL properties (carbon content and density), E-Flux then estimates LNAPL loss rates, as gallons per acreper year equivalents.

Additionally, radiocarbon analysis (^{14}C) will be used to differentiate anthropogenic (due to fossil fuel-burning) and natural sources of atmospheric carbon monoxide (CO), CO_2, and methane (see *Basis for ^{14}C Analysis*; Zimbron 2014). Contemporary (modern) organic carbon is ^{14}C-rich, while fossil fuel carbon is ^{14}C-depleted. The ^{14}C-based technique offers a built-in correction for fossil fuel as an alternative to the background correction method (see Sihota and Mayer 2012).

The bottom sorbent sample in each E-Flux carbon trap can undergo radiocarbon (^{14}C) dating in order to determine the amount of fossil fuel derived carbon in the sample. Total CO_2 flux captured by the trap represents a two-source model, with CO_2 generated from natural soil organic matter (modern carbon) and LNAPL (fossil fuel carbon).

C.3 REFERENCES

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E-FLUX

SOP

STANDARD OPERATING PROCEDURE:
Receiver Pipe Installation Guide

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INTRODUCTION

The following document describes protocols for installation of receiver pipes for E-Flux CO_2 traps. CO_2 traps are installed to evaluate rates of natural attenuation of light non-aqueous phase liquid hydrocarbons (LNAPL) in soil. Since the CO_2 traps measure flux of CO_2 coming out of the soil surface, it is necessary to install them in the ground via a receiver pipe with a secure seal. This document presents several alternatives for receiver pipes and describes procedures for installation of each one. A list of tools required for each method of installation is provided. Upon review of this document, the user will be able to choose the installation method that is best suited for the site.

GENERAL PLACEMENT GUIDELINES

- It is recommended that trap locations are near existing groundwater monitoring wells. This is important for data discussion and potential correlation of CO_2 fluxes to known geologic, hydrogeologic, and hydrocarbon distribution conditions.
- Suitable trap locations require soils that are permeable to gas transport. Pavement or low permeability surface covers (including free standing water, ice, or extremely compacted soils) should be avoided.
- In some sites, variability within close locations due to soil heterogeneity can be large. If testing for variability, replicate traps should be located within 10 feet of each other.
- Excess surface vegetation should be cleared from directly beneath the proposed trap location prior to installation of the in-ground receiver.
- If desired, background locations (unimpacted) should be chosen where soils, vegetation, and general site conditions are similar to the LNAPL monitoring locations.
- Additional site data that might be useful for data discussion includes:

    1. Groundwater temperatures. Due to the exotherminc nature of biodegradation, in some cases groundwater temperatures have correlated to biodegradation rates estimated based on soil gas fluxes (McCoy, et al, 2014).
    2. Soil gas concentration profiles at discrete vadose zone locations or in well headspace (Wilson et al, 2013) might reveal high CO_2 and/or methane concentrations and thus be indicative of areas of high rates of LNAPL degradation.
    3. Groundwater and LNAPL levels in wells (if applicable).

GENERAL NOTES

- Keep traps upright.
- Typical deployment period is 2 weeks.
- If traps deployed for ~1 week and rain, pull out.
- Avoid saturated soil (w/standing water).
- Traps are moisture resistant but, not water proof.
- Sample preservation not needed.
- Shipping next day optional.
- Travel blank handling.

INSTALLATION METHODS

Several different methods for receiver pipe installation are available. Choosing a method depends on characteristics of soil present at the site, the desired installation period, and the propensity for trap disturbance (i.e. a cow kicking the trap over). Table 1 gives an overview of each method.

Table 1: Installation Methods

Option	Receiver Depth	Seal Strength	Advantages	Disadvantages	When to Install
Direct Push	2 in	Medium	• easier /quicker installation • minimal soil disturbance	• might lose the seal if disturbed • might require reinstallation with each trap deployment	• softer soils (few cobbles) • shorter installation periods (no more than 4 rounds of samples)
8" Collar + Reducer	2 in	Medium	• larger sampling surface area • minimal soil disturbance • Larger area/perimeter ratio (less prone to leaks)	• requires more materials for installation • might lose the seal if disturbed • might require re-installation with each trap deployment	• sites with previously installed chamber collars • for comparison with chamber methods • when a larger sampling area is desired • soils with cobbles (~ 1 in)
At Grade	10 in (housing + trap depth)	High	• placement underground allows for minimal environmental and/or human disturbance • minimal visual impact	• longer installation time • complex installation procedure • disturbs soil	• sites where trap disturbance is a concern (i.e. areas with human traffic) • long installation periods (more than 4 rounds of samples)
Hole + Backfill	7 in	High	• deep secure lasting seal	• longer installation time • requires careful recompaction to minimize soil disturbance	• soils with sand or gravel • long installation periods (more than 4 rounds of samples)
Concrete Ring	7 in	High	• long lasting secure seal • permanent sampling port	• longer installation time • disturbs the soil	• soils with sand or gravel • long installation periods (more than 4 rounds of samples)

