

Caitlin  
#18693

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# Quality Assurance Project Plan (Revision 6)

# Section A: Project Management

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## A1 Title and Approval Page

Quality Assurance Project Plan  
Addendum to the DHEC UST Programmatic QAPP  
For  
Products (SE) Pipe Line Corporation/Site ID No. 18693

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Lewis Drive, Belton, Anderson County, South Carolina

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Prepared by: Jacobs

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Date: February 9, 2015  
Revised: June 7, 2023

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### Approvals

Jeffrey Mendenhall  
DHEC Project Manager

\_\_\_\_\_  
Signature

\_\_\_\_\_  
Date

Tom Wiley  
Senior Technical Consultant

  
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Signature

6/7/2023

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Date

William Waldron  
Contractor Project Manager

  
\_\_\_\_\_  
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6/7/2023

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Date

Melissa Warren  
Plan Preparer

  
\_\_\_\_\_  
Signature

6/7/2023

\_\_\_\_\_  
Date

Jon Burns  
Director Lab Operations



\_\_\_\_\_  
Date

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## A3 Distribution and Project Organization List

Name	Title/Role from UST Master QAPP	License/ Number/ Exp. Date	Organization/Address	Telephone Number	Email Address
Jeffrey Mendenhall	DHEC Technical Section Manager		DHEC, UST Management Division, 2600 Bull St. Columbia, SC, 29201	803-898-7542	<a href="mailto:mendenje@dhec.sc.gov">mendenje@dhec.sc.gov</a>
Tom Wiley	Senior Technical Consultant/Lead Geologist	PG/2787/6/30/23	Jacobs 10 10 <sup>th</sup> Street Suite 1400 Atlanta, GA 30309	404-751-5690	<a href="mailto:tom.wiley@jacobs.com">tom.wiley@jacobs.com</a>
William Waldron	Project Manager		Jacobs 111 Corning Rd. Suite 200 Cary, NC 27518	919-859-5789	<a href="mailto:wwaldron@jacobs.com">wwaldron@jacobs.com</a>
Melissa Warren	Assistant Project Manager		Jacobs 10 10 <sup>th</sup> Street Suite 1400 Atlanta, GA 30309	678-530-4388	<a href="mailto:melissa.warren@jacobs.com">melissa.warren@jacobs.com</a>
Scott Smida	Environmental Scientist/ Operations Manager		Jacobs 14120 Ballantyne Corporate Place Suite 200 Charlotte, NC 28277	704-654-7511	<a href="mailto:scott.smida@jacobs.com">scott.smida@jacobs.com</a>
Chris McCord	Laboratory Manager		Pace Analytical National 12065 Lebanon Rd. Mt. Juliet, TN 37122	615-773-3281	<a href="mailto:Chris.McCord@pacelabs.com">Chris.McCord@pacelabs.com</a>
Jon Burns	Director Lab Operations		Pace Analytical National 12065 Lebanon Rd. Mt. Juliet, TN 37122	563-599-0026	<a href="mailto:Jon.burns@pacelabs.com">Jon.burns@pacelabs.com</a>
Randy Philips	Drilling Manager	Driller 1096-A/6-30-23	Innovative Environmental Technologies, Inc 30 Grant Park PI #7864 Piedmont, SC 29673	864-288-1986	<a href="mailto:randyphillips@iet-inc.net">randyphillips@iet-inc.net</a>
James M. Pearsall, PLS	Surveyor	Surveyor/ L-4650/ 12-31-23	Taylor Wiseman & Taylor 700 Forest Point Circle Suite 166 Charlotte, NC 28273	704-527-2535	<a href="mailto:pearsall@taylorwiseman.com">pearsall@taylorwiseman.com</a>
Ryan Brown	Engineer		Jacobs 1041 East Butler Road P.O. Box 5456 Greenville, SC 29607-5456	404-978-7417	<a href="mailto:Ryan.Brown10@jacobs.com">Ryan.Brown10@jacobs.com</a>

It is understood that certification records must be produced if requested by South Carolina Department of Health and Environmental Control (DHEC). All personnel listed on the Distribution and Project Organization List in Section A3 have received the most recent version of the Site-Specific Quality Assurance Project Plan for the Products (SE) Pipe Line Corporation (PPL) Lewis Drive Release and the UST Management Division Quality Assurance Program Plan (QAPP).

## A4 Project/Task Organization

The Assistant Project Manager and Project Manager as defined in Section A3 will be responsible for maintaining the QAPP. Please see Section A3 for laboratory information, individuals involved in the project, and individual roles on the project.

## A5 Problem Definition/Background

Discuss the background (as much as is known) of the site and appropriate historical information, and why this site is being assessed.

- PPL operates a 26-inch fuel transmission line that passes along the western edge of Lewis Drive near Belton, Anderson County, South Carolina. On December 8, 2014, a fuel release occurred on the 26-inch line approximately 600 feet north of the intersection of Lewis Drive and W Calhoun Road (State RD S-4 205) (Figure 1). Between December 8, 2014 and February 2, 2015, PPL determined the release to be gasoline with a minor amount of diesel. PPL and its contractors repaired the pipeline, installed product recovery sumps, product recovery wells, temporary wells, and product interceptor trenches upgradient of Browns Creek (removed 2020; Figure 1). Between December 2014 and May 2022, PPL and its contractors currently have installed 86 monitoring wells, 5 piezometers, and a sparging remediation system consisting of 58 vertical wells and 6 horizontal wells. Figure 1 also shows the extent of benzene, toluene, ethylbenzene, and total xylenes (BTEX) as of September 2022 in the residuum aquifer. Figure 2 shows the extent of BTEX as of September 2022 in the bedrock aquifer.
- The proposed 2023 annual, semiannual, quarterly, and monthly sampling events; semiannual product recovery; and semiannual reporting schedule are based on the current Table 1 – Groundwater and Surface Water Monitoring and Reporting Plan (January 1, 2023 to December 31, 2023) accepted by DHEC on December 21, 2022 (Jacobs, 2022).
- Impacts to groundwater have been contained, with the exception of a small area where impacted groundwater flows to Browns Creek. Through the groundwater monitoring network at the site, the vertical and horizontal extents of petroleum hydrocarbons in soil and groundwater have been sufficiently defined such that a corrective action plan (CAP) was developed (CH2M, 2016). Several addendums have since been submitted to South Carolina DHEC, and the most recent addendum included expansion of the horizontal air sparge system (Jacobs, 2021). The lateral extent of impacts to groundwater extend in three directions from the release point: to the north approximately 900 feet into an adjacent hayfield; to the northeast approximately 1,000 feet to Browns Creek; and to the south approximately 300 feet to Calhoun Road. The vertical extent of impacts outside the plume boundaries are defined by bedrock monitoring wells located to the north, south, east, and west.
- A local well survey will be performed in April 2023 to identify public or private water wells that are being used within a 1,000-foot radius of the edge of the dissolved plume. Potable water is supplied to the surrounding area by the City of Belton. A 10-inch water supply main runs along West Calhoun Road. An initial receptor survey was completed in February 2015 which indicated no potable wells within a 1,500-foot radius of the site.
- Minor impacts to surface water in Browns Creek have occurred in the area where impacted groundwater discharges to Browns Creek. Surface water analytical data indicate that benzene is the only compound present at a concentration exceeding DHEC water quality standards. These exceedances are currently limited to location SW-02 and SW-04. Numerous downstream sampling locations have not had hydrocarbon detections since the discovery of the release.

Please answer the following: Does this project fall under UST or Brownfields area?

- The site has never operated USTs, but this release will be regulated by rules promulgated under the DHEC UST Management Division.

## A6 Project/Task Description

1. Summarize what is known about the work to be done. This can be a short sentence indicating what the Scope of this project is (see Master QAPP Section A6).
  - The work proposed includes: 1) the installation of additional overburden monitoring wells and bedrock monitoring wells to evaluate the distribution of dissolved hydrocarbons in groundwater along the periphery of the BTEX plume north of Lewis Drive and adjacent to Browns Creek (Figures 1 and 2). 2) the potential installation of up to 16 vertical overburden sparging wells adjacent to Browns Creek and north of Lewis Drive to allow the injected air to be distributed via the same network that transmitted impacts to groundwater in this zone. 3) Potential dual row of injectable activated carbon points in the Cupboard Creek area at 6 feet intervals (approximately 100 points), downgradient of the existing vertical air sparge curtain.
  - The proposed 2023 annual, semiannual, quarterly, and monthly sampling events; semiannual product recovery; and semiannual reporting schedule are based on the current Table 1 – Groundwater and Surface Water Monitoring and Reporting Plan (January 1, 2023 to December 31, 2023) accepted by DHEC on December 21, 2022 (Jacobs, 2022) with locations shown on Figures 1 and 2.
2. Are there any time or resource constraints? Include those factors that may interfere with the tentative schedule.
  - Constraints may include weather, equipment failure/availability, subcontractor availability, and property access.

## A7 Data Quality Objectives (DQOs) and Data Quality Indicators (DQIs)

Detail the geographical area that is to be part of the project. Maps should be included to show not only the topography and the geographical area of the State, but also to show more detail of the site itself including property lines.

- Figures 1 and 2 show the locations of existing monitoring wells, sparging wells, temporary wells, and recovery features. Figure 3 shows the locations of surface water sampling points.

## Monitoring Well Installation and Development

### Regolith Monitoring Well Construction

Regolith monitoring wells will be constructed as Type III 2-inch diameter monitoring wells, and will be constructed in accordance with DHEC Well Standards R.61-71 and standard operating procedure (SOP) A1 of Appendix A. All wells will be drilled and constructed by a South Carolina certified well driller in accordance with 40-23-10 seq. The wells will be drilled using hollow-stem auger (HSA). The wells will be constructed using 10 to 15 feet of 2-inch inside diameter (ID) Schedule 40 polyvinyl chloride (PVC) well screen and a variable amount of 2-inch ID Schedule 40 PVC riser. The screen will have a slot size of 0.010-inches, and the screen will be positioned to straddle the water table to allow product (if present) to enter the well, and to

account for seasonal fluctuations of the water table. Sand pack will be placed in the annular space between the borehole and well screen and will be brought to a height 2-feet above the top of the well screen. A 2-foot bentonite seal will be placed above the sand pack and will be hydrated. The seal will be allowed to hydrate for a minimum of 1 hour before placing grout above the seal. A grout seal containing Portland cement mixed with 3 to 5 percent bentonite will be placed above the grout seal by forced injection via tremie pipe and will be brought to within 1 foot of ground surface.

### **Bedrock Monitoring Wells**

Bedrock wells will be constructed as Type I wells (open hole in bedrock aquifer). The wells will be constructed in accordance with DHEC Well Standards R.61-71 and SOPs A2 and A3 of Appendix A. All wells will be drilled and constructed by a South Carolina certified well driller in accordance with 40-23-10 seq. The wells will be drilled using a combination of HSA, rock coring, and air rotary or hammer. In each case, HSA drilling techniques will be used to drill through the regolith until auger refusal is encountered. A temporary casing will be installed and NQ-sized (75.7 mm outside diameter/47.6 inside diameter) rock coring advanced until competent bedrock is encountered, as defined by a rock quality designation of 75% or greater. Following completion of rock coring, a nominal 10-inch borehole will be advanced 5 to 10 feet into competent bedrock. A six-inch steel casing will be installed in the borehole and grouted in place using by a forced-injection method via tremie pipe. Once the grout has cured for a minimum of 24 hours, a nominal 6-inch borehole will be advanced using air rotary or air hammer techniques approximately 10 to 20 feet or until the first water bearing fracture is encountered.

### **Well Completions (Regolith and Bedrock)**

The wells will be finished as either flush-mount completions, or aboveground locations depending on specific well location requirements and installed in accordance with SOPs A1 and A2 of Appendix A. Flush-mount wells will be installed in areas that are subject to vehicle and/or equipment traffic (roads, lawns), and while aboveground completions will be installed in areas not subject to vehicle/equipment traffic (peripheral edge of field), or in areas where a flush-mount well would be difficult to locate (woods). The flush-mount wells will be constructed using a watertight 8-inch diameter well vault set in a 2-foot square concrete pad recessed to surrounding grade. The aboveground completions will be constructed using a locking well vault set in a 2-foot square concrete pad that is surrounded by four, steel bollards.

Each well will be secured with a locking well cap. In addition, a durable, weatherproof, rustproof, name plate that contains the following information will be affixed to the well vault:

- Company name and certification number of the driller who installed the well
- Date the well was completed
- Total depth (feet bTOC)
- Casing depth (feet bTOC)
- Screen interval (feet bTOC)
- Well identification

### **Well Development**

The wells will be developed by the well driller using a one or more of the following techniques:

- Airlift
- Surge block and well pump

The wells will be developed until the water produced is clear and free of sediment in accordance with SOPs A1 and A2 of Appendix A.

## A8 Certification

The following laboratory will be used for this project:

### Commercial Lab(s)

**Full Name of the Laboratory:** Pace Analytical National

**Name of Lab Director:** Jon Burns

**DHEC Certification Number:** 84004002

Please note: DHEC may require that the contractor submit some or all of the Laboratory's SOPs as part of this QAPP. The laboratory certification is included as Appendix C to this QAPP.

## A9 Documents and Records

Personnel will receive the most current version of the QAPP Contractor Addendum and UST QAPP via:

(Check all that apply)

US Mail  Courier  Hand delivered

Other (please specify): \_\_\_\_\_

**Table A9A**

*Record Identification, Storage, and Disposal*

Record	Produced By	Hardcopy/ Electronic	Storage Location For how long?	Archival
Monitoring Report	Jacobs	Hardcopy and electronic copies to be provided to DHEC	Five years from date of report	Electronic copy is stored on Jacobs and PPL network



# Section B: Measurement/Data Acquisition

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## B1 Sampling Process/Experimental Design

The proposed 2023 annual, semiannual, quarterly, and monthly sampling events; semiannual product recovery; and semiannual reporting schedule are based on the current Table 1 – Groundwater and Surface Water Monitoring and Reporting Plan (January 1, 2023 to December 31, 2023), submitted to DHEC on November 28, 2022, and accepted by DHEC on December 21, 2022 (Jacobs, 2022). The 2023 proposed sampling features are shown on Figures 1 and 2 and are listed in Appendix B.

## B2 Sampling Methods

Please note: The contractor must follow sampling protocols as given in the UST QAPP.

Estimate the number of samples of each matrix that are expected to be collected:

**Table B2A**  
*Sampling Methods*

Matrix	Event	Number of Samples (per event)
Groundwater from monitoring wells	March Annual Sampling	84
	June Quarterly Sampling	24
	September Semiannual Sampling	66
	December Quarterly Sampling	24
From surface water	Monthly Surface Water Sampling	13
Duplicate samples	Each Groundwater Sampling Event	Up to 9
Field blanks	Each Groundwater Sampling Events	Up to 4
Trip blanks	Each Sampling Event	Up to 8
<b>Total number of samples</b>		<b>Up to 118</b>

The samples will be (check all that apply):  Grab  Homogenized  Split

- Sample collection will be performed in accordance with the media specific requirements and techniques outlined in the DHEC UST Division Programmatic QAPP (July 2022).
- HydraSleeve™ sampling techniques, in accordance with SOP A4 of Appendix A, will be used to collect groundwater samples from the monitoring wells for laboratory analysis. If there is not a sufficient water column in a well to fully submerge the HydraSleeve™ then low-flow purging and sampling techniques will be used to collect the groundwater samples, as described below.
- Low-flow purging techniques, in accordance with SOP A5 of Appendix A, will be used to collect groundwater samples from the monitoring wells for laboratory analysis, when HydraSleeve™ sampling techniques are not feasible. During purging and sampling drawdown will be no greater than 4 inches, and the tubing will be placed within the screened interval of the well. Groundwater samples will be collected from the monitoring wells using low-flow purging and sampling techniques no earlier than seven days after well development to ensure that the aquifer is fully recovered.
- If HydraSleeve™ or low-flow sampling techniques with peristaltic, submersible, or bladder pumps are not feasible (possibly due to a limited water column and/or a water level depth exceeding 25 feet [typical peristaltic pump limitation]), then multi-volume purge techniques, using a bailer or submersible pump will be considered. If the well is purged to dryness, sampling will occur as soon as adequate volume has recovered.
- The surface water samples will be collected in accordance with SOP A6 of Appendix A, by dipping the sample bottles into the creek at each sampling station to fill the bottles. Sampling will begin at the most downstream location and proceed to the most upstream location to eliminate the effects of streambed disturbance on sample integrity.

If split samples are checked, please indicate how will it be done, and the equipment needed.

- Duplicate samples of groundwater will be collected by dividing the bailer volumes or pumped water volumes (if low-flow sampling techniques used) into separate container sets.