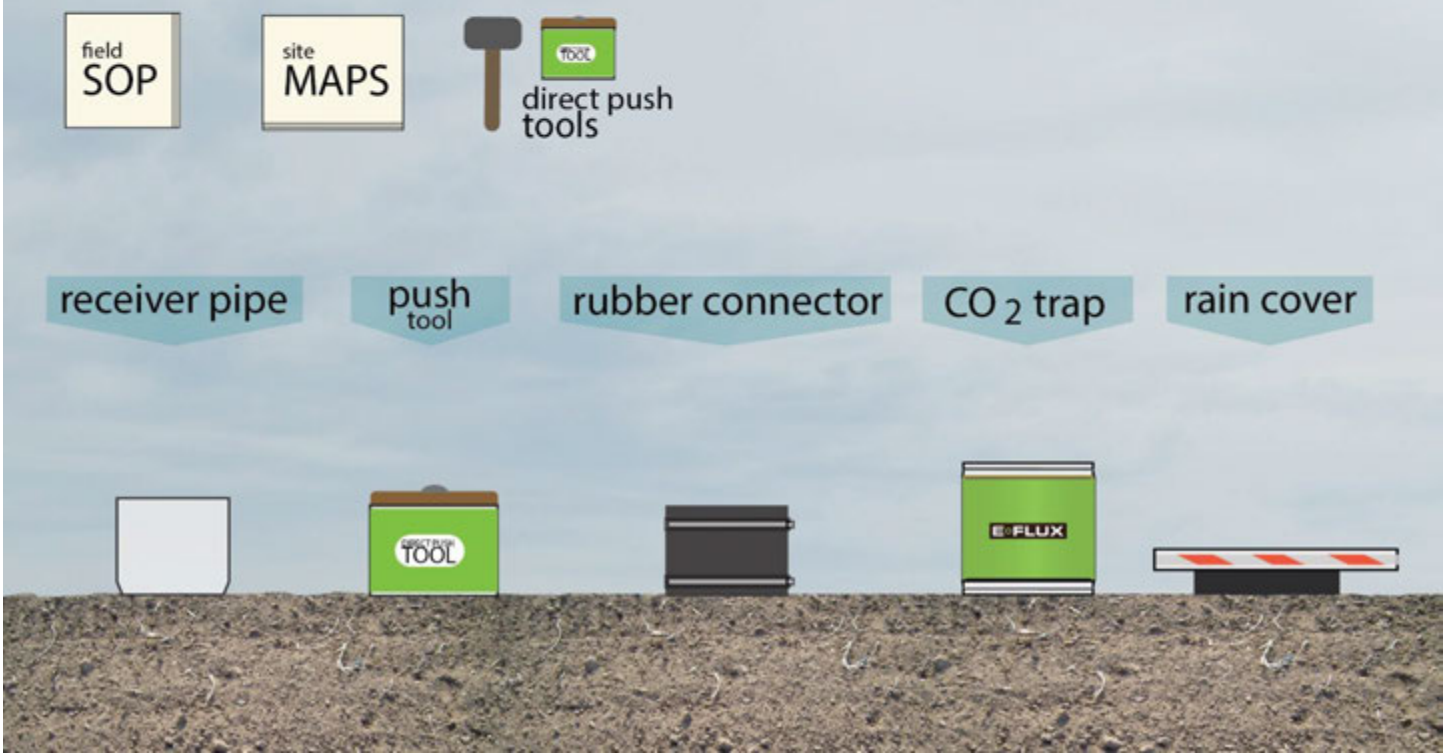
DIRECT PUSH METHOD

The direct push method involves hammering a beveled receiver pipe about 2 inches into the soil. This method is good for temporary monitoring in soils that once disturbed might not be recompacted back to their original condition. Soil disturbance is minimized with this method, reducing the possibility of interfering with soil gas transport by receiver pipe installation.

This method offers minimal stability to the receiver pipe, so the seal achieved is only adequate for a short term (no more than 4 sampling events). A rubber connector with hose clamps is used to attach the receiver pipe to the CO_2 trap so that disturbance to the seal between the receiver pipe and soil is minimized. A knife can be used to cut a ring into the soil that the beveled receiver is placed into. This groove eases installation and can help create a deeper seal without completely digging out and disturbing the soil.

Option	Depth	Seal Strength	Advantages	Disadvantages	When to Install
Direct Push	2 in	Medium	• easier /quicker installation • minimal soil disturbance	• might lose the seal if disturbed • might require reinstallation with each trap deployment	• softer soils (few cobbles) • shorter installation periods (no more than 4 rounds of samples)

EQUIPMENT



PROCEDURE

Cut a ring with the same diameter as the receiver pipe in the soil with a knife for easier and deeper installation (optional).

Place the beveled side of the receiver pipe vertically on the ground.

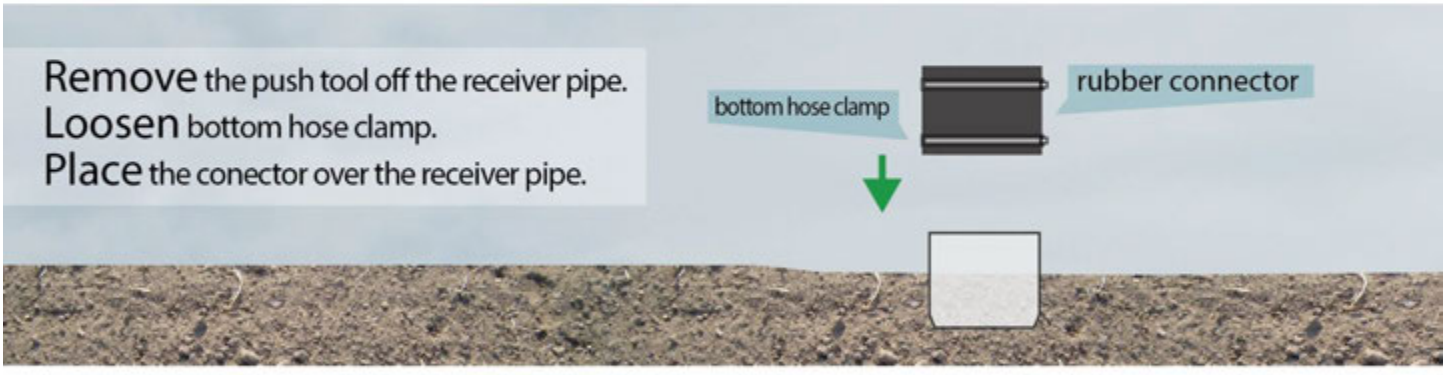
Place the push tool on top of the receiver.

Hammer into the ground with the rubber mallet or slide hammer. Keep the receiver vertical.

Remove the push tool off the receiver pipe.

Loosen bottom hose clamp.

Place the conector over the receiver pipe.



Add-on Security.

go to page 17 to see add-ons for this method.

Trap installation.

go to page 15 to see trap installation guide.

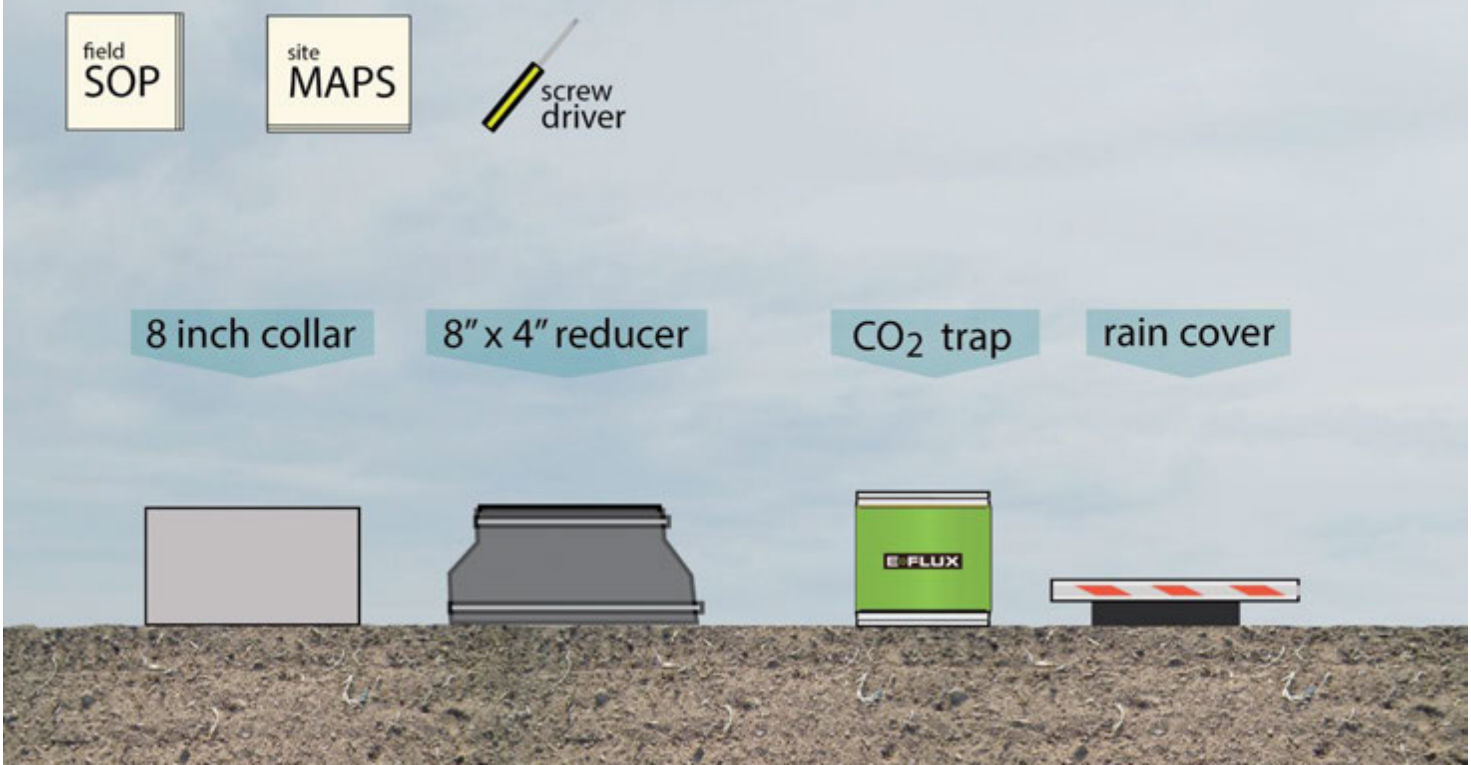
8” COLLAR METHOD

The 8” collar method is an adaptation for sites that already have 8” collars installed for use with the chamber method. Installation involves mounting the reducer between the collar in the soil and the CO_2 trap. This method is quick if collars are already installed in the soil.

This method allows the trap to collect gas over a large sampling area. 20 cm (8 inch) collars can be installed with the push method if a larger sampling footprint is desired.

Option	Depth	Seal Strength	Advantages	Disadvantages	When to Install
8” Collar + Reducer	2 in	Medium	• larger sampling surface area • minimal soil disturbance • Larger area/perimeter ratio (less prone to leaks)	• requires more materials for installation • might lose the seal if disturbed • might require re-installation with each trap deployment	• sites with previously installed chamber collars • for comparison with chamber methods • when a larger sampling area is desired • soils with cobbles (~ 1 in)

EQUIPMENT

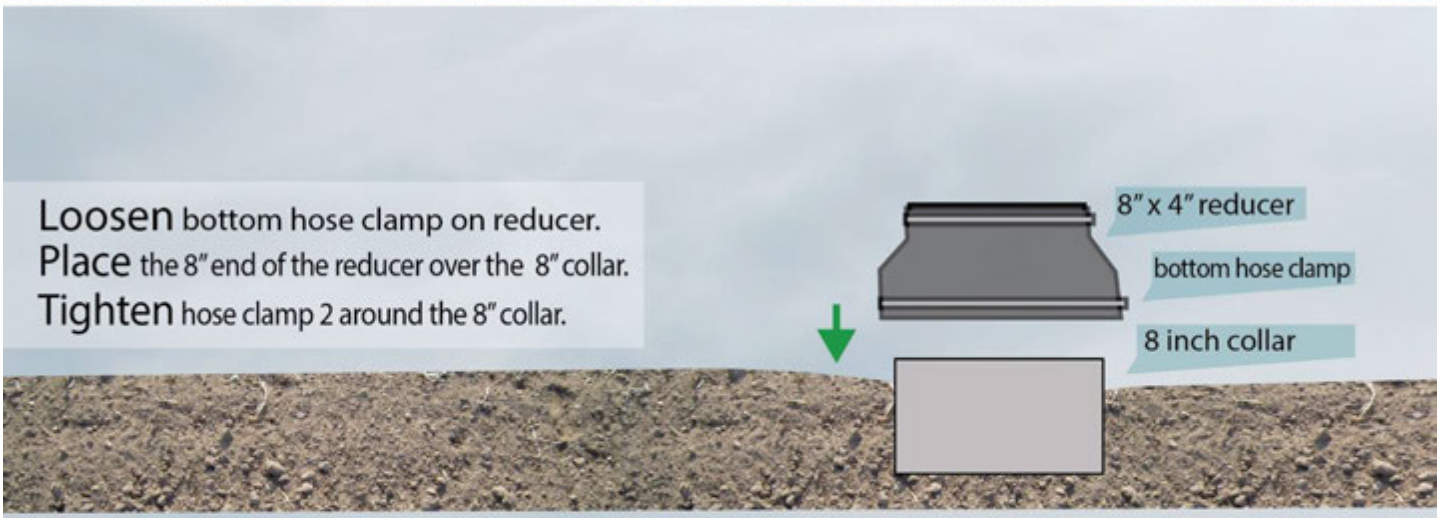


PROCEDURE

Locate the already installed 8″ collars

If 8″ collars have not been installed
go to direct push method on page
to see installation guide.