Decontamination procedures will be performed in accordance with the procedures described in SOP A11 of Appendix A.

Identify any equipment and support facilities needed. This may include such things as FedEx® to ship the samples, a Geoprobe®, field analysis done by another contractor (who must be certified), or electricity to run sampling equipment.

- Jacobs field staff will transport samples directly to the shipping carrier (i.e., FedEx®) following standard chain-of-custody (CoC) procedures.

Address the actions to be taken when problems occur in the field, and the person responsible for taking corrective action and how the corrective action will be documented.

**Table B2B**  
*Field Corrective Action*

<b>Failure</b>	<b>Response</b>	<b>Documentation</b>	<b>Individual Responsible</b>
PID does not calibrate or malfunctions	Re-calibrate. Follow trouble-shooting guide in manual and contact rental company. If unable to calibrate and/or instrument functions erratically return meter and obtain replacement meter.	Document in Field Notebook	Field personnel
Groundwater multi-meter (pH, temperature, conductivity, redox)	Re-calibrate. Follow trouble-shooting guide in manual and contact rental company. If unable to calibrate and/or instrument functions erratically return meter and obtain replacement meter.	Document in Field Notebook	Field personnel
Interface Probe does not function properly	Following trouble-shooting guide in manual and contact rental company. If instrument functions erratically return meter and obtain replacement meter.	Document in Field Notebook	Field personnel
All remaining equipment	Re-calibrate. Follow trouble-shooting guide in manual and contact rental company. If unable to calibrate and/or instrument functions erratically return meter and obtain replacement meter.	Document in Field Notebook	Field personnel

## B3 Sample Handling and Custody

1. How will the samples get from the Site to the Lab to ensure holding requirements are met?
  - FedEx® or another overnight courier. Additionally, field staff may hand deliver samples to laboratory.
2. If sample preservation procedures differ from the UST Programmatic QAPP, please provide details.
  - No deviation from UST Programmatic QAPP.
3. If CoC procedures differ from the UST Programmatic QAPP, please provide details.
  - No deviation from UST Programmatic QAPP.

## B4 Analytical Methods

1. Identify the SOPs which will be used to analyze the samples, the method which the SOP references and the equipment or instrumentation that is needed:

**Table B4A**  
*Analytical SOPs and Referenced Methods*

Parameter	Method Referenced	Comments/SOP
<b>Soils</b>		
BTEX, naphthalene	EPA Method 8260D	Volatile Organic Compounds by GC/MS (EPA 8260B, 8260C, 624 and SM6200B) Rev. 23, 10/18/2016*
<b>Groundwater</b>		
BTEX Naphthalene MTBE 1,2-DCA	EPA Method 8260D	Volatile Organic Compounds by GC/MS (EPA 8260D, 8260C, 624 and SM6200B) Rev. 23, 10/18/2016*
<b>Surface Water</b>		
BTEX, naphthalene	EPA Method 8260D	Volatile Organic Compounds by GC/MS (EPA 8260D, 8260C, 624 and SM6200B) Rev. 23, 10/18/2016*
<b>MNA Parameters</b>		
Nitrate and Sulfate	EPA Method 300.0/9056	Inorganic Anions by EPA 300.0; Rev 3, 9/2/2022
Alkalinity	SM 2320B	Total Alkalinity, Manual and Automated (Titration) by SM 2320B; Rev 7, 2/21/2022
Carbon Dioxide	Calculation from alkalinity	Carbon Dioxide by Calculation; Rev 4, 9/6/2022
Methane	RSK-175	RSK175, 8015, TO-3; Rev 4, 4/4/2023
<b>Water Quality Parameters</b>		
Turbidity Dissolved Oxygen Specific Conductance Temperature pH		Parameters collected using a YSI 6920 multimeter or equivalent. DO measurements also collected using a YSI- Pro ODO meter**

\*Included as SOP A7 in Attachment A

\*\*Included as SOP A8 in Attachment A

2. Provide SOPs for the Kerr Method or the Ferrous Iron Method if these are parameters for this study. This can be attached or written here. If attached, please note that it is an attachment and where it is located (if applicable).
  - Not applicable.

## B5 Quality Control Requirements

All QC will follow the requirements laid out in Section B5 of the UST Programmatic QAPP. If procedures for QC differ from the UST Programmatic QAPP, please provide details.

## B6 Field Instrument and Equipment Testing, Inspection and Maintenance

1. Identify all field equipment needing periodic maintenance, the schedule for this, and the person responsible.

**Table B6A**  
*Instrument and Equipment Maintenance*

Instrument	Serial Number	Type of Maintenance	Frequency	Person Responsible
YSI 6920 v2 Multi-meter or equivalent	TBD as equipment is rented	Ensure instrument is able to accurately correlate with calibration standards	Monthly	Vendor Equipment Manager (i.e., Pine Environmental)
Solinst Model 122 Oil Water Interface Probe or equivalent	TBD as equipment is rented	Ensure instrument is able to accurately correlate with calibration standards	Monthly	Vendor Equipment Manager (i.e., Pine Environmental)
MiniRAE 3000 PID PGM 7320 or equivalent	TBD as equipment is rented	Ensure instrument is able to accurately correlate with calibration standards	Monthly	Vendor Equipment Manager (i.e., Pine Environmental)
MultiRAE +4 gas meter PGM 66228 or equivalent	TBD as equipment is rented	Ensure instrument is able to accurately correlate with calibration standards	Monthly	Vendor Equipment Manager (i.e., Pine Environmental)
GeoPump™ Peristaltic Pump Series II	TBD as equipment is rented	Ensure pump suction and battery voltage	Monthly	Vendor Equipment Manager (i.e., Pine Environmental)
QED 1.75 Bladder Pump	TBD as equipment is rented	Control box pressure check, leakage check, and cycle check	Monthly	Vendor Equipment Manager (i.e., Pine Environmental)
In-Situ Rugged TROLL 100	TBD as equipment is rented	Battery check	Yearly	Vendor Equipment Manager (i.e., Pine Environmental)

## B7 Instrument Calibration and Frequency

1. Identify equipment, tools, and instruments for field or lab work that should be calibrated and the frequency.
2. Describe how the calibrations should be performed and documented, indicating test criteria and standards or certified equipment.
3. Identify how deficiencies should be resolved and documented. Identify the person responsible for corrective action.

**Table B7A**  
*Instrument Calibration Criteria and Corrective Action*

Instrument	Serial Number	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA
YSI 6920 v2 Multi-meter or equivalent	TBD as equipment is rented	Procedures adheres to standards outlined in manual for instrument	Daily	Within 0.01 of calibration standard	Re-calibrate; then replace probes or instrument	Field personnel
Solinst Model 122 Oil Water Interface Probe or equivalent	TBD as equipment is rented	Procedures adheres to standards outlined in manual for instrument	Manufacturer calibration	Manufacturer calibration – accurate to 0.01-feet	Return to vendor than obtain replacement	Field personnel
MiniRAE 3000 PID PGM 7320 or equivalent	TBD as equipment is rented	Procedures adheres to standards outlined in manual for instrument	TBD per manufacturer recommendations	0.5 ppm	Re-calibrate; then replace lamp, filters or instrument	Field personnel
MultiRAE +4 gas meter PGM 66228 or equivalent	TBD as equipment is rented	Procedures adheres to standards outlined in manual for instrument	TBD per manufacturer recommendations	0.5 ppm	Re-calibrate; then replace lamp, filters or instrument	Field personnel

\* This can be a full name of a SOP, an abbreviation, or a number. In the latter two cases, the abbreviation or number must be associated with the full name of the SOP.

## B8 Inspection/Acceptance Requirements for Supplies and Consumables

1. If procedures for storage, handling or transport of supplies/consumables differ from the UST Programmatic QAPP, please provide details.
  - No deviation from DHEC UST Programmatic QAPP.

## B9 Data Acquisition Requirements (Non-Direct Measurements)

1. Identify data sources, for example, computer databases or literature files, or models that should be accessed or used.
2. Describe the intended use of this information and the rationale for their selection.
3. Provide its relevance to the project.
4. Indicate the justification criteria for use of these data sources and/or models.
5. Identify key resources/support facilities needed.
  - Not applicable.

**Table B9A**  
*Non-Direct Measurements*

<b>Data Source</b>	<b>Used for</b>	<b>Relevance</b>	<b>Justification for Use in This Project</b>	<b>Comments</b>
Tax Map and utility maps	Determine/verify property ownership and utility locations	Used to ensure contact property owners and obtain access to property – determine locations and depths of utilities	Site access and evaluate depth of utility with respect to hydrocarbons	
USGS and DHEC Databases	Obtain geologic information and water resource information	Understanding of site stratigraphy and well records	Evaluate local stratigraphy beneath site and obtain well construction details	

# B10 Data Management

1. Describe the data management scheme from field to final use and storage.
  - The samples collected will be recorded on the laboratory CoC form as well as documented in the field logbook by the sample collection team. The samples and CoC will be relinquished to the laboratory following standard CoC methodology. Following analysis, the laboratory will perform internal data validation. The laboratory will issue a written report and submit an electronic copy to via email. The electronic copy will be stored on the Jacobs computer network in a file dedicated to the Lewis Drive project.
2. How does the lab and field staff ensure that no unauthorized changes are made to the CoC, sampling notebooks, laboratory notebooks and computer records?
  - Documents will be noted with written or electronic signature and date/time stamp. A review of all written and electronic documents by a project team member who has been assigned this task by a project leadership member to ensure integrity of the project documents.
3. CoC forms, sampling notebooks and sample collection summary sheets will be completed in the field with indelible ink. Any changes to the CoC that is not marked through and initialed will be flagged by the laboratory and an inquiry will be made. The procedures for laboratory record keeping are included in the laboratory QAM which can be provided upon request.
  - Paper copies generated during field activities will be scanned and stored electronically on Jacobs networks that are backed up each day on to an offsite tape drive. All paper copies will be maintained in project files in a secure building with 24-hour, restricted access.
4. How does the lab ensure that there are no errors in sample records including times when sample information is compiled, data calculated and/or transmitted?
  - When the laboratory receives samples for analysis, a “Review of Sample Login” report is created by the sample custodian and is reviewed by the laboratory project manager (PM) for errors. If problems are encountered, the laboratory PM contacts the Jacobs PM, and a corrective action is agreed upon and then corrected by the laboratory PM.
5. How will the data be archived once the report is produced? How can it be retrieved? (This applies to both electronic and hard copies).
  - The laboratory will store readily available electronic copies online for two years through the laboratory’s “PacePort” web link. After two years, the reports will be archived electronically onsite or offsite for an additional eight years. The archived reports can be retrieved by the laboratory through an IT request. Each project is given a unique number and is entered into an archive log to allow for retrieval. Hard copies are scanned in .pdf format and are stored electronically on the Jacobs server with the same unique number as the hard copy.
  - Jacobs assigns a unique project number to each project which is stored in sequential order by project number at the branch and are stored at a secure, restricted access location for a minimum of five years.



# Section C: Assessment and Oversight

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## C1 Assessment and Response Actions

1. The Contractor is supposed to observe field personnel daily during sampling activities to ensure samples are collected and handled properly and report problems to DHEC within 24 hours. Please state who is responsible for doing this, what observations will be made, and how those observations will be made. Will this person have the authority to stop work if severe problems are seen?
  - All Jacobs employees working on this project will verify that the samples are collected and handled properly. Additionally, all Jacobs employees working on this project have the authority to stop work, report the problem and effect a correction that is agreed upon by the Jacobs PM.
2. The DHEC UST QAPP states that the Laboratory will receive an Offsite Technical System Audit. For this project, what assessments will be done by the Contractor on the Commercial Lab(s) that are being used—other than their certification audit? When or how often are these done? Who will the results be given to and who has the ability to stop work if problems are severe?
  - The laboratory participates in semi-annual proficiency testing through an approved vendor, Phenova. The results of this proficiency testing are provided to the DHEC Office of Environmental Laboratory Certification. The laboratory is accredited by the DHEC Office of Environmental Laboratory Certification, and performs internal audits annually for each department in compliance with the laboratory's quality program.

## C2 Reports to Management

See the DHEC UST Programmatic QAPP (UST Master QAPP).

# Section D: Data Validation and Usability

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See the DHEC UST Programmatic QAPP (UST Master QAPP).

## Section E: References

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CH2M HILL (CH2M). 2016. *Corrective Action Plan Lewis Drive Release Site Belton, SC. Site ID Number 18693 ("Kinder Morgan Belton Pipeline Release")*. September 1.

Jacobs Engineering Group Inc. (Jacobs). 2021. *Corrective Action Plan Addendum #2*. May 24.

Jacobs Engineering Group Inc. (Jacobs). 2022. *Groundwater and Surface Water Monitoring and Reporting Plan (January 1, 2023 to December 31, 2023). Lewis Drive Remediation Site, Products (SE) Pipeline Corporation, Belton, South Carolina. Site ID Number 18693, "Kinder Morgan Belton Pipeline Release."* November 28.

South Carolina Department of Health and Environmental Control (DHEC). 2014. *R. 61-68, Water Classifications & Standards*. June 27.

South Carolina Department of Health and Environmental Control (DHEC). 2020. *Quality Assurance Program Plan For the Underground Storage Tank Management Division*. July.

## Figures

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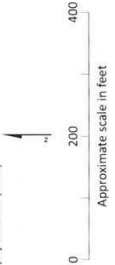
**LEGEND**

- ★ Release Point
- Residuum Monitoring Well
- ◆ Seep Location
- Recovery Well (4-inch diameter)
- ▲ Recovery Sump
- ✕ HAS-1 Manway
- ✕ HAS-4/HAS-5 Manway (Distal End)
- ✕ HAS-4/HAS-5 Manway (Proximal End)
- HAS-6 Manway (Proximal End)
- Main Valve Box
- ▬ Grout
- ▬ Horizontal Sparging Well Screen
- ▬ Horizontal Sparging Well Risers
- ▬ Dissolved Benzene Plume Extent as of December 2022 (ug/L) (dashed where inferred)
- ▬ Surface Water Flow Direction
- ▬ Waterbody
- ▬ Intermittent Stream
- ▬ Delineated Wetland

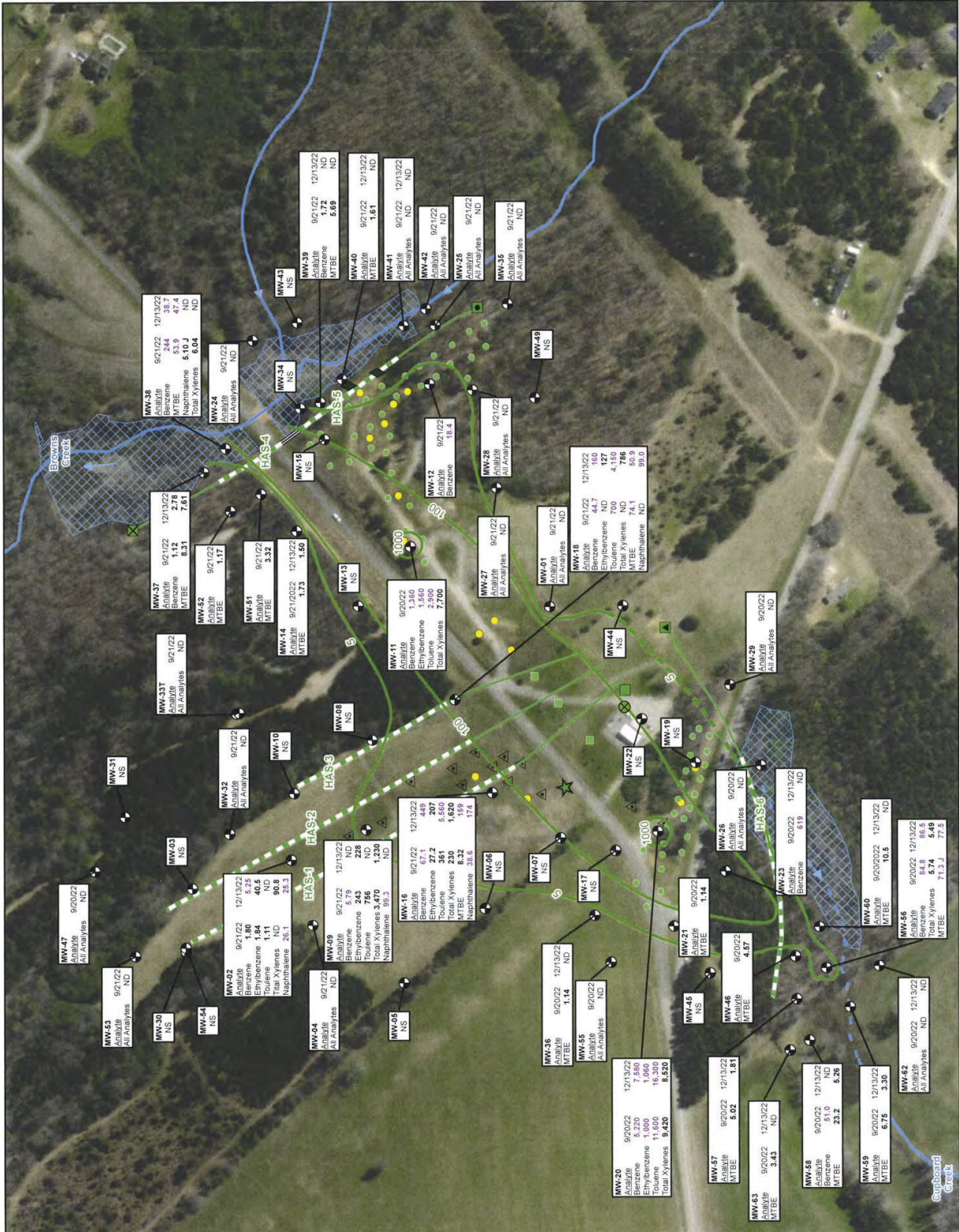
- NOTES**
1. Total Xylenes is the sum of m-, o-, and p-xylene
  2. ND = Not Detected
  3. Analyte concentration in microgram(s) per liter (ug/L)
  4. Only detected analytes are shown on map
  5. J = Estimated value
  6. MW = monitoring well
  7. ND = Groundwater was collected and analyzed, but no analytes were detected above the reported sample quantitation limit
  8. NS = Sample not collected
  9. Isocoumbars are based on the December 2022 Semiannual Monitoring Event Results

Purple indicates the analyte exceeded risk-based screening levels (RBSLs) identified in South Carolina Underground Storage Tank Management Division Programmatic Quality Assurance Program Plan Revision 3.1, Table D1 "RBSLs for Groundwater", February 2016.