Loosen bottom hose clamp on reducer.
Place the 8″ end of the reducer over the 8″ collar.
Tighten hose clamp 2 around the 8″ collar.



Add-on Security.
go to page 17 to see add-ons for this method.

Ready for trap installation.
go to page 15 to see trap installation guide.

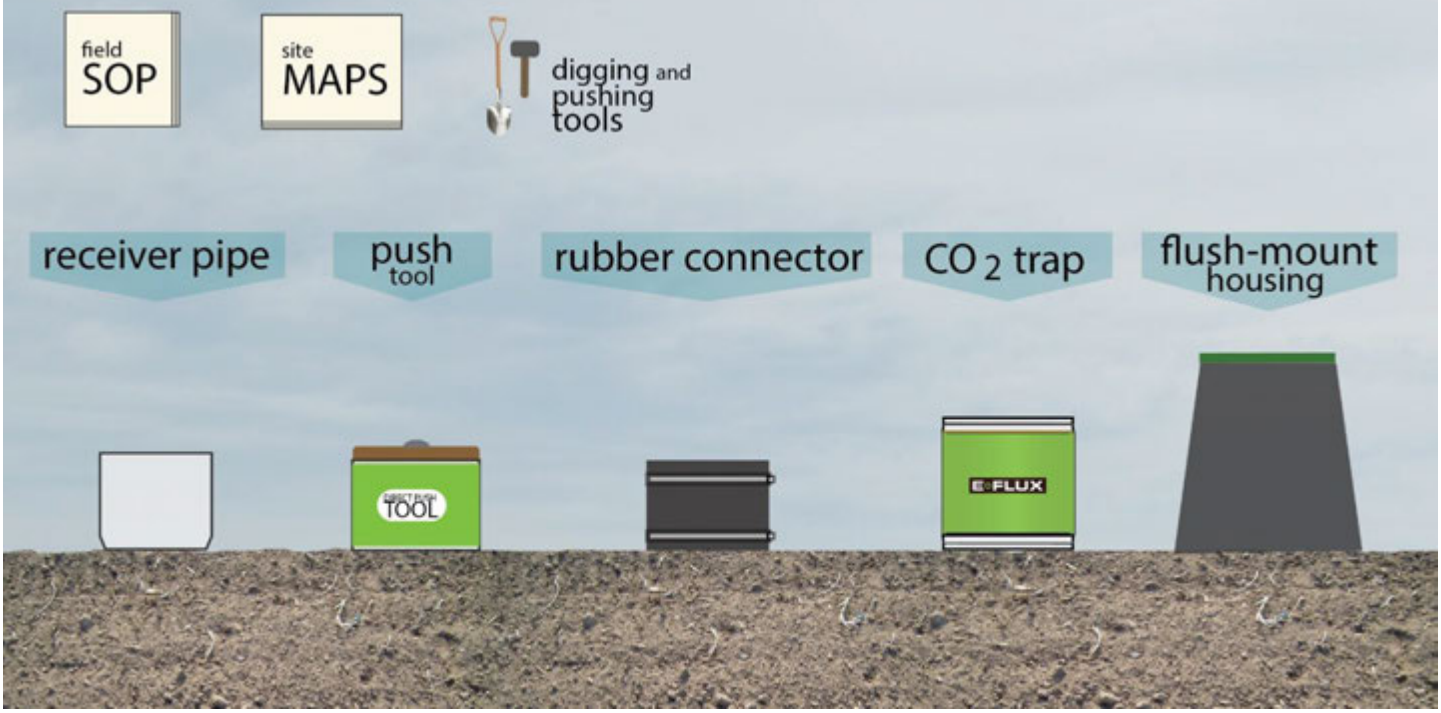
AT GRADE METHOD

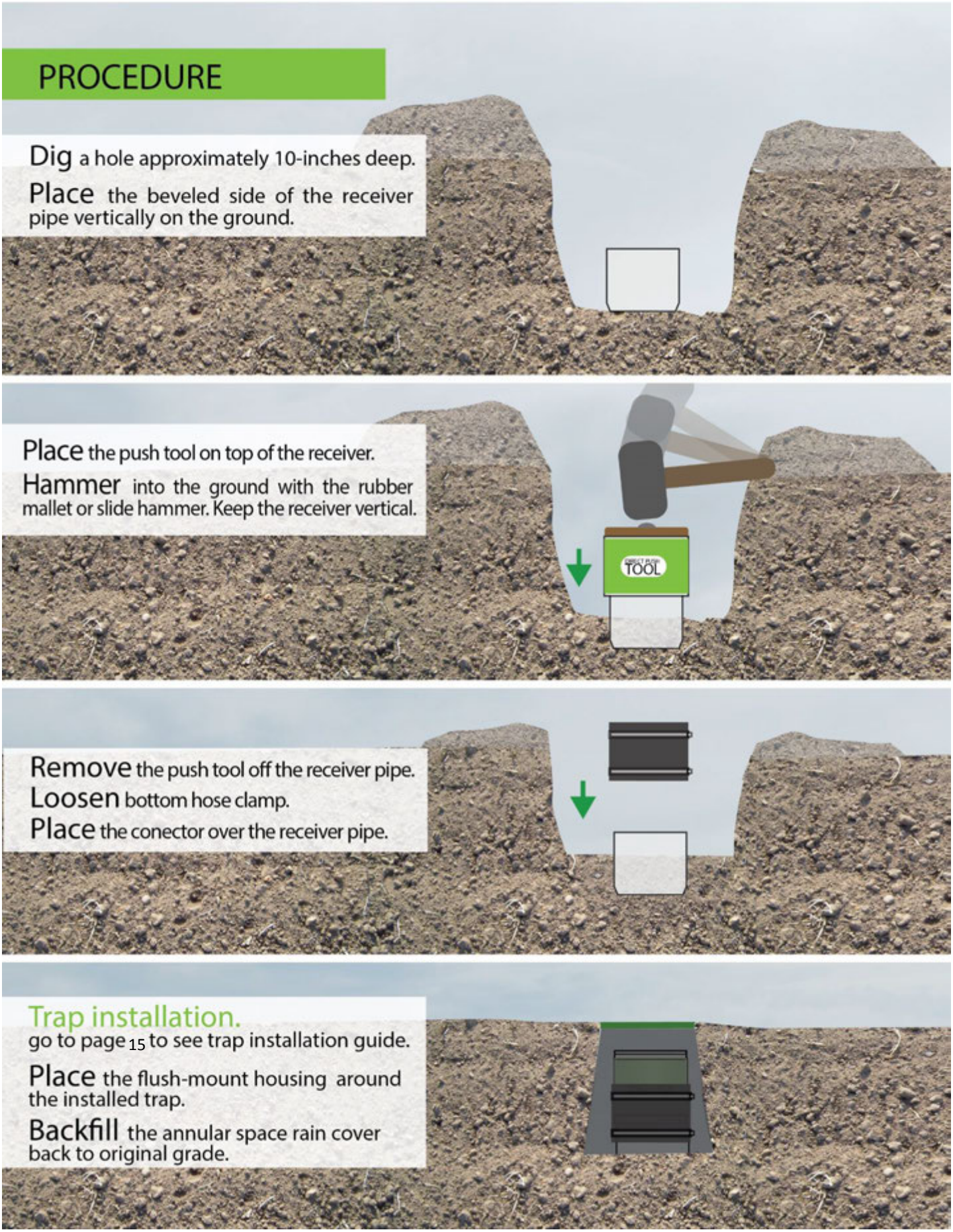
At grade installation is for situations where CO_2 trap disturbance is a primary concern. For this method a large hole is dug so that the receiver pipe is put in the ground at a depth of 11 inches. Rain cover is placed over the top of the trap and backfilled so that the trap is essentially underground. A perforated lid is placed over the top of the housing so that the trap cannot be tampered with.