Base Map Sources:  
 Environmental Systems Research Institute (Esri)  
 National Imagery and Mapping Agency, 2021. Basemap features are approximate.  
 "United States Geological Survey (USGS) National Hydrography Dataset (NHD)



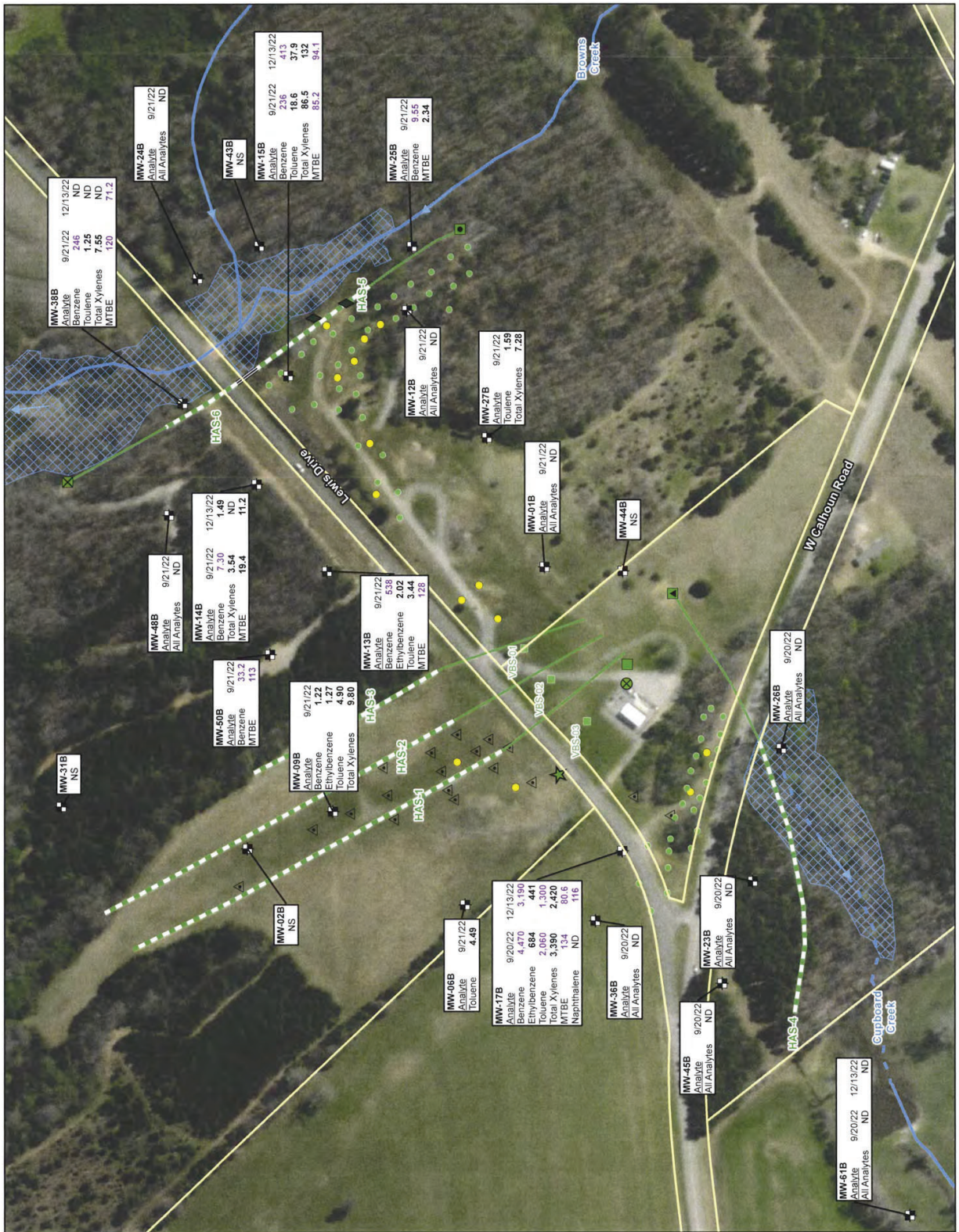
**Figure 1. Groundwater Analytical Results in Residuum Aquifer, September and December 2022**  
 Lewis Drive Remediation Site  
 Belton, South Carolina  
 Site ID #18693 "Kinder Morgan Belton Pipeline Release"



C:\Users\jacobson\Documents\18693\GIS\RESULTS\RESULTS\_2022\MW\_RESULTS\_2022\MW\_RESULTS\_2022\_09\_15\_2022.aprx

**LEGEND**

- ★ Release Point
- ⊠ Bedrock Monitoring Well
- ◆ Seep Location
- Recovery Well (4-inch diameter)
- ▲ Recovery Sump
- ⊗ HAS-1 Manway
- ⊗ HAS-4/HAS-5 Manway (Distal End)
- ⊗ HAS-4/HAS-5 Manway (Proximal End)
- ⊗ HAS-6 Manway (Proximal End)
- ⊗ Main Valve Box
- Surface Water Flow Direction
- ▬ Grout
- ▬ Horizontal Sparging Well Screen
- ▬ Horizontal Sparging Well Riser
- ▬ Waterbody
- ▬ Intermittent Stream
- ▬ Delineated Wetland
- ▬ Parcel Boundary



**NOTES:**

- Total Xylenes is the sum of m-, o-, and p-xylene
- MTBE = Methyl Tertiary Butyl Ether
- ND = Not Detected
- Only detected analytes are shown on map
- MW = monitoring well
- ND = Groundwater was collected and analyzed, but no analytes were detected above the reported sample quantitation limit

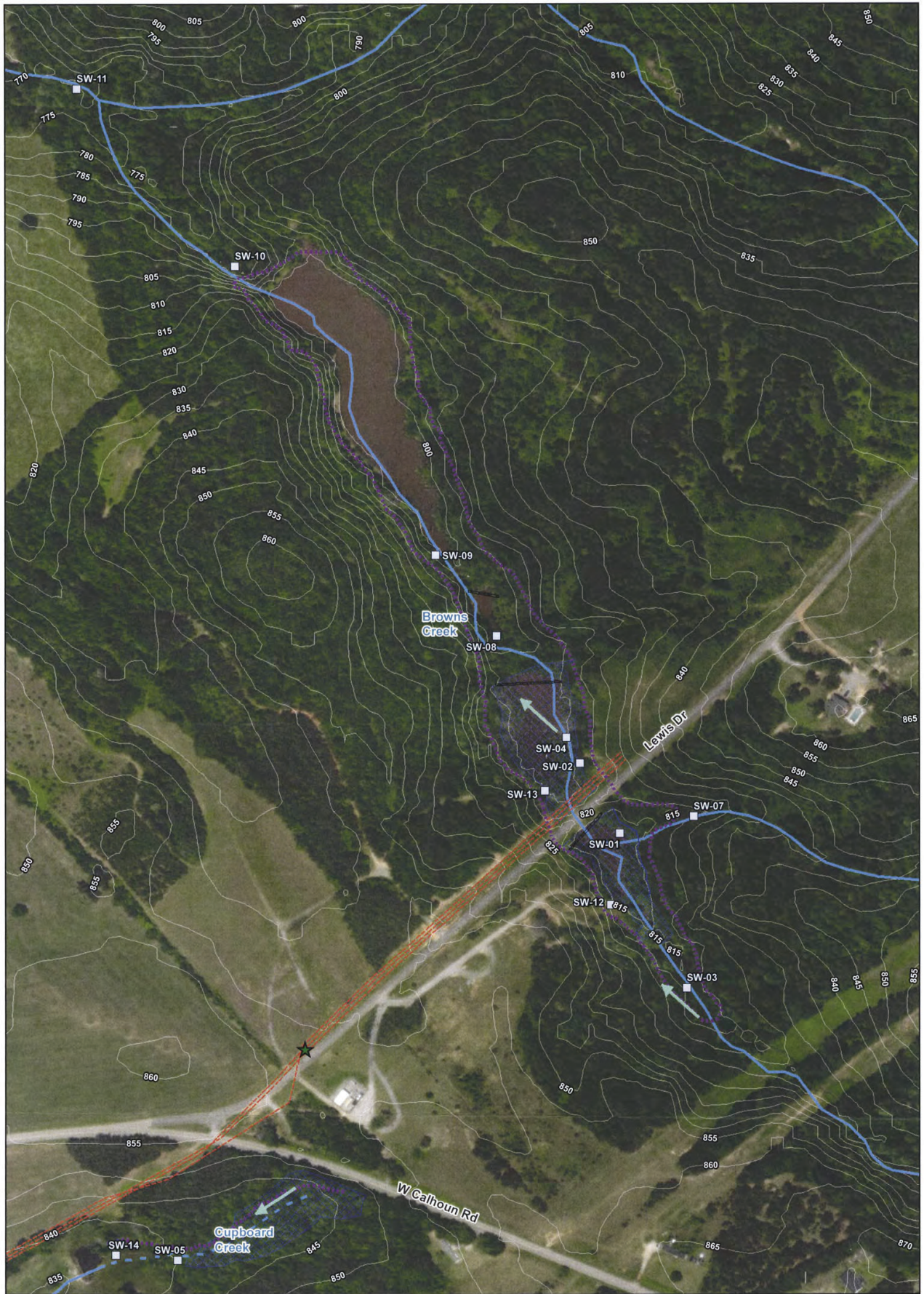
Purple indicates the analyte exceeded risk-based screening levels (RBSLs) identified in South Carolina Underground Storage Tank Management Division Programmatic Quality Assurance Program Plan Revision 3.1, Table D1 "RBSLs for Groundwater", February 2016.

**Base Map Sources:**

- Environmental Systems Research Institute (Esri)
- Aerial Imagery, 2021. Basemap features are provided by Esri
- United States Geological Survey (USGS) National Hydrography Dataset (NHD)



**Figure 2. Groundwater Analytical Results in Bedrock Aquifer, September and December 2022**  
Lewis Drive Remediation Site  
Belton, South Carolina  
Site ID #18693 "Kinder Morgan Belton Pipeline Release"



**LEGEND**

- ★ Release Point
- Surface Water Sampling Location
- Pipeline
- Inspection Route for Sheen or Distressed Vegetation
- Flow Direction of Creek
- Waterbody
- Intermittent Stream
- Delineated Wetland
- ⊗ Beaver Dam



Base Map Source:  
 \*Environmental Systems Research Institute (Esri) ArcMap  
 World Imagery, 2020  
 \*United States Geological Survey (USGS) National  
 Hydrography Dataset (NHD)

**Figure 3. Surface Water Sampling Plan**  
 Lewis Drive Remediation Site  
 Belton, South Carolina  
 Site ID #18693 "Kinder Morgan Belton Pipeline Release"

**Appendix A**  
**Standard Operating Procedures**

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# Installation of Shallow Monitoring Wells

## A1.1 Purpose and Scope

The purpose of this guideline is to describe methods for drilling and installation of shallow monitoring wells and piezometers in unconsolidated or poorly consolidated materials using hollow stem augers, air rotary, or mud rotary. Methods for drilling and installing bedrock monitoring wells and deep, surface-cased wells in unconsolidated materials are presented in SOPs *Installation of Bedrock Monitoring Wells* and *Installation of Surface-Cased Monitoring Wells*, respectively. This SOP is consistent with the installation and completion of a Type III well per South Carolina Well Standards R.61-71

## A1.2 Equipment and Materials

### A1.2.1 Drilling

- Drilling rig (hollow stem auger, air rotary or mud rotary) and associated tools and equipment

### A1.2.2 Well Riser/Screen and Associated Materials

- Polyvinyl chloride (PVC), Schedule 40, minimum 2-inch ID, flush-threaded riser; alternatively, stainless-steel riser
- PVC, Schedule 40, minimum 2-inch ID, flush-threaded, factory slotted screen; alternatively, stainless-steel screen
- PVC bottom cap, threaded to match the well screen; alternatively, stainless steel
- PVC or stainless-steel centering guides (if used)
- Above-grade well completion: PVC well cap, threaded or push-on type, vented
- Flush-mount well completion: PVC well cap, locking, leak-proof seal
- Stainless steel to be used as appropriate

### A1.2.3 Sand

- Clean silica sand, provided in factory-sealed bags, well-rounded, containing no organic material, anhydrite, gypsum, mica, or calcareous material; primary (coarse – e.g., Morie #1) filter pack, and secondary (fine sand seal) filter pack. Grain size determined based on sediments observed during drilling.

### A1.2.4 Bentonite

- Pure, additive-free bentonite pellets or chips
- Pure, additive-free powdered bentonite
- Coated bentonite pellets; coating must biodegrade within 7 days
- Cement-Bentonite Grout: proportion of 6 to 8 gallons of water per 94-pound bag of Portland cement; 3 to 6 pounds of bentonite added per bag of cement to reduce shrinkage.

### A1.2.5 Protective Casing

- Above-grade well completion: 6-inch minimum ID black iron steel pipe with locking cover, diameter at least 2 inches greater than the well casing, painted with epoxy paint for rust protection; heavy duty lock; protective posts if appropriate
- Flush-mount well completion: Traffic rated steel 9-inch or 12-inch 519 manhole cover, or equivalent; rubber seal to prevent leakage; locking cover inside of road box

### A1.2.6 Well Development

- Surge block
- Well-development pump and associated equipment
- Calibrated meters to ensure pH, temperature, specific conductance, ORP, and dissolved oxygen of development water
- Containers (e.g., DOT-approved 55-gallon drums) for water produced from well.

## A1.3 Procedures and Guidelines

### A1.3.1 Drilling Method

Typically, continuous-flight hollow-stem augers with a minimum 4.25-inch inside diameter (ID) will be used to drill shallow monitoring well boreholes for 2-inch diameter monitoring wells. Alternatively, air or mud rotary may be used.

The bit of the auger is placed at the ground surface and then turned with the drilling rig. To collect split spoon samples, the auger is advanced to the top of the sampling depth, and the split-spoon sample is collected from below the auger head. The split spoon is advanced through repeated blows from a 140- or 300-pound hammer dropped from a height of 30 inches. Thin-walled tube samplers are advanced by pressing down on the rods with the weight of the drilling rig. Split-spoon samples may be collected at selected intervals for chemical analysis and/or lithologic classification.

The use of water to assist in hollow-stem auger drilling for monitoring well installation will be avoided, unless required for such conditions as running sands.

Hollow-stem augers, drilling bits, rods, split-spoon samplers, and other downhole drilling tools will be properly decontaminated prior to the initiation of drilling activities and between each borehole location. Split-spoon samplers and other downhole soil sampling equipment will also be properly decontaminated before and after each use. *SOP Decontamination of Drill Rigs and Equipment* details proper decontamination procedures.

Drill cuttings and decontamination fluids generated during well drilling activities will be contained as Investigative Derived Waste (IDW).

Air or mud rotary drilling may be used instead of hollow-stem augers. The use of added mud should be kept to a minimum.

### A1.3.2 Monitoring-Well Installation

Shallow monitoring wells will be constructed inside the hollow-stem augers, once the borehole has been advanced to the desired depth, or in the mudded borehole once the drilling rods have been withdrawn. If the borehole has been drilled to a depth greater than that at which the well is to be set, the borehole will be backfilled with bentonite pellets or chips or a bentonite-cement slurry to a depth approximately

1 foot below the intended well depth. Approximately 1 foot of clean sand will be placed on top of the bentonite to return the borehole to the proper depth for well installation.

The appropriate lengths of well screen, nominally 10 feet (with bottom cap), and casing will be joined watertight and lowered inside the augers to the bottom of the borehole. Centering guides, if used, will be placed at the bottom of the screen and above the interval in which the bentonite seal is placed.

Selection of the filter pack and well screen intervals for the shallow monitoring wells shall be made in the field.

A primary sand pack consisting of clean DSI No. 1 or equivalent silica sand for 10-slot screen and DSI No. 2 or equivalent for 20-slot screen silica sand will be placed around the well screen. The sand will be placed into the borehole at a uniform rate, in a manner that will allow even placement of the sand pack. The augers will be raised gradually during sand pack installation to avoid caving of the borehole wall; at no time will the augers be raised higher than the top of the sand pack during installation. During placement of the sand, the position of the top of the sand will be continuously sounded. The primary sand pack will be extended from the bottom of the borehole to a minimum height of 2 feet above the top of the well screen. A secondary, finer-grained (fine sand seal), sand pack will be installed for a minimum of 1 foot above the coarse sand pack. Heights of the coarse and fine sand packs and bentonite seal may be modified in the field to account for a shallow water table and a small saturated thickness of the surficial aquifer.

A bentonite seal at least 2 feet thick will be placed above the sand pack. The seal will be placed into the borehole in a manner that will prevent bridging. The position of the top of the bentonite seal will be verified using a weighted tape measure. If all or a portion of the bentonite seal is above the water table, clean water will be added to hydrate the bentonite. A hydration period of at least 30 minutes will be required following installation of the bentonite seal.

Above the bentonite seal, an annular seal of cement-bentonite grout will be placed. The cement-bentonite grout will be installed continuously in one operation from the bottom of the space to be grouted to the ground surface through a tremie pipe. The tremie pipe must be plugged at the bottom and have small openings along the sides of the bottom 1-foot length of pipe. This will allow the grout to diffuse laterally into the borehole and not disturb the bentonite pellet seal.

### A1.3.3 Well Completion

For monitoring wells that will be completed above-grade, a locking steel protective casing set in a concrete pad will be installed. The steel protective casing will extend at least 3 feet into the ground and 2 feet above ground but should not penetrate the bentonite seal. The concrete pad will be square, approximately 2 feet per side (unless otherwise specified in the project plans), and poured into wooden forms. The concrete will be sloped away from the protective casing.

Guard posts may be installed in high-traffic areas for additional protection. Four steel guard posts will be installed around the protective casing. Guard posts would be concrete-filled, at least 2 inches in diameter, and would extend at least 2 feet into the ground and 3 feet above the ground. The protective casing and guard posts will be painted with an epoxy paint to prevent rust.

For monitoring wells with flush-mount completions, Morrison 9-inch or 12-inch 519 manhole cover or equivalent, with a rubber-sealed cover and drain will be installed. The top of the manhole cover will be positioned approximately 1 inch above grade. A square concrete pad, approximately 2 feet per side (unless otherwise specified in the project plans), will be installed as a concrete collar surrounding the road box cover, and will slope uniformly downward to the adjacent grade. The road box and installation thereof will be of sufficient strength to withstand normal vehicular traffic.

Concrete pads installed at all wells will be a minimum of 6 inches below grade. The concrete pad will be 12 inches thick at the center and taper to 6-inch thick at the edge. The surface of the pad should slope away from the protective casing to prevent water from pooling around the casing. Protective casing, guard posts, and flush mounts will be installed into this concrete.

Each well will be properly labeled on the exterior of the locking cap or protective casing with a metal stamp indicating the permanent well number.

### A1.3.4 Well Development

- New monitoring wells will be developed after the well has been completely installed and the grout has hardened (at least 24 hours)
- The well will be developed by surging and pumping.
- Equipment placed in the well will be decontaminated before use.
- If information is available, begin developing in the least-contaminated well first.
- Development will include surging the well by either abruptly stopping flow and allowing water in the well column to fall back into the well or through the use of a surge block that is slightly smaller in diameter than the well casing inner diameter.
- Pipes and pumps must not be fitted with foot valves or other devices that might inhibit the return flow of water to the well.
- Surging should continue throughout the development process.
- The air-lift method may be used to pump materials out of the well. The air compressor will be fitted with filters to remove all oil and the air lift hose used will be made of inert materials.
- Well development will continue until the water produced is free of turbidity, sand, and silt. A Horiba-U22 meter, YSI meter, or equivalent should be used to determine when the turbidity is low and parameters have stabilized.

## A1.4 Attachments

Schematic diagram of shallow monitoring-well construction (MWSingleDiag.xls)

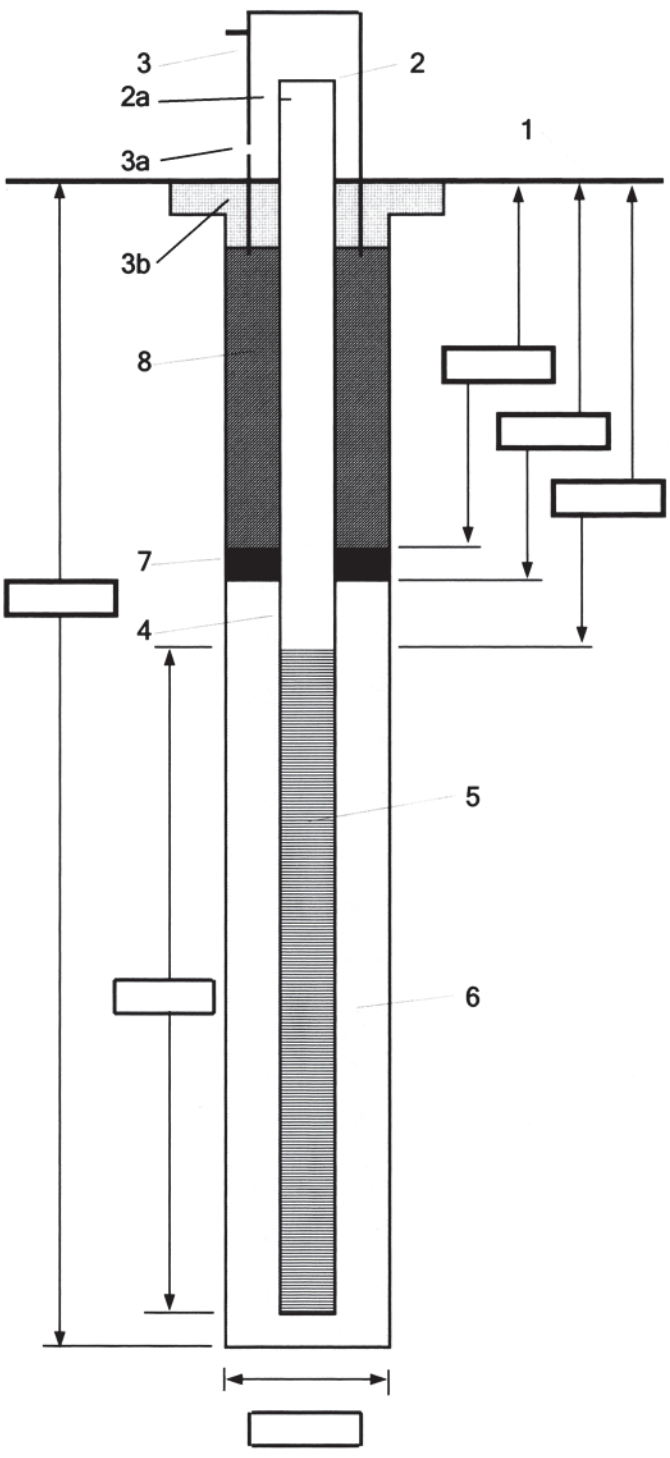
# WELL COMPLETION DIAGRAM

PROJECT : \_\_\_\_\_ LOCATION : \_\_\_\_\_

INSTALLING CONTRACTOR : \_\_\_\_\_

INSTALLING METHOD AND EQUIPMENT USED : \_\_\_\_\_

INSTALLER LEVELS : \_\_\_\_\_ START : \_\_\_\_\_ END : \_\_\_\_\_ LOGGER : \_\_\_\_\_



1- Ground elevation at well \_\_\_\_\_

2- Top of casing elevation  
a) vent hole? \_\_\_\_\_

3- Wellhead protection cover type \_\_\_\_\_  
a) weep hole? \_\_\_\_\_  
b) concrete pad dimensions \_\_\_\_\_

4- Dia./type of well casing \_\_\_\_\_

5- Type/slot size of screen \_\_\_\_\_

6- Type screen filter  
a) Quantity used \_\_\_\_\_

7- Type of seal  
a) Quantity used \_\_\_\_\_

8- Grout  
a) Grout mix used \_\_\_\_\_  
b) Method of placement \_\_\_\_\_  
c) Vol. of well casing grout \_\_\_\_\_

Development method \_\_\_\_\_

Development time \_\_\_\_\_

Estimated purge volume \_\_\_\_\_

Comments \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

# Installation of Bedrock Monitoring Wells

## A2.1 Purpose and Scope

The purpose of this procedure is to outline equipment and methods that will be used for bedrock well installation and development. Methods for drilling and installing shallow and deep, surface-cased wells in unconsolidated materials are presented in SOPs *Installation of Shallow Monitoring Wells* and *Installation of Surface-Cased Monitoring Wells*, respectively. This SOP is consistent with the installation and completion of a Type I well or Type III well if not completed as open hole per South Carolina Well Standards R61-71.

## A2.2 Equipment and Materials

### A2.2.1 Drilling

- Drilling Rig (hollow-stem auger and air-rotary or air-hammer)

### A2.2.2 Surface Casing

- 6-inch to 12-inch ID steel or Schedule 80 PVC, depending upon conditions.
- Temporary bottom plug.
- Bentonite for grout: pure, additive-free powdered bentonite.
- Cement-Bentonite Grout. Proportion: 6 to 8 gallons of water per 94-pound bag of Portland cement; 3 to 6 pounds of bentonite added per bag of cement to reduce shrinkage.

### A2.2.3 Well Installation (if not completed as open borehole)

- Polyvinyl chloride (PVC), Schedule 40, minimum 2-inch ID, flush-threaded well riser: alternatively, stainless-steel riser.
- PVC, Schedule 40, minimum 2-inch ID, flush-threaded, factory slotted well screen: alternatively, stainless-steel screen.
- PVC or stainless steel bottom cap, threaded to match the well screen.
- Centering guides (if used). Same material as the casing, except stainless steel may be used in lieu of PVC.
- Clean silica sand, provided in factory-sealed bags, well-rounded, containing no organic material, anhydrite, gypsum, mica, or calcareous material; primary (coarse – e.g., Morie #1) filter pack, and secondary (fine sand seal) filter pack. Grain size determined based on sediments observed during drilling.
- Bentonite seal: Pure, additive-free bentonite pellets or chips.
- Bentonite for grout: Pure, additive-free powdered bentonite.
- Cement-Bentonite Grout: Proportion 6 to 8 gallons of water per 94-pound bag of Portland cement; 3 to 6 pounds of bentonite added per bag of cement to reduce shrinkage.

- Above-grade protective casing: Permanent isolation casing with heavy duty locking cover, painted with epoxy paint for rust protection, industrial lock.
- Flush-mount protective casing: Morrison 9-inch or 12-inch 519 manhole cover; rubber seal for cover; heavy duty locking cap on permanent isolation casing.

## A2.2.4 Development

- Surge block
- Pump and associated development equipment
- Calibrated meters to ensure pH, temperature, specific conductance, Eh, and dissolved oxygen of development water
- Containerization for water produced from well

## A2.3 Procedures and Guidelines

### A2.3.1 Drilling Methods

Boreholes for the bedrock monitoring wells will be drilled in several stages. Hollow-stem auger drilling and air-rotary or air-hammer drilling procedures are detailed below.

#### A2.3.1.1 Hollow Stem Auger Drilling through Unconsolidated Materials

Hollow stem auger drilling techniques may be used to drill boreholes for installation of surface isolation casing. Alternatively, mud-rotary drilling also may be used. 10-1/4-inch minimum ID HSA will be used to drill the borehole to competent bedrock.

The use of water or other fluid to assist in hollow stem drilling is to be avoided.

The bit of the auger is placed at the ground surface and then turned with the drilling rig. To collect split spoon samples, the auger is advanced to the top of the sampling depth, and the split-spoon sample is collected from below the auger head. The split spoon is advanced through repeated blows from a 140- or 300-pound hammer dropped from a height of 30 inches. Thin-walled tube samplers are advanced by pressing down on the rods with the weight of the drilling rig. Split-spoon samples may be collected at selected intervals for chemical analysis and/or lithologic classification.

The borehole will be advanced as deep as possible until auger refusal is met. Ultimately it will be necessary to extend the borehole a minimum of 5 feet into competent bedrock for seating of the surface casing. This will be addressed in the next section.

Soil brought to the surface on the outside of the augers should be containerized at a convenient space away from the working area. Soil may be stored on plastic sheeting and containerized at the completion of activities at the well cluster.

Drilling equipment will be decontaminated before drilling.

#### A2.3.1.2 Bedrock Drilling

The borehole will be advanced into the bedrock for a minimum of 5 feet using air rotary or air hammer. The borehole will be of sufficient diameter (at least 8 inches) so that a 6-inch or greater surface casing can be installed into the borehole. Once the borehole is drilled to at least 5 feet into the bedrock, the surface casing is installed per the procedure described in Section B below.

Air-rotary or air-hammer drilling techniques will be used to install wells in each of the bedrock monitoring boreholes. When the borehole is advanced beyond the 6-inch surface casing, the borehole will have a diameter of 5-7/8 inches.

The bit, drill rods, and other borehole rotary drilling equipment will be decontaminated prior to the initiation of drilling and between each borehole location.

Prior to the continuing air-rotary or air-hammer drilling in boreholes where a surface casing has been installed, the bit, drill rods, and other downhole rotary drilling equipment will be thoroughly decontaminated before being inserted into the borehole.

Drill cuttings and decontamination fluids generated during rotary drilling activities will be contained as IDW in accordance with DHEC requirements.

### A2.3.2 Surface Casing Installation

The surface casing will be constructed of 6-inch or greater ID black iron steel or PVC with a minimum wall thickness of 0.20 inches. Casing lengths will be welded or connected by threaded connections sealed with Teflon tape. The steel casing and threaded couplings must be free of paint, varnish, or coatings of any kind, both inside and outside. Threaded connections must be free of oils or grease. Welding of the casing is permissible provided that the welds meet the Standards of the American Welding Society.

Surface casing will be decontaminated prior to installation.

Permanent surface casing will be placed in the bottom of the borehole. The surface casing will be grouted in place by installing the grout through a tremie pipe from the bottom of the annulus to the ground surface.

Alternatively, the surface casing will be installed and grouted in place by a grout displacement method. The bottom of the surface casing is fitted with a tight, drillable plug. The borehole is then filled with the estimated volume of cement-bentonite grout to fill the annular space, and the casing is lowered to the bottom of the borehole (displacement method). If the weight of the casing is not sufficient to displace the grout and allow the casing to sink to the bottom of the borehole, the casing may be filled with clean water.

After the surface casing installation, the grout will be allowed to set up for at least 16-24 hours before drilling proceeds. All water in the surface casing will be removed before drilling resumes in order to prevent carrying contamination downward into deeper intervals of the water-bearing unit.

### A2.3.3 Monitoring Well Installation – Well Material Installation

Following the surface casing installation, the borehole will be completed with either air-rotary or air-hammer methods to the proper depth. If the borehole has been drilled to a depth greater than that at which the well is to be set, the borehole will be backfilled with bentonite pellets or chips or a bentonite-cement slurry to a depth approximately 1 foot below the intended well depth. Approximately 1 foot of clean sand will be placed on top of the bentonite to return the borehole to the proper depth for well installation.

The appropriate lengths of well screen, nominally 10 feet (with bottom cap), and casing will be joined watertight, and lowered to the bottom of the borehole. Centering guides, if used, will be placed at intervals around the well casing, at the base of the screen, and 5 feet above the top of the well screen.