This installation method is preferable only for sites that might experience significant trap disturbance (such as places open to pedestrian traffic). This method can result in soil disturbance.

Option	Depth	Seal Strength	Advantages	Disadvantages	When to Install
At Grade	10 in (housing + trap depth)	High	• placement underground allows for minimal environmental and/or human disturbance • minimal visual impact	• longer installation time • complex installation procedure • disturbs soil	• sites where trap disturbance is a concern (i.e. areas with human traffic) • long installation periods (more than 4 rounds of samples)

EQUIPMENT





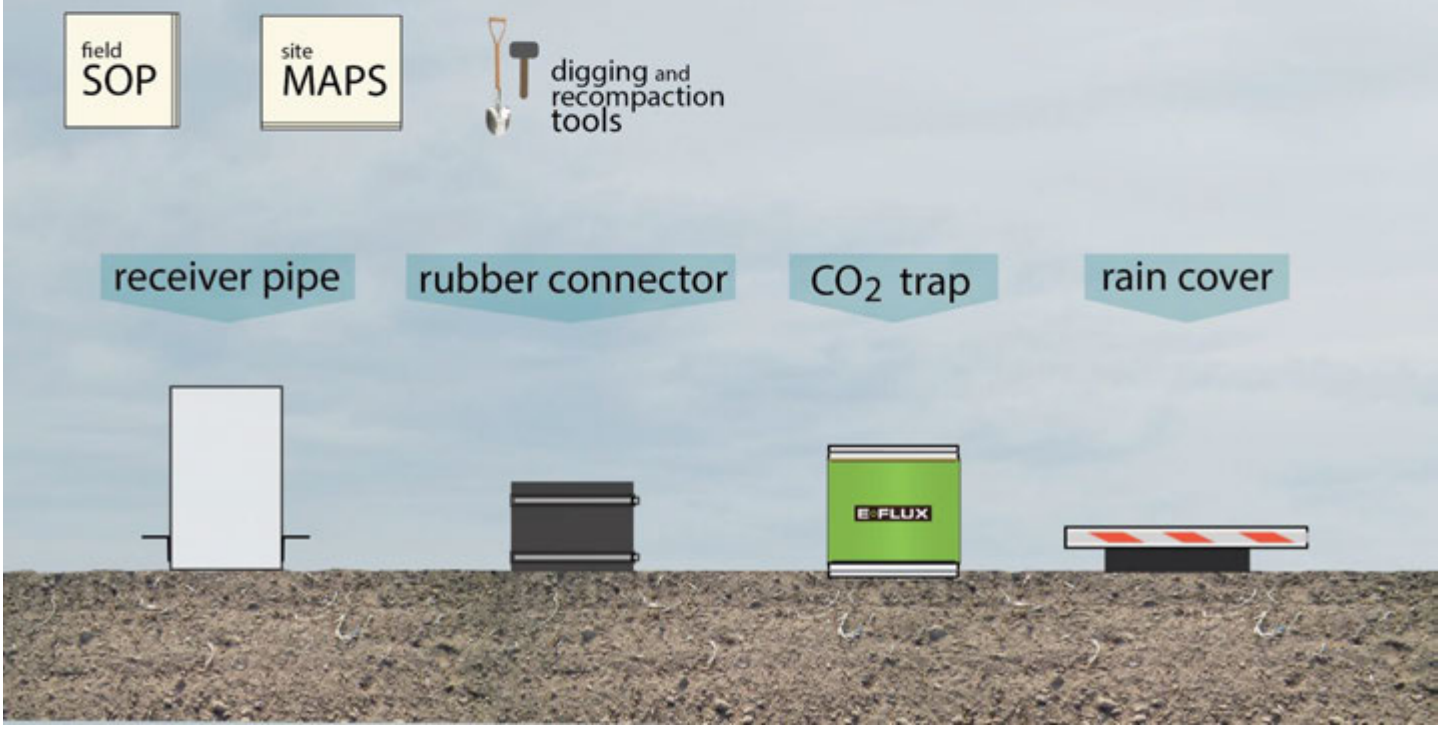
HOLE + BACKFILL METHOD

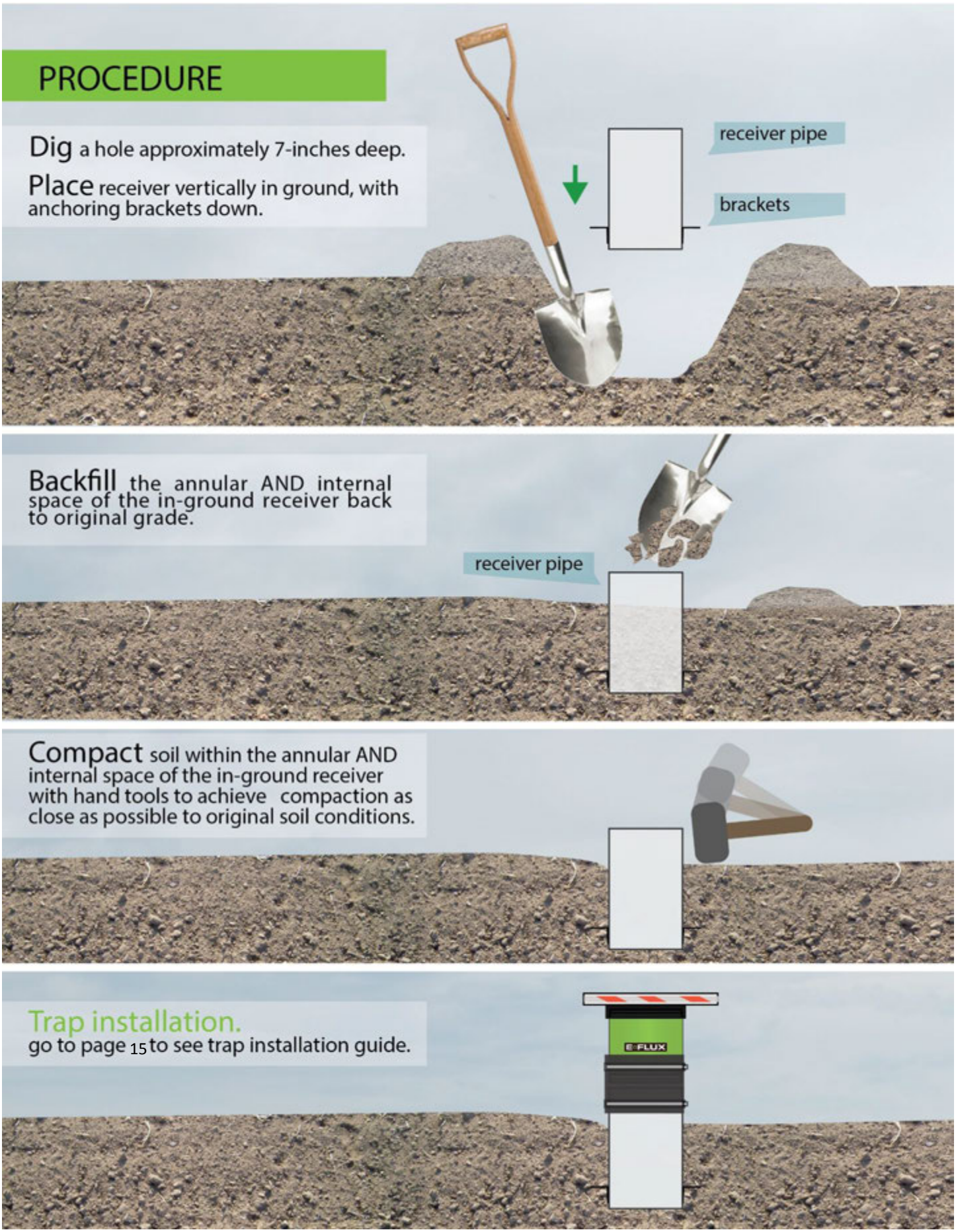
The hole + backfill option can be used for temporary monitoring in most uncompacted soils (i.e. sandy soils, vegetated areas). This method involves digging a hole to a depth of 7 inches and backfilling around the receiver pipe. This ensures that the receiver pipe maintains a secure and lasting seal with the surrounding soil. Backfilling needs recompaction to original conditions to avoid disturbing gas transport processes.

Although this method is simple to implement and a good choice for most situations, it might result in soil disturbance and should not be used if recompaction to original conditions cannot be accomplished (i.e. if there is a highly compacted clay layer within the excavation depth).

Option	Depth	Seal Strength	Advantages	Disadvantages	When to Install
Hole + Backfill	7 in	High	• deep secure lasting seal	• longer installation time • requires careful recompaction to minimize soil disturbance	• soils with sand or gravel • long installation periods (more than 4 rounds of samples)