Selection of final filter pack and well screen depths for the wells shall be made in the field.

A primary sand pack consisting of clean DSI No. 1 or equivalent silica sand for 10-slot screen and DSI No. 2 or equivalent for 20-slot screen silica sand will be placed around the well screen. The sand will be placed into the borehole at a uniform rate, in a manner that will allow even placement of the sand pack. During placement of the sand, the position of the top of the sand will be continuously sounded using a stainless steel weight attached to a fiberglass tape measure. The primary sand pack will be extended



from the bottom of the borehole to a minimum height of 2 feet above the top of the well screen. A secondary fine grained sand pack may then be installed to a minimum of 1 foot above the primary sand pack.

A non-additive bentonite seal (coated chips or pellets) at least 2 feet thick will be placed above the sand pack. The seal will be placed into the borehole in a manner that will prevent bridging. The position of the top of the bentonite seal will be verified using a weighted tape measure. A hydration period of at least 30 minutes will be allowed following installation of the bentonite seal.

Above the bentonite seal, an annular seal of cement-bentonite grout will be placed. The cement-bentonite grout will be installed continuously in one operation from the top of the bentonite seal to the ground surface. The cement-bentonite grout will be installed through a tremie pipe. The tremie pipe must be plugged at the bottom. Small openings along the bottom 1-foot length of pipe will allow the grout to diffuse laterally into the borehole and to avoid disturbance the bentonite seal.

#### A2.3.4 Monitoring Well Installation – Open Borehole

Following the surface casing installation, the borehole may be completed with either air-rotary or air-hammer methods to the proper depth. Instead of setting a PVC well screen and casing, the borehole may be left open to allow for downhole geophysics to be completed. This option is not feasible for weathered bedrock or bedrock with silted-in fractures that would allow the borehole to fill in with sediment over time. Once the borehole has been logged and the fracture zones identified, a PVC well screen may be installed within a discrete fracture zone if applicable as per instruction in Section C above.

#### A2.3.5 Monitoring-Well Completion

For monitoring wells that will be completed above-grade, the surface casing itself may serve as the protective casing. However, a separate steel protective casing may be used instead. The protective surface casing will be finished 2 to 3 feet above grade and fitted with a locking steel cap. A concrete pad with four guard posts will be installed.

The concrete pad will be square, approximately 2 feet per side (unless otherwise specified in the project plans), and poured into wooden forms. The concrete will be sloped away from the protective casing. The concrete pad will extend at least 6 inches below and 6 inches above the ground surface.

Four steel guard posts will be installed around the locking casing. Guard posts would be concrete-filled, at least 2 inches in diameter, and would extend at least 2 feet into the ground and 3 feet above the ground. The protective casing and guard posts will be painted with an epoxy paint to prevent rust.

For monitoring wells with flush-mount completions, a traffic rated steel manhole cover with a rubber-sealed cover and drain will be installed. The top of the manhole will be positioned approximately 1 inch above grade. A square concrete pad, approximately 2 feet per side (unless otherwise specified in the project plans), will be installed as a concrete collar surrounding the road box cover, and will slope uniformly downward to the adjacent grade. The road box and installation thereof will be of sufficient strength to withstand normal vehicular traffic. The concrete pad will extend at least 12 inches below the ground surface.

Inside the manhole, a locking cap will be placed over the permanent casing.

Each well will be labeled on the exterior of the locking cap with a metal stamp indicating the permanent well number.

### A2.3.6 Well Development

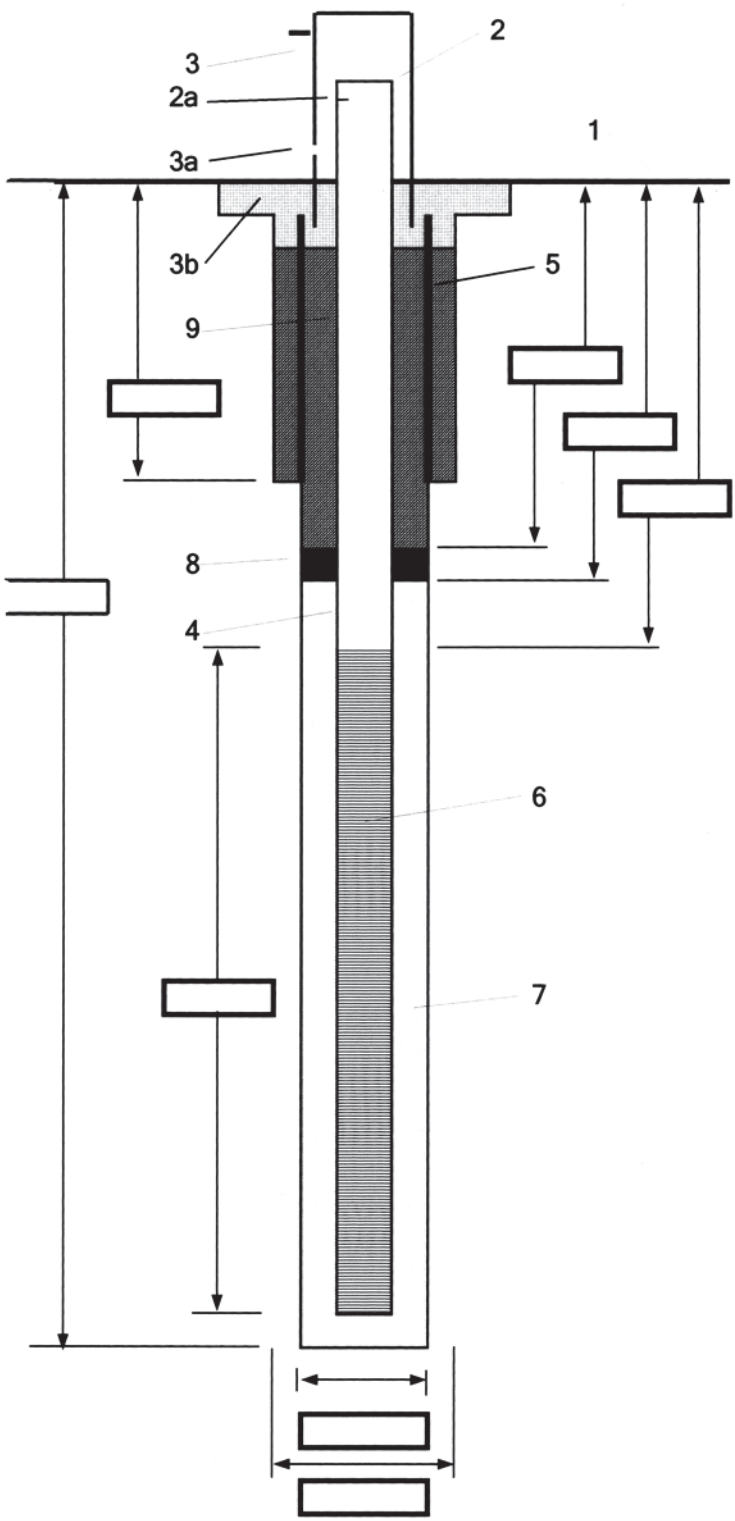
- New monitoring wells will be developed after the well has been completely installed and the grout has hardened (at least 24 hours)
- The well will be developed by surging and pumping.
- Equipment placed in the well will be decontaminated before use.
- If information is available, begin developing in the least-contaminated well first.
- Development will include surging the well by either abruptly stopping flow and allowing water in the well column to fall back into the well or through the use of a surge block that is slightly smaller in diameter than the well casing inner diameter.
- Pipes and pumps must not be fitted with foot valves or other devices that might inhibit the return flow of water to the well.
- Surging should continue throughout the development process.
- The air-lift method may be used to pump materials out of the well. The air compressor will be fitted with filters to remove all oil and the air lift hose used will be made of inert materials.
- Well development will continue until the water produced is free of turbidity, sand, and silt. A Horiba-U22 meter, YSI meter, or equivalent should be used to determine when the turbidity is low and parameters have stabilized.

## A2.4 Attachments

Schematic diagram of bedrock monitoring-well construction (MWDoublingDiag.xls)

# WELL COMPLETION DIAGRAM

PROJECT : \_\_\_\_\_ LOCATION : \_\_\_\_\_  
 DRILLING CONTRACTOR : \_\_\_\_\_  
 DRILLING METHOD AND EQUIPMENT USED : \_\_\_\_\_  
 WATER LEVELS : \_\_\_\_\_ START : \_\_\_\_\_ END : \_\_\_\_\_ LOGGER : \_\_\_\_\_



1- Ground elevation at well	_____
2- Top of casing elevation	_____
a) vent hole?	_____
3- Wellhead protection cover type	_____
a) weep hole?	_____
b) concrete pad dimensions	_____
4- Dia./type of well casing	_____
5- Dia./type of surface casing	_____
6- Type/slot size of screen	_____
7- Type screen filter	_____
a) Quantity used	_____
8- Type of seal	_____
a) Quantity used	_____
9- Grout	_____
a) Grout mix used	_____
b) Method of placement	_____
c) Vol. of surface casing grout	_____
d) Vol. of well casing grout	_____
Development method	_____
Development time	_____
Estimated purge volume	_____
Comments	_____
	_____
	_____
	_____
	_____

# Installation of Surface-Cased Monitoring Wells

## A3.1 Purpose and Scope

The purpose of this procedure is to outline equipment and methods that will be used to install surface casings to isolate shallow intervals from deeper drilling. The guideline only addresses installation in unconsolidated materials. Installation of monitoring wells in bedrock is discussed in *SOP Installation of Bedrock Monitoring Wells* and installation of shallow, single-cased monitoring wells is discussed in *SOP Installation of Shallow Monitoring Wells*. This SOP is consistent with the installation and completion of a surfaced-cased monitoring well per South Carolina Well Standards R61-71.

## A3.2 Equipment and Materials

### A3.2.1 Drilling

- Drilling rig (hollow stem auger, sonic, air rotary, or mud rotary).

### A3.2.2 Surface Casing

- 6-inch to 12-inch ID steel or Schedule 80 polyvinyl chloride (PVC) surface casing, depending upon application.
- Temporary bottom plug or grout shoe.
- Bentonite for grout: pure, additive-free powdered bentonite.
- Cement-Bentonite Grout. Proportion: 6 to 8 gallons of water per 94-pound bag of Portland cement; 3 pounds of bentonite added per bag of cement to reduce shrinkage.

### A3.2.3 Well Installation

- PVC, Schedule 40, minimum 2-inch ID, flush-threaded well riser; alternatively, stainless-steel riser.
- PVC, Schedule 40, minimum 2-inch ID, flush-threaded, factory slotted well screen; alternatively, stainless-steel screen.
- PVC or stainless steel bottom cap, threaded to match the well screen.
- Centering guides (if used); same material as the casing, except stainless steel may be used in lieu of PVC.
- Clean silica sand, provided in factory-sealed bags, well-rounded, containing no organic material, anhydrite, gypsum, mica, or calcareous material; primary (coarse – e.g., Morie #1) filter pack, and secondary (fine sand seal) filter pack. Grain size determined based on sediments observed during drilling.
- Bentonite seal: Pure, bentonite pellets or chips.

- Bentonite for grout: Pure, powdered bentonite.
- Cement-Bentonite Grout: Proportion 6 to 8 gallons of water per 94-pound bag of Portland cement; 3 pounds of bentonite added per bag of cement to reduce shrinkage.
- Above-grade well completion: PVC or stainless-steel well cap, threaded or push-on type, vented.
- Flush-mount well completion: PVC or stainless-steel well cap, locking, leak-proof seal.
- Above-grade protective casing: Permanent isolation casing with heavy duty locking cover, painted with epoxy paint for rust protection, industrial lock.
- Flush-mount protective casing: Morrison 9-inch or 12-inch 519 manhole cover; rubber seal for cover; heavy duty locking cap on permanent isolation casing.

### A3.2.4 Well Development

- Surge block.
- Pump and associated development equipment.
- Calibrated meters to measure pH, temperature, specific conductance, and turbidity of development water.
- Containerization for water produced from well.

## A3.3 Procedures and Guidelines

### A3.3.1 Drilling Method

Boreholes for the surface casing can be drilled with hollow-stem auger, air rotary, or mud rotary. Boreholes will be drilled to various diameters, depending upon applications. In the case of temporary surface casing, where grout will not be used to install the casing, the borehole for the casing will be drilled with a method by which the borehole will be as close to the diameter of the surface casing as possible to minimize the size of the annular space. This may be by a rotary method or by using a hollow-stem auger with as small an inside diameter as possible. For permanent surface casing, typically hollow-stem auger will be used.

#### A3.3.1.1 Hollow-Stem Auger Drilling

Hollow-stem auger (HSA) drilling techniques can be used to drill boreholes for installation of surface casing. The borehole will be drilled into a clay layer of significant thickness. Minimum 8-1/4-inch ID HSA will be used to drill the borehole a minimum of 5 feet into a clay layer of significant thickness.

The use of water or other fluid to assist in hollow-stem auger drilling is to be avoided.

The bit of the auger is placed at the ground surface and then turned with the drilling rig. To collect split- spoon or other samples, the auger is advanced to the top of the sampling depth, and the sample is collected from below the auger head. The split spoon is advanced through repeated blows from a 140- pound or 300-pound hammer dropped from a height of 30 inches. Thin-walled tube samplers are advanced by pressing down on the rods with the weight of the drilling rig. Split-spoon samples will be collected at selected intervals for chemical analysis and/or lithologic classification. Soil sampling procedures are detailed in SOPs *Soil Boring Sampling – Split Spoons* and *Soil Sampling*.

Material brought to the surface on the outside of the augers should be containerized at a convenient space away from the working area. Material may be stored on plastic sheeting and containerized at the completion of activities at the drilling location. Such material will be disposed of according to SOP *Disposal of Waste Fluids and Solids* and the Investigation Derived Waste Management Plan (IDWMP).

Drilling equipment will be decontaminated before drilling in accordance with the procedures detailed in *SOP Decontamination of Drilling Rigs and Equipment*.

#### A3.3.1.2 Rotary Drilling

Mud rotary or other rotary drilling techniques can be used to install surface casing.

To collect split spoon samples, the drill bit is advanced to the depth to be sampled, the bit is removed from the borehole, and the split-spoon sample is collected from the borehole.

Drill cuttings and fluids generated during rotary drilling activities will be contained until completion of activities at the drilling location. Such material will be disposed of according to *SOP Disposal of Waste Fluids and Solids* and the IDWMP.

Drilling equipment will be decontaminated before drilling in accordance with the procedures detailed in *SOP Decontamination of Drilling Rigs and Equipment*.

### A3.3.2 Surface-Casing Installation

Surface casing will be constructed of minimum 6-inch ID or greater black iron steel with a minimum wall thickness of 0.20 inches or Schedule 80 PVC. Casing lengths for permanent surface casings will be welded or connected by threaded connections sealed with Teflon tape, while casing lengths for temporary surface casings will be either threaded connections of casing installed as part of the rotary-drilling method. The steel casing and threaded couplings must be free of paint, varnish, or coatings of any kind, both inside and outside. Threaded connections must be free of oils or grease. Welding of the casing is permissible provided that the welds meet the Standards of the American Welding Society.

Surface casing will be decontaminated prior to installation in accordance with the procedures detailed in *SOP Decontamination of Drilling Rigs and Equipment*.

Permanent surface casing will be placed in the bottom of the borehole and the surface casing will be pressed into a low permeability layer, if present. The surface casing will be grouted in place by installing the grout through a tremie pipe connected to the grout shoe, or placed at the bottom of the annulus.

Alternatively, the surface casing may be installed and grouted in place by a grout displacement method. The bottom of the surface casing is fitted with a tight, drillable plug. The borehole is then filled with the estimated volume of cement-bentonite grout to fill the annular space, and the casing is lowered to the bottom of the borehole (displacement method). If the weight of the casing is not sufficient to displace the grout and allow the casing to sink to the bottom of the borehole, the casing may be filled with clean water.

Well installation will proceed inside the surface casing once the grout has been allowed to cure for approximately 16 to 24 hours. All water in the surface casing will be removed before drilling resumes in order to prevent carrying contamination downward into deeper intervals of the water-bearing unit. All water in the casing will be disposed of according to *SOP Disposal of Waste Fluids and Solids* and the IDWMP before drilling is resumed.

Temporary surface casing installed independently of the drilling process also will be placed in the bottom of the borehole and the surface casing will be pressed into a low permeability layer, if present. However, it will not be grouted into place.

### A3.3.3 Monitoring-Well Installation

Typically, the well is completed within the surface casing using mud rotary techniques. Sonic drilling methods may also be used through permanent surface casings.

If the borehole has been drilled to a depth greater than that at which the well is to be completed, the borehole will be backfilled with bentonite pellets or chips or a bentonite-cement slurry to a depth

approximately 1 foot below the intended well depth. Approximately 1 foot of clean sand will be placed on top of the bentonite to return the borehole to the correct depth for well installation.

The appropriate lengths of well screen, nominally 10 feet (with bottom cap), and casing will be lowered to the bottom of the borehole. Centering guides, if used, will be placed at intervals around the well casing, at the base of the screen, and 5 feet above the top of the well screen.

Selection of final filter pack and well screen depths for the wells shall be made in the field.

A primary sand pack consisting of clean Morie No. 00 (or DSI No. 1) silica sand for 0.010-inch slotted screen and Morie No. 01 (or DSI No. 2) silica sand for 0.020-inch slotted screen will be placed around the well screen. The sand will be placed into the borehole at a uniform rate, in a manner that will allow even placement of the sand pack. During placement of the sand, the position of the top of the sand will be continuously sounded using a stainless steel weight attached to a tape measure. The primary sand pack will extend from the bottom of the borehole to a minimum of 2 feet above the top of the well screen. A secondary (fine sand seal) sand pack will then be installed to a minimum of 1 foot above the primary sand pack.

A bentonite seal at least 2 feet thick will be placed above the sand pack. The seal will be placed into the borehole in a manner that will prevent bridging. The position of the top of the bentonite seal will be verified using a weighted tape measure. A hydration period of at least 30 minutes will be allowed following installation of the bentonite seal.

Above the bentonite seal, an annular seal of cement-bentonite grout will be placed. The cement-bentonite grout will be installed continuously in one operation from the top of the bentonite seal to the ground surface. The cement-bentonite grout will be installed through a side-discharge tremie pipe plugged at the bottom. The openings in the tremie pipe will allow the grout to discharge laterally into the borehole and avoid disturbance the bentonite seal.

### A3.3.4 Monitoring-Well Completion

For monitoring wells that will be completed above-grade, the surface casing itself may serve as the protective casing. However, a separate steel protective casing may be used instead. The protective surface casing will be finished 2 to 3 feet above grade and fitted with a locking steel cap. A concrete pad with four guard posts will be installed.

The concrete pad will be square, approximately 2 feet per side (unless otherwise specified in the project plans), and poured into wooden forms. The concrete will be sloped away from the protective casing. The concrete pad will extend at least 6 inches below and 2 inches above the ground surface.

Four steel guard posts will be installed around the locking casing. Guard posts shall be concrete-filled, at least 2 inches in diameter, and extend at least 2 feet into the ground and 3 feet above the ground. The protective casing and guard posts will be painted with an epoxy paint to prevent rust.

For monitoring wells with flush-mount completions, an 8 to 12-inch dia. manhole cover with a rubber gasket and drain will be installed. The top of the manhole will be positioned approximately 1 inch above grade. A square concrete pad, approximately 2 feet per side (unless otherwise specified in the project plans), will be installed as a concrete collar surrounding the road box cover, and will slope uniformly downward to the adjacent grade. The road box and installation thereof will be of sufficient strength to withstand normal vehicular traffic. The concrete pad will extend at least 6 inches below the ground surface.

Inside the manhole, a locking cap will be placed on the inner well casing.

Each well will be labeled on the exterior of the locking cap with a metal stamp indicating the permanent well number.

### A3.3.5 Well Development

Well development will be accomplished using a combination of surging throughout the well screen and pumping, until the physical and chemical parameters of the discharge water that are measured in the field have stabilized and the turbidity of the discharge water is substantially reduced. Fine-grained materials in the surficial aquifer may not allow low turbidity results to be achieved.

The surging apparatus will include a tight-fitting surge block. Well development will begin by surging the well screen, starting at the bottom of the screen and proceeding upwards, throughout the screened zone. Following surging, the well will be pumped to remove the fine materials that have been drawn into the well. During pumping, measurements of pH, temperature, turbidity, and specific conductance will be recorded.

Development will continue by alternately surging and pumping until the discharge water is free from sand and silt, the turbidity is substantially reduced, and the pH, temperature, and specific conductance have stabilized at regional background levels, based on historical data. Development will continue for a minimum of one hour and until the water removed from the well is as clear of turbidity as practicable.

Well development equipment will be decontaminated prior to initial use and after the development of each well. Decontamination procedures are detailed in *SOP Decontamination of Personnel and Equipment*. Water generated during well development will be contained and managed as detailed in the *SOP Disposal of Waste Fluids and Solids* and the IDWMP.

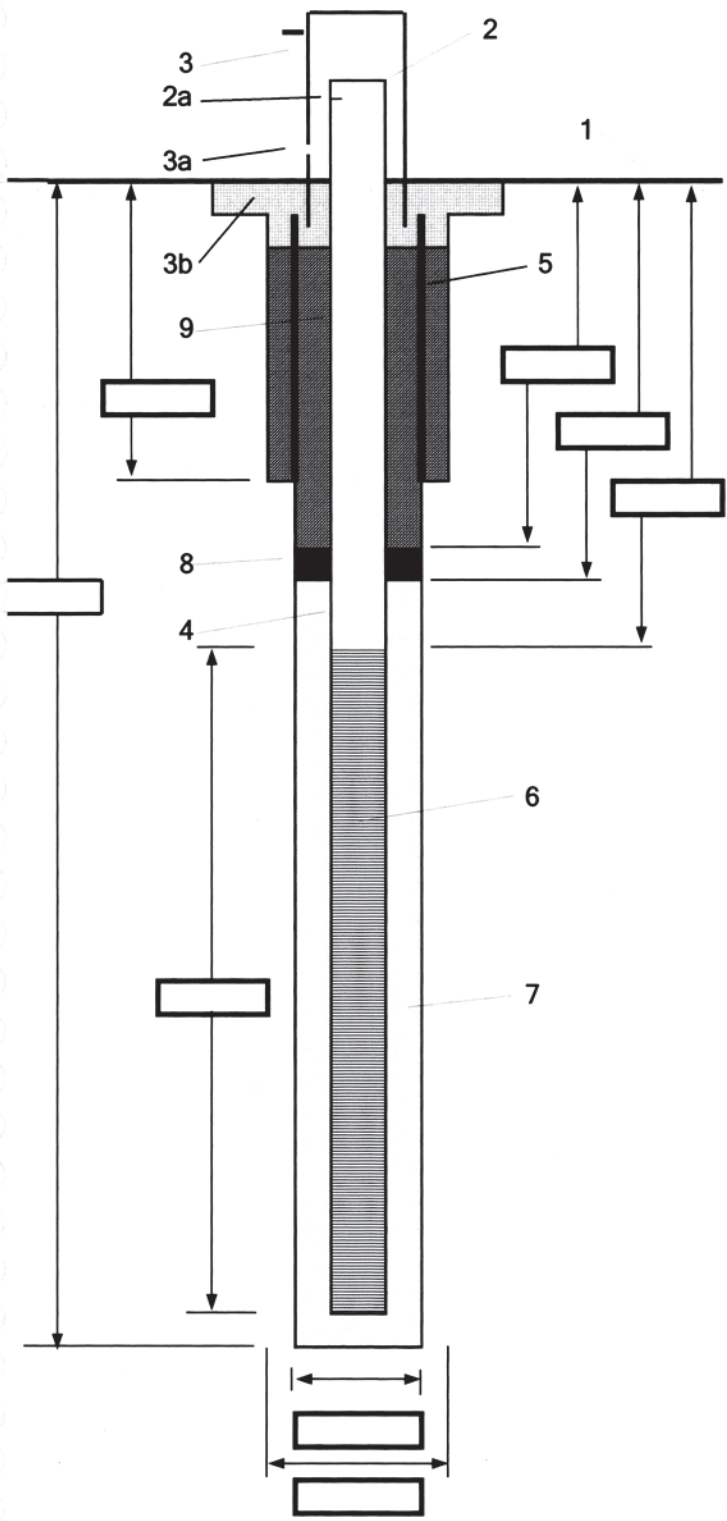
## A3.4 Attachments

Schematic diagram of double-cased monitoring-well construction (MWDoublingDiag.xls)



# WELL COMPLETION DIAGRAM

SUBJECT : \_\_\_\_\_ LOCATION : \_\_\_\_\_  
 DRILLING CONTRACTOR : \_\_\_\_\_  
 DRILLING METHOD AND EQUIPMENT USED : \_\_\_\_\_  
 WATER LEVELS : \_\_\_\_\_ START : \_\_\_\_\_ END : \_\_\_\_\_ LOGGER : \_\_\_\_\_



- 1- Ground elevation at well \_\_\_\_\_
- 2- Top of casing elevation  
a) vent hole? \_\_\_\_\_
- 3- Wellhead protection cover type  
a) weep hole? \_\_\_\_\_  
b) concrete pad dimensions \_\_\_\_\_
- 4- Dia./type of well casing \_\_\_\_\_
- 5- Dia./type of surface casing \_\_\_\_\_
- 6- Type/slot size of screen \_\_\_\_\_
- 7- Type screen filter  
a) Quantity used \_\_\_\_\_
- 8- Type of seal  
a) Quantity used \_\_\_\_\_
- 9- Grout  
a) Grout mix used \_\_\_\_\_  
b) Method of placement \_\_\_\_\_  
c) Vol. of surface casing grout \_\_\_\_\_  
d) Vol. of well casing grout \_\_\_\_\_

Development method \_\_\_\_\_

Development time \_\_\_\_\_

Estimated purge volume \_\_\_\_\_

Comments \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

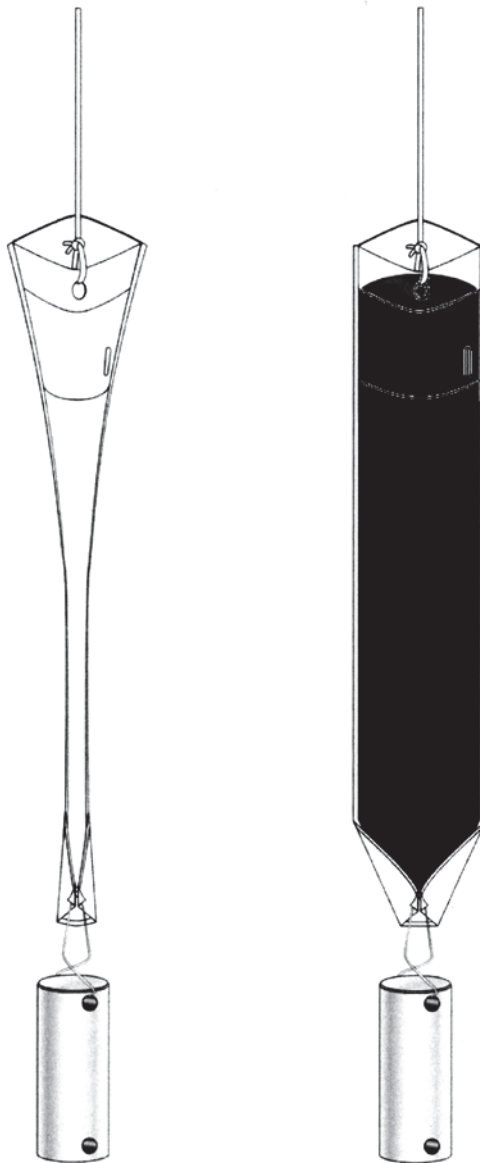
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# HYDRASleeve™

Simple by Design US Patent No. 6,481,300; No. 6,837,120 others pending

## Standard Operating Procedure: Sampling Ground Water with a HydraSleeve



This Guide should be used in addition to field manuals appropriate to sampling device (i.e., HydraSleeve or Super Sleeve).

Find the appropriate field manual on the HydraSleeve website at <http://www.hydrasleeve.com>.

For more information about the HydraSleeve, or if you have questions, contact: GeoInsight, 2007 Glass Road, Las Cruces, NM 88005, 1-800-996-2225, [info@hydrasleeve.com](mailto:info@hydrasleeve.com).

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## **Introduction**

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The HydraSleeve is classified as a no-purge (passive) grab sampling device, meaning that it is used to collect ground-water samples directly from the screened interval of a well without having to purge the well prior to sample collection. When it is used as described in this Standard Operating Procedure (SOP), the HydraSleeve causes no drawdown in the well (until the sample is withdrawn from the water column) and only minimal disturbance of the water column, because it has a very thin cross section and it displaces very little water (<100 ml) during deployment in the well. The HydraSleeve collects a sample from within the screen only, and it excludes water from any other part of the water column in the well through the use of a self-sealing check valve at the top of the sampler. It is a single-use (disposable) sampler that is not intended for reuse, so there are no decontamination requirements for the sampler itself.

The use of no-purge sampling as a means of collecting representative ground-water samples depends on the natural movement of ground water (under ambient hydraulic head) from the formation adjacent to the well screen through the screen. Robin and Gillham (1987) demonstrated the existence of a dynamic equilibrium between the water in a formation and the water in a well screen installed in that formation, which results in formation-quality water being available in the well screen for sampling at all times. No-purge sampling devices like the HydraSleeve collect this formation-quality water as the sample, under undisturbed (non-pumping) natural flow conditions. Samples collected in this manner generally provide more conservative (i.e., higher concentration) values than samples collected using well-volume purging, and values equivalent to samples collected using low-flow purging and sampling (Parsons, 2005).

## **Applications of the HydraSleeve**

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The HydraSleeve can be used to collect representative samples of ground water for all analytes (volatile organic compounds [VOCs], semi-volatile organic compounds [SVOCs], common metals, trace metals, major cations and anions, dissolved gases, total dissolved solids, radionuclides, pesticides, PCBs, explosive compounds, and all other analytical parameters). Designs are available to collect samples from wells from 1" inside diameter and larger. The HydraSleeve can collect samples from wells of any yield, but it is especially well-suited to collecting samples from low-yield wells, where other sampling methods can't be used reliably because their use results in dewatering of the well screen and alteration of sample chemistry (McAlary and Barker, 1987).

The HydraSleeve can collect samples from wells of any depth, and it can be used for single-event sampling or long-term ground-water monitoring programs. Because of its thin cross section and flexible construction, it can be used in narrow, constricted or damaged wells where rigid sampling devices may not fit. Using multiple HydraSleeves deployed in series along a single suspension line or tether, it is also possible to conduct in-well vertical profiling in wells in which contaminant concentrations are thought to be stratified.

As with all groundwater sampling devices, HydraSleeves should not be used to collect groundwater samples from wells in which separate (non-aqueous) phase hydrocarbons (i.e., gasoline, diesel fuel or jet fuel) are present because of the possibility of incorporating some of the separate-phase hydrocarbon into the sample.

## Description of the HydraSleeve

The HydraSleeve (Figure 1) consists of the following basic components:

- A suspension line or tether (A.), attached to the spring clip or directly to the top of the sleeve to deploy the device into and recover the device from the well. Tethers with depth indicators marked in 1-foot intervals are available from the manufacturer.
- A long, flexible, 4-mil thick lay-flat polyethylene sample sleeve (C.) sealed at the bottom (this is the sample chamber), which comes in different sizes, as discussed below with a self-sealing reed-type flexible polyethylene check valve built into the top of the sleeve (B.) to prevent water from entering or exiting the sampler except during sample acquisition.
- A reusable stainless-steel weight with clip (D.), which is attached to the bottom of the sleeve to carry it down the well to its intended depth in the water column. Bottom weights available from the manufacturer are 0.75" OD and are available in three sizes: 5 oz. (2.5" long); 8 oz. (4" long); and 16 oz. (8" long). In lieu of a bottom weight, an optional top weight may be attached to the top of the HydraSleeve to carry it to depth and to compress it at the bottom of the well (not shown in Figure 1);
- A discharge tube that is used to puncture the HydraSleeve after it is recovered from the well so the sample can be decanted into sample bottles (not shown).
- Just above the self-sealing check valve at the top of the sleeve are two holes which provide attachment points for the spring clip and/or suspension line or tether. At the bottom of the sample sleeve are two holes which provide attachment points for the weight clip and weight.

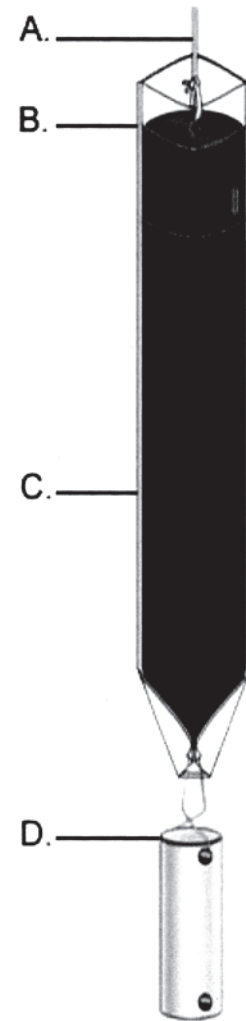


Figure 1. HydraSleeve components.

**Note:** The sample sleeve and the discharge tube are designed for one-time use and are disposable. The spring clip, weight and weight clip may be reused after thorough cleaning. Suspension cord is generally disposed after one use although, if it is dedicated to the well, it may be reused at the discretion of the sampling personnel.

## Selecting the HydraSleeve Size to Meet Site-Specific Sampling Objectives

It is important to understand that each HydraSleeve is able to collect a finite volume of sample because, after the HydraSleeve is deployed, you only get one chance to collect an undisturbed sample. Thus, the volume of sample required to meet your site-specific sampling and analytical requirements will dictate the size of HydraSleeve you need to meet these requirements.

The volume of sample collected by the HydraSleeve varies with the diameter and length of the HydraSleeve. Dimensions and volumes of available HydraSleeve models are detailed in Table 1.

**Table 1. Dimensions and volumes of HydraSleeve models.**

Diameter	Volume	Length	Lay-Flat Width	Filled Dia.
<i>2-Inch HydraSleeves</i>				
Standard 625-ml HydraSleeve	625 ml	< 30"	2.5"	1.4"
Standard 1-Liter HydraSleeve	1 Liter	38"	3"	1.9"
1-Liter HydraSleeve SS	1 Liter	36"	3"	1.9"
2-Liter HydraSleeve SS	2 Liters	60"	3"	1.9"
<i>4-Inch HydraSleeves</i>				
Standard 1.6-Liter HydraSleeve	1.6 Liters	30"	3.8"	2.3"
Custom 2-Liter HydraSleeve	2 Liters	36"	4"	2.7"

HydraSleeves can be custom-fabricated by the manufacturer in varying diameters and lengths to meet specific volume requirements. HydraSleeves can also be deployed in series (i.e., multiple HydraSleeves attached to one tether) to collect additional sample to meet specific volume requirements, as described below.

If you have questions regarding the availability of sufficient volume of sample to satisfy laboratory requirements for analysis, it is recommended that you contact the laboratory to discuss the minimum volumes needed for each suite of analytes. Laboratories often require only 10% to 25% of the volume they specify to complete analysis for specific suites of analytes, so they can often work with much smaller sample volumes that can easily be supplied by a HydraSleeve.

## **HydraSleeve Deployment**

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### **Information Required Before Deploying a HydraSleeve**

Before installing a HydraSleeve in any well, you will need to know the following:

- The inside diameter of the well
- The length of the well screen
- The water level in the well
- The position of the well screen in the well
- The total depth of the well

The inside diameter of the well is used to determine the appropriate HydraSleeve diameter for use in the well. The other information is used to determine the proper placement of the HydraSleeve in the well to collect a representative sample from the screen (see HydraSleeve Placement, below), and to determine the appropriate length of tether to attach to the HydraSleeve to deploy it at the appropriate position in the well.

Most of this information (with the exception of the water level) should be available from the well log; if not, it will have to be collected by some other means. The inside diameter of the well can be measured at the top of the well casing, and the total depth of the well can be measured by sounding the bottom of the well with a weighted tape. The position and length of the well screen may have to be determined using a down-hole camera if a well log is not available. The water level in the well can be measured using any commonly available water-level gauge.



## HydraSleeve Placement

The HydraSleeve is designed to collect a sample directly from the well screen, and it fills by pulling it up through the screen a distance equivalent to 1 to 1.5 times its length. This upward motion causes the top check valve to open, which allows the device to fill. To optimize sample recovery, it is recommended that the HydraSleeve be placed in the well so that the bottom weight rests on the bottom of the well and the top of the HydraSleeve is as close to the bottom of the well screen as possible. This should allow the sampler to fill before the top of the device reaches the top of the screen as it is pulled up through the water column, and ensure that only water from the screen is collected as the sample. In short-screen wells, or wells with a short water column, it may be necessary to use a top-weight on the HydraSleeve to compress it in the bottom of the well so that, when it is recovered, it has room to fill before it reaches the top of the screen.

### Example

2" ID PVC well, 50' total depth, 10' screen at the bottom of the well, with water level above the screen (the entire screen contains water).

*Correct Placement (figure 2):* Using a standard HydraSleeve for a 2" well (2.6" flat width/1.5" filled OD x 30" long, 650 ml volume), deploy the sampler so the weight (an 8 oz., 4"-long weight with a 2"-long clip) rests at the bottom of the well. The top of the sleeve is thus set at about 36" above the bottom of the well. When the sampler is recovered, it will be pulled upward approximately 30" to 45" before it is filled; therefore, it is full (and the top check valve closes) at approximately 66" (5 1/2 feet) to 81" (6 3/4 feet) above the bottom of the well, which is well before the sampler reaches the top of the screen. In this example, only water from the screen is collected as a sample.

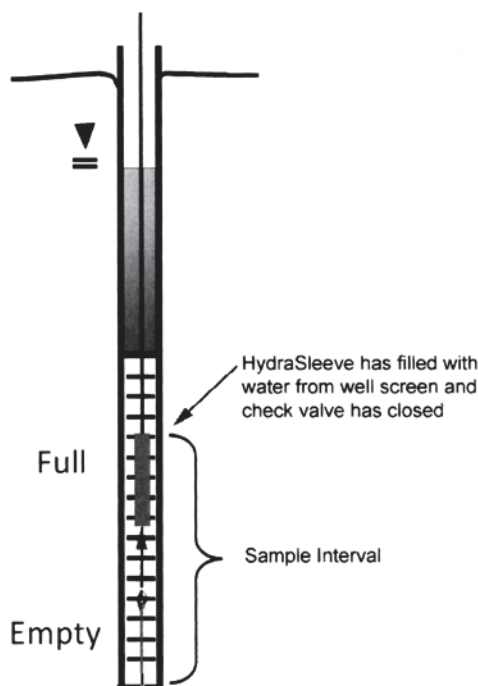


Figure 2. Correct placement of HydraSleeve.

*Incorrect Placement (figure 3):* If the well screen in this example was only 5' long, and the HydraSleeve was placed as above, it would not fill before the top of the device reached the top of the well screen, so the sample would include water from above the screen, which may not have the same chemistry.

*The solution?* Deploy the HydraSleeve with a top weight, so that it is collapsed to within 6" to 9" of the bottom of the well. When the HydraSleeve is recovered, it will fill within 39" (3 ¼ feet) to 54" (4 ½ feet) above the bottom of the well, or just before the sampler reaches the top of the screen, so it collects only water from the screen as the sample.

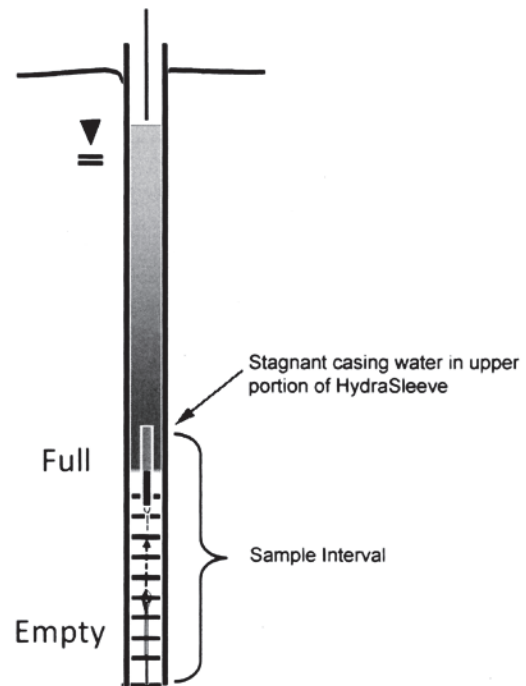


Figure 3. Incorrect placement of HydraSleeve.

This example illustrates one of many types of HydraSleeve placements. More complex placements are discussed in a later section.

## Procedures for Sampling with the HydraSleeve

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Collecting a ground-water sample with a HydraSleeve is a simple one-person operation.

**Note:** Before deploying the HydraSleeve in the well, collect the depth-to-water measurement that you will use to determine the preferred position of the HydraSleeve in the well. This measurement may also be used with measurements from other wells to create a ground-water contour map. If necessary, also measure the depth to the bottom of the well to verify actual well depth to confirm your decision on placement of the HydraSleeve in the water column.

Measure the correct amount of tether needed to suspend the HydraSleeve in the well so that the weight will rest on the bottom of the well (or at your preferred position in the well). Make sure to account for the need to leave a few feet of tether at the top of the well to allow recovery of the sleeve

**Note:** Always wear sterile gloves when handling and discharging the HydraSleeve.

### I. Assembling the HydraSleeve

1. Remove the HydraSleeve from its packaging, unfold it, and hold it by its top.
2. Crimp the top of the HydraSleeve by folding the hard polyethylene reinforcing strips at the holes.
3. Attach the spring clip to the holes to ensure that the top will remain open until the sampler is retrieved.
4. Attach the tether to the spring clip by tying a knot in the tether.

**Note:** Alternatively, attach the tether to one (NOT both) of the holes at the top of the Hydrasleeve by tying a knot in the tether.

5. Fold the flaps with the two holes at the bottom of the HydraSleeve together and slide the weight clip through the holes.
6. Attach a weight to the bottom of the weight clip to ensure that the HydraSleeve will descend to the bottom of the well.

## II. Deploying the HydraSleeve

1. Using the tether, carefully lower the HydraSleeve to the bottom of the well, or to your preferred depth in the water column

During installation, hydrostatic pressure in the water column will keep the self-sealing check valve at the top of the HydraSleeve closed, and ensure that it retains its flat, empty profile for an indefinite period prior to recovery.

**Note:** Make sure that it is not pulled upward at any time during its descent. If the HydraSleeve is pulled upward at a rate greater than 0.5'/second at any time prior to recovery, the top check valve will open and water will enter the HydraSleeve prematurely.

2. Secure the tether at the top of the well by placing the well cap on the top of the well casing and over the tether.

**Note:** Alternatively, you can tie the tether to a hook on the bottom of the well cap (you will need to leave a few inches of slack in the line to avoid pulling the sampler up as the cap is removed at the next sampling event).

## III. Equilibrating the Well

The equilibration time is the time it takes for conditions in the water column (primarily flow dynamics and contaminant distribution) to restabilize after vertical mixing occurs (caused by installation of a sampling device in the well).

- Situation: The HydraSleeve is deployed for the first time or for only one time in a well

The HydraSleeve is very thin in cross section and displaces very little water (<100 ml) during deployment so, unlike most other sampling devices, it does not disturb the water column to the point at which long equilibration times are necessary to ensure recovery of a representative sample.

In most cases, the HydraSleeve can be recovered immediately (with no equilibration time) or within a few hours. In regulatory jurisdictions that impose specific requirements for equilibration times prior to recovery of no-purge sampling devices, these requirements should be followed.

- Situation: The HydraSleeve is being deployed for recovery during a future sampling event

In periodic (i.e., quarterly or semi-annual) sampling programs, the sampler for the current sampling event can be recovered and a new sampler (for the next sampling event)

deployed immediately thereafter, so the new sampler remains in the well until the next sampling event.

Thus, a long equilibration time is ensured and, at the next sampling event, the sampler can be recovered immediately. This means that separate mobilizations, to deploy and then to recover the sampler, are not required. HydraSleeves can be left in a well for an indefinite period of time without concern.

#### **IV. HydraSleeve Recovery and Sample Collection**

1. Hold on to the tether while removing the well cap.
2. Secure the tether at the top of the well while maintaining tension on the tether (but without pulling the tether upwards)
3. Measure the water level in the well.
4. In one smooth motion, pull the tether up between 30" to 45" (36" to 54" for the longer HydraSleeve) at a rate of about 1' per second (or faster).

The motion will open the top check valve and allow the HydraSleeve to fill (it should fill in about 1 to 1.5 times the length of the HydraSleeve). This is analogous to coring the water column in the well from the bottom up.

When the HydraSleeve is full, the top check valve will close. You should begin to feel the weight of the HydraSleeve on the tether and it will begin to displace water. The closed check valve prevents loss of sample and entry of water from zones above the well screen as the HydraSleeve is recovered.

5. Continue pulling the tether upward until the HydraSleeve is at the top of the well.
6. Decant and discard the small volume of water trapped in the Hydrasleeve above the check valve by turning the sleeve over.

#### **V. Sample Collection**

**Note:** Sample collection should be done immediately after the HydraSleeve has been brought to the surface to preserve sample integrity.

1. Remove the discharge tube from its sleeve.
2. Hold the HydraSleeve at the check valve.
3. Puncture the HydraSleeve just below the check valve with the pointed end of the discharge tube
4. Discharge water from the HydraSleeve into your sample containers.

Control the discharge from the HydraSleeve by either raising the bottom of the sleeve, by squeezing it like a tube of toothpaste, or both.

5. Continue filling sample containers until all are full.

## Measurement of Field Indicator Parameters

Field indicator parameter measurement is generally done during well purging and sampling to confirm when parameters are stable and sampling can begin. Because no-purge sampling does not require purging, field indicator parameter measurement is not necessary for the purpose of confirming when purging is complete.

If field indicator parameter measurement is required to meet a specific non-purging regulatory requirement, it can be done by taking measurements from water within a HydraSleeve that is not used for collecting a sample to submit for laboratory analysis (i.e., a second HydraSleeve installed in conjunction with the primary sample collection HydraSleeve [see Multiple Sampler Deployment below]).

## Alternate Deployment Strategies

### Deployment in Wells with Limited Water Columns

For wells in which only a limited water column exists to be sampled, the HydraSleeve can be deployed with an optional top weight instead of a bottom weight, which collapses the HydraSleeve to a very short (approximately 6" to 9") length, and allows the HydraSleeve to fill in a water column only 36" to 45" in height.

### Multiple Sampler Deployment

Multiple sampler deployment in a single well screen can accomplish two purposes:

- It can collect additional sample volume to satisfy site or laboratory-specific sample volume requirements.
- It can accommodate the need for collecting field indicator parameter measurements.
- It can be used to collect samples from multiple intervals in the screen to allow identification of possible contaminant stratification.

It is possible to use up to 3 standard 30" HydraSleeves deployed in series along a single tether to collect samples from a 10' long well screen without collecting water from the interval above the screen.

The samplers must be attached to the tether at both the top and bottom of the sleeve. Attach the tether at the top with a stainless-steel clip (available from the manufacturer). Attach the tether at the bottom using a cable tie. The samplers must be attached as follows (figure 4):

- The first (attached to the tether as described above, with the weight at the bottom) at the bottom of the screen
- The second attached immediately above the first
- The third (attached the same as the second) immediately above the second

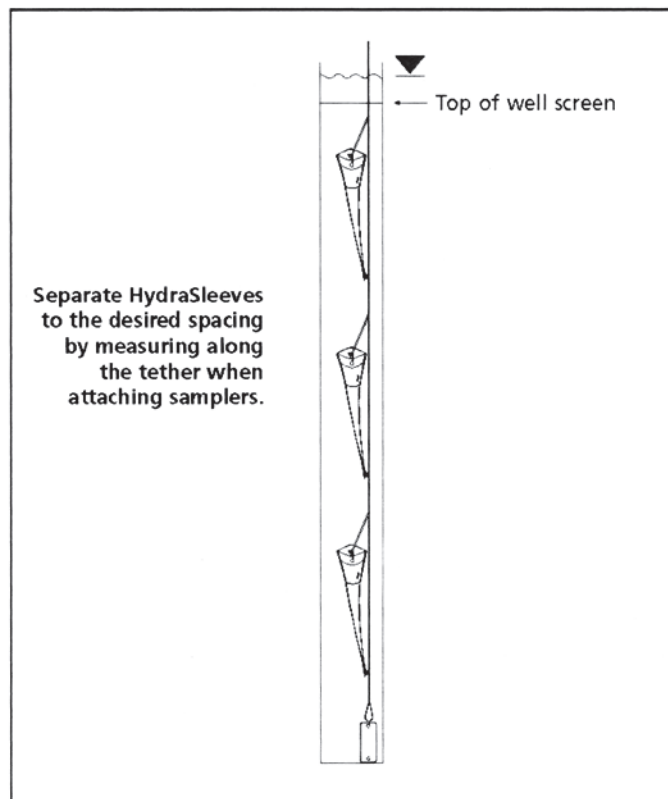


Figure 4. Multiple HydraSleeve deployment.

Alternately, the first sampler can be attached to the tether as described above, a second attached to the bottom of the first using a short length of tether (in place of the weight), and the third attached to the bottom of the second in the same manner, with the weight attached to the bottom of the third sampler (figure 5).

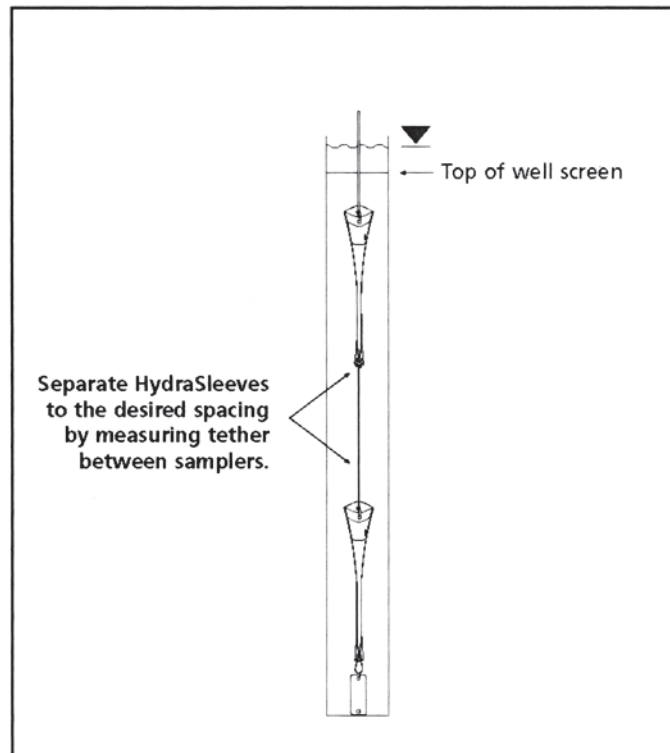


Figure 5. Alternative method for deploying multiple HydraSleeves.

In either case, when attaching multiple HydraSleeves in series, more weight may be required to hold the samplers in place in the well than would be required with a single sampler. Recovery of multiple samplers and collection of samples is done in the same manner as for single sampler deployments.



## **Post-Sampling Activities**

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The recovered HydraSleeve and the sample discharge tubing should be disposed as per the solid waste management plan for the site. To prepare for the next sampling event, a new HydraSleeve can be deployed in the well (as described previously) and left in the well until the next sampling event, at which time it can be recovered.

The weight and weight clip can be reused on this sampler after they have been thoroughly cleaned as per the site equipment decontamination plan. The tether may be dedicated to the well and reused or discarded at the discretion of sampling personnel.

## References

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McAlary, T. A. and J. F. Barker, 1987, Volatilization Losses of Organics During Ground-Water Sampling From Low-Permeability Materials, Ground-Water Monitoring Review, Vol. 7, No. 4, pp. 63-68

Parsons, 2005, Results Report for the Demonstration of No-Purge Ground-Water Sampling Devices at Former McClellan Air Force Base, California; Contract F44650-99-D-0005, Delivery Order DKO1, U.S. Army Corps of Engineers (Omaha District), U.S. Air Force Center for Environmental Excellence, and U.S. Air Force Real Property Agency

Robin, M. J. L. and R. W. Gillham, 1987, Field Evaluation of Well Purging Procedures, Ground-Water Monitoring Review, Vol. 7, No. 4, pp. 85-93

# Low-Flow Groundwater Sampling from Monitoring Wells – EPA Region IV

## A5.1 Purpose and Scope

This SOP presents general guidelines for the collection of groundwater samples from monitoring wells using low-flow purging and sampling procedures, following EPA guidance detailed in *Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures* (EPA, 1996) and *Region 4 Science and Ecosystem Support Division (SESD) Operating Procedure (OP) for Groundwater Sampling, SESDPROC-301-R3*. (EPA, 2013).

## A5.2 Equipment and Materials

- Adjustable-rate positive-displacement pump, submersible pump, or peristaltic pump
- Horiba® U-22 or equivalent water quality meter to monitor pH, specific conductance, turbidity, dissolved oxygen, oxidation-reduction potential (ORP), and temperature
- Air monitoring equipment
- Personal protective equipment
- Flow-through cell with inlet/outlet ports and watertight ports for each probe
- Generator or alternate power source depending on pump type
- Water-level indicator
- Disposable Teflon, Teflon-lined polyethylene tubing or polyethylene tubing for metals and other inorganics
- Plastic sheeting
- Well-construction information
- Calibrated container and stopwatch to determine flow rate
- Purged water containers
- Sample containers
- Waste container labels
- In-line disposable 0.45 µm filters (QED® FF8100 or equivalent)
- Shipping supplies (labels, coolers, and ice)
- Aluminum foil
- Field book

## A5.3 Procedures and Guidelines

### A5.3.1 Setup and Purging

- Obtain information on well location, diameter(s), depth, and screen interval(s), and the method for disposal of purged water.
- Calibrate instruments according to manufacturer's instructions.
- Record well number, site, date, and condition in the field logbook.
- Place plastic sheeting on the ground surrounding well head. All decontaminated equipment to be used in sampling will be placed only on the plastic sheeting until after the sampling has been completed. Do not let any downhole equipment touch the ground.
- Open the well and begin screening breathing zone with the air monitoring device until sampling is complete, in accordance with the HASP.
- All sampling equipment and any other equipment to be placed in the well is cleaned and decontaminated before sampling in accordance with SOP *Decontamination of Personnel and Equipment*.
- Water level measurements are collected in accordance with the *Water Level Measurements* SOP. **Do not measure the depth to the bottom of the well at this time**; this reduces the possibility that any accumulated sediment in the well will be disturbed. Obtain depth to bottom information from well construction log.
- Attach and secure the tubing to the pump. Lower the pump slowly into the well and set it at approximately the middle of the screen, or wetted screen interval, and at least two feet above the bottom of the well to avoid disturbance of sediment. Submersible pumps should be lowered by the suspension cable rather than the discharge tubing.
- Insert the water quality measurement probes into the flow-through cell and place in a shaded area. The purged groundwater must enter the flow through the cell by the lower port and exit via the upper port. Wrap exposed tubing and the flow through cell in aluminum foil to minimize heat loss/gain due to environmental conditions.
- Generators and fuel, if used, must be located at least 30 feet downwind from the well to avoid exhaust fumes contaminating the samples.
- Begin purging the well at 0.2 to 0.5 liters per minute. Avoid surging. Purging rates for more transmissive formations could be started at 0.5-liter to 1 liter per minute. The initial field parameters of pH, specific conductance, dissolved oxygen, ORP, turbidity, and temperature of water are measured and recorded in the field logbook.
- Contain purged water for placement in labeled 55-gallon drum or tank, as appropriate.
- The water level should be monitored frequently during purging, and, ideally, the purge rate should equal the well recharge rate so that there is little or no drawdown in the well (i.e., less than 0.3-foot). The water level should stabilize for the specific purge rate. Record adjustments in the purge rate and changes in depth to water in the logbook. Purge rates should, if needed, be decreased to the minimum capabilities of the pump (0.1- to 0.2-liter per minute) to avoid affecting well drawdown. If the water level is drawn down by more than 1 foot, or 5% of the static water column, purging should be conducted in accordance with SOP *Groundwater Sampling from Monitoring Wells*.

- During purging, the field parameters are measured frequently (every 5 minutes) until the parameters have stabilized. Field parameters are considered stable when measurements meet the following criteria:
  - pH: within 0.1 pH units
  - Specific conductance: within 10 percent
  - Turbidity: <10 NTU or within 10 percent
  - Temperature: constant

### A5.3.2 Sample Collection

Once purging is complete the well is ready to sample. The pump should be allowed to operate at the same rate as the purge cycle until sampling begins, whereupon the discharge should be reduced to 0.1 L/m.

VOC samples are normally collected first and directly into pre-preserved sample containers (see Special Conditions for Sampling with Peristaltic Pumps).

The steps to be followed for sample collection are as follows:

- The cap is removed from the sample bottle, and the bottle is tilted slightly.
- The sample is slowly poured so that it runs down the inside of the sample bottle with a minimum of splashing. The pumping rate should be reduced to approximately 100 ml per minute when sampling VOCs.
- Inorganics, including metals, may be collected and preserved in the filtered form as well as the unfiltered form. Disposable in-line filters (0.45 micron filter), connected to the end of the sample tubing, are typically used for field filtration. Samples are field filtered as the water is being placed into the sample container. If a bailer is used, filtration may be driven by a peristaltic pump.
- Adequate space is left in the bottle to allow for expansion, except for VOC vials, which are filled to the top with a positive meniscus. VOC vials should be capped slowly to prevent introduction of air bubbles in the sample. Once capped, the VOC vial should be inverted and tapped to detect the presence of air bubbles.
- Immediately upon collection, all samples for chemical analysis are to be labeled and placed on ice.
- Re-usable equipment is cleaned and decontaminated.

The following information, at a minimum, will be recorded in the log book:

- Sample identification (site name, location, and project number; sample name/number and location; sample type and matrix; time and date; sampler's identity)
- Sample source and source description
- Field observations and measurements (appearance, volatile screening, field chemistry, sampling method), volume of water purged prior to sampling, number of well volumes purged, and field parameter measurements
- Sample disposition (preservative; laboratory name, date and time sent; laboratory sample number, chain-of-custody number, sample bottle lot number)
- Additional remarks

### Special Conditions for Sampling with Peristaltic Pumps

It is not acceptable to collect samples for organic compounds analyses through the flexible tubing used in the pump head. When collecting samples for organic compound analyses it is necessary to use a vacuum container, placed between the pump and the well for sample collection.

The following step-by-step procedures describe the process of sampling with a peristaltic pump and vacuum jug (see note following these procedures for collection of VOC samples):

- Disconnect the purge tubing from the pump. Make sure the tubing is securely attached to the protective casing or other secure object.
- Insert the tubing into one of the ferrule nut fittings of a Teflon® vacuum container transfer cap assembly.
- Place a suitable length of Teflon® tubing between the remaining transfer cap assembly ferrule nut fitting and the vacuum side of the flexible tubing in the peristaltic pump head. Securely hand-tighten both fittings.
- Turn the pump on. Water should begin to collect in the transfer container (typically a 1-liter “Boston round” glass sample container) within a few minutes. If water does not begin to flow into the container within several minutes, check the transfer cap fittings and make sure the assembly is tightly attached to the container. It may be necessary to tighten the ferrule nuts with a wrench or pliers to achieve a vacuum in the system, particularly when approaching the maximum head difference between the pump and water table (limit of suction).
- When the transfer container is nearly full, turn off the pump, remove the transfer cap assembly, and pour the sample into the appropriate containers.
- If additional sample volume is needed, replace the transfer cap assembly, turn the pump on, and collect additional volume.

NOTE: Samples for volatile organic compound analyses cannot be collected using the vacuum jug method. If samples for VOC analyses are required, they must be collected with a bailer or by other approved methods, such as the “soda straw” method. The “soda straw” method involves allowing the tubing to fill, by either lowering it into the water column (A) or by filling it via suction applied by the pump head (B). If method (A) is used, the tubing is removed from the well after filling and the captured sample is allowed to drain into the sample vial. If method (B) is used, after running the pump and filling the tubing with sample, the pump speed is reduced and the direction reversed to push the sample out of the tubing into the vials. Avoid completely emptying the tubing when filling the sample vials when using method (B) to prevent introducing water that was in contact with the flexible pump head tubing. Either method is repeated, as necessary, until all vials are filled.

Samples for some constituents, primarily inorganic analytes such as metals and cyanide, may be collected directly from the peristaltic pump head tubing. This method is acceptable under the following conditions:

- The pump head tubing must be changed between sampling locations;
- An equipment rinse blank must be collected by pumping de-ionized water through a piece of the tubing.

### A5.3.3 Additional Remarks

- If the well goes dry during purging, wait until it recovers sufficiently to remove the required volumes to sample all parameters. It may be necessary to return periodically to the well but a particular sample (e.g., large amber bottles for semivolatile analysis) should be filled at one time rather than over the course of two or more visits to the well.
- Disposable tubing is disposed of with PPE and other site trash.

## A5.4 Attachments

White paper on reasons and rationale for low-flow sampling.

## A5.5 Key Checks and Preventive Maintenance

- The drawdown in the well should be minimized as much as possible (preferably no more than 0.5-foot to 1 foot) so that natural groundwater-flow conditions are maintained as closely as possible.
- The highest purging rate should not exceed 1 liter per minute. This is to keep the drawdown minimized.
- Stirring up of sediment in the well should be avoided so that turbidity containing adsorbed chemicals is not suspended in the well and taken in by the pump.
- Overheating of the pump should be avoided to minimize the potential for losing VOCs through volatilization. Submersible pumps used in large diameter wells should be equipped with a shroud to force water flow across the pump motor to dissipate heat build-up.
- Keep the working space clean with plastic sheeting and good housekeeping.
- Maintain field equipment in accordance with the manufacturer's recommendations. This will include, but is not limited to:
  - Inspect sampling pump regularly and replace as warranted
  - Inspect quick-connects regularly and replace as warranted
  - Verify battery charge, calibration, and proper working order of field measurement equipment prior to initial mobilization and daily during field efforts

## **Attachment to the SOP on Low-Flow Sampling Groundwater Sampling from Monitoring Wells**

### **White Paper on Low-Flow Sampling**

*Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures (EPA, 1996).*

EPA recommends low-flow sampling as a means of collecting groundwater samples in a way that minimizes the disturbance to the natural groundwater flow system and minimizes the introduction of contamination into the samples from extraneous sources. The following are details about these issues.

When a pump removes groundwater from the well at the same rate that groundwater enters the well through the screen, the natural groundwater-flow system around the well experiences a minimum of disturbance. Some disturbance is bound to occur because you are causing groundwater to flow to the well in a radial fashion that otherwise would have flowed past it. However, the resulting low-flow sample provides the most-representative indication we can get of groundwater quality in the immediate vicinity of the well.

Normally, when a well is pumped at an excessive rate that drops the water level in the well below the water level in the aquifer, the water cascades down the inside of the well screen when it enters the well. The turbulence from this cascading causes gases such as oxygen and carbon dioxide to mix with the water in concentrations that are not representative of the native groundwater and are higher than expected. This causes geochemical changes in the nature of the water that can change the concentrations of some analytes, particularly metals, in the groundwater sample, not mention its effect on the dissolved oxygen levels that then will be measured in the flow-through cell. Such turbulence also may cause lower-than-expected concentrations of volatile organic compounds due to volatilization.

For wells in which the water level is above the top of the screen, the water up in the riser is out of the natural circulation of the groundwater and, therefore, can become stagnant. This stagnant water is no longer representative of natural groundwater quality because its pH, dissolved-oxygen content, and other geochemical characteristics change as it contacts the air in the riser. If we minimize the drawdown in the well when we pump, then we minimize the amount of this stagnant water that is brought down into the well screen and potentially into the pump. As a result, a more-representative sample is obtained.

Typically, wells contain some sediment in the bottom of the well, either as a residue from development that has settled out of the water column or that has sifted through the sand pack and screen since the well was installed. This sediment commonly has adsorbed on it such analytes as metals, SVOCs, and dioxins that normally would not be dissolved in the groundwater. If these sediments are picked up in the groundwater when the well is disturbed by excessive pumping, they can:

- Make filtering the samples for metals analysis more difficult
- Add unreasonably to the measured concentration of SVOCs and other organic compounds

The SOP for low-flow sampling has been modified recently and should be consulted for additional information about low-flow sampling and ways of dealing with wells in which the water level cannot be maintained at a constant level.



Purge Methodology:  Depth (ft): Diameter (in):	Date:				
	Diameter	Gal. Per Foot		Diameter	Gal.
	2"	0.163		5"	
	3"	0.367		6"	
	4"	0.653		8"	

Water level indicator, serial number: \_\_\_\_\_  
 Pump type (please circle): \_\_\_\_\_ Peristaltic \_\_\_\_\_ E  
 Pump serial number: \_\_\_\_\_

opening well      after venting, if initially high      middle of sampling      closing well

Field Parameters									
DTW (toc)	Flow Rate (ml/min)	Total Volume (gal)	pH (Std. Units)	Temp (°C)	Cond. (mS/cm)	ORP (mV)	D.O. (mg/L)	Turbidity (NTU)	Co
<0.33' or 4"	100-500	NA	±0.1 SU	±1°C	±5%	±20 mV	within 0.2 mg/L	±10% or < 10	

**SAMPLING INFORMATION:**

Before Sampling: \_\_\_\_\_ Depth sample was acquired: \_\_\_\_\_  
 Methodology: \_\_\_\_\_  
 Date/Time: \_\_\_\_\_

# Surface Water Sampling

## A6.1 Purpose and Applicability

This procedure conforms