EQUIPMENT





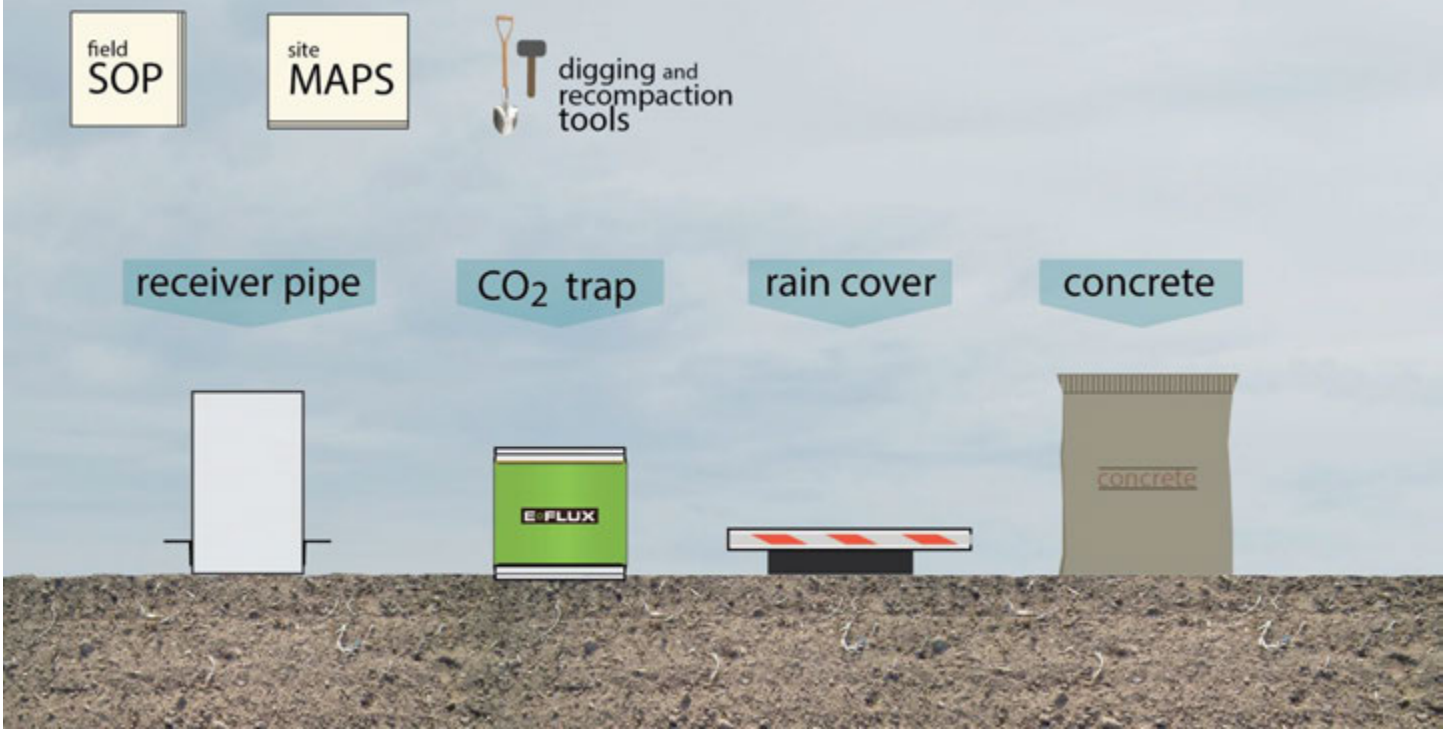
CONCRETE RING METHOD

The concrete ring method provides the most stability to the receiver pipe (suitable for long term use). The PVC receiver pipe is installed using the same procedure as the hole + backfill option but is secured with concrete after backfilling.

This method ensures that the receiver pipe will not move or shift, achieving a seal stable for long periods. Since this method is permanent, it is recommended for long installation periods (multiple rounds of sampling).

Option	Depth	Seal Strength	Advantages	Disadvantages	When to Install
Concrete Ring	7 in	High	• long lasting secure seal • permanent sampling port	• longer installation time • disturbs the soil	• soils with sand or gravel • long installation periods (more than 4 rounds of samples)

EQUIPMENT



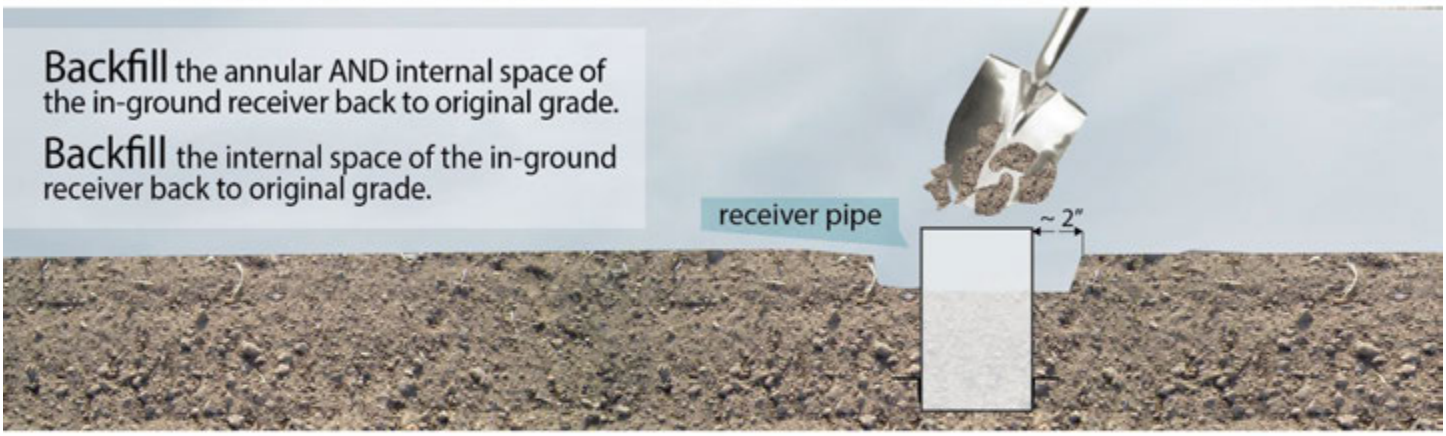
PROCEDURE

Dig a hole approximately 7-inches deep.

Place receiver vertically in ground, with anchoring brackets down.

Backfill the annular AND internal space of the in-ground receiver back to original grade.

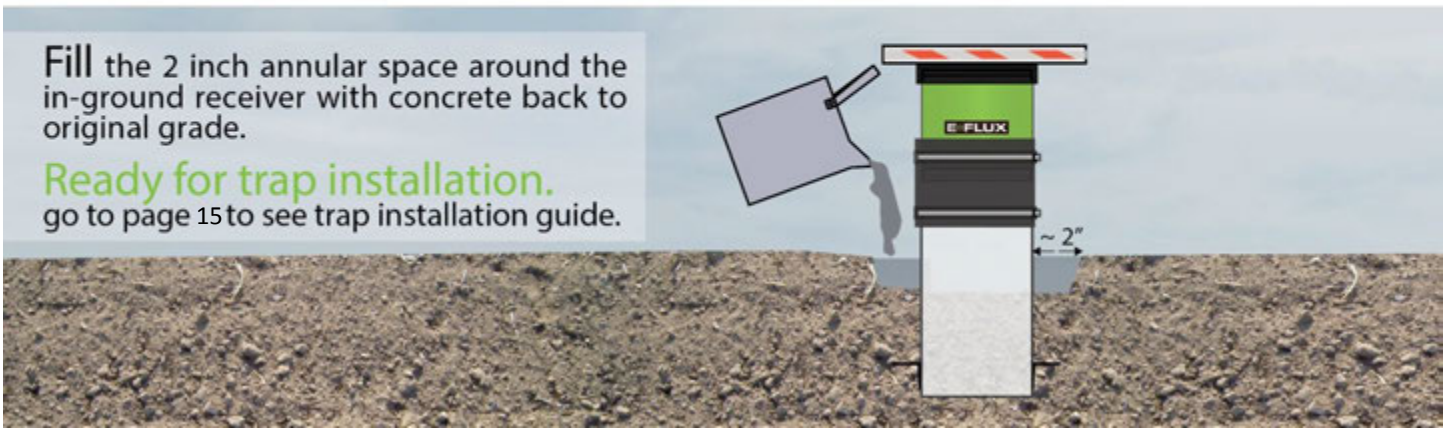
Backfill the internal space of the in-ground receiver back to original grade.



Compact soil within the annular AND internal space of the in-ground receiver with hand tools to achieve compaction as close as possible to original soil conditions.

Fill the 2 inch annular space around the in-ground receiver with concrete back to original grade.

Ready for trap installation.
go to page 15 to see trap installation guide.



TRAP INSTALLATION

EQUIPMENT

field SOP

screw driver

installed receiver pipe

CO_2 trap

rubber conector

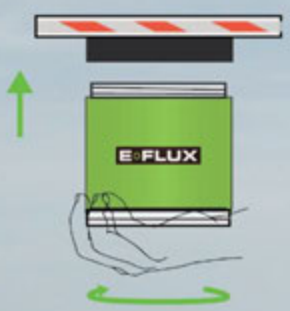
rain cover

PROCEDURE

1 SELECT the trap to be deployed. WRITE information on the installation log

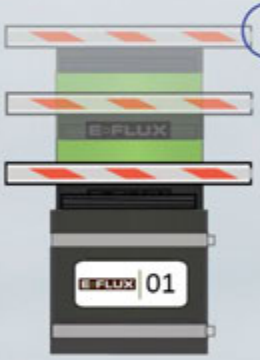
2 REMOVE top cap off the trap.

3 SCREW trap into the rain cover.



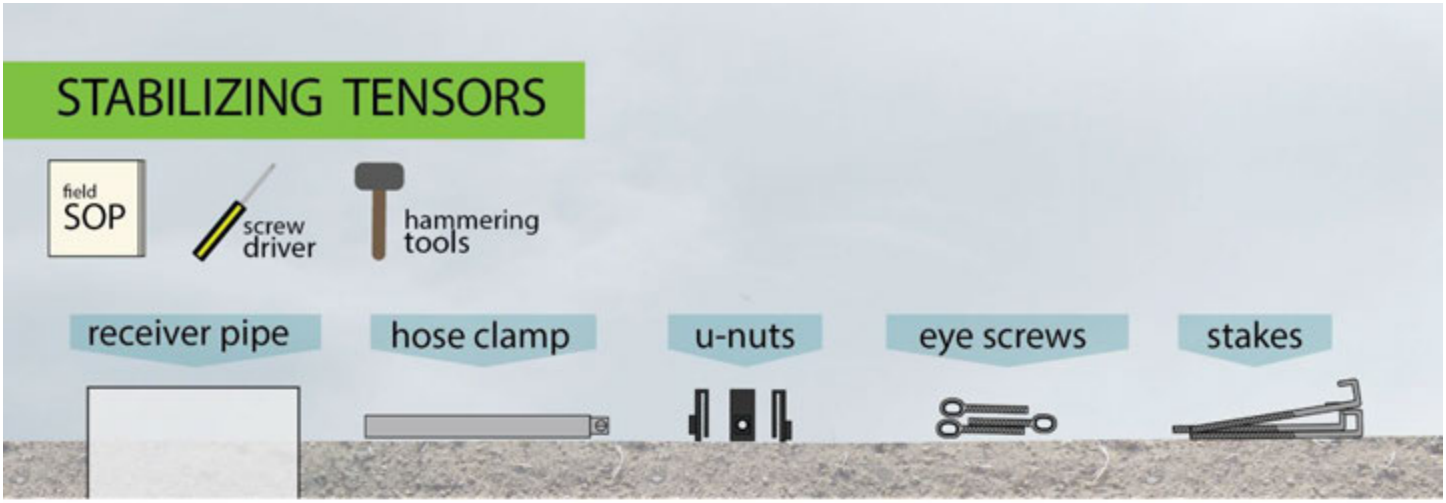
4 REMOVE bottom cap off the trap.

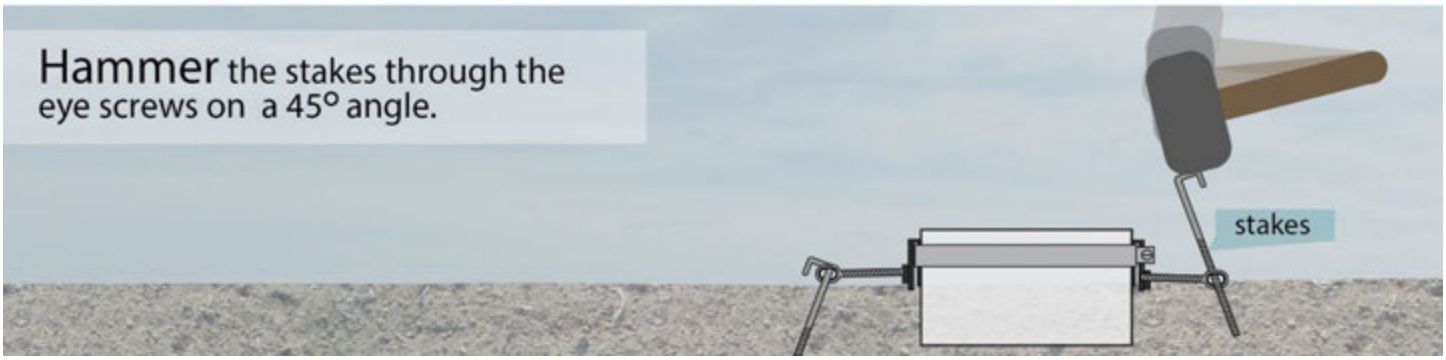
5 SLIDE trap onto the rubber conector. (carefully)
PLACE identification label on the rubber conector .



6 SLIDE trap onto the installed receiver pipe.
CAREFULLY tighten the conector's top and bottom clamps

ADD-ONS





For questions contact

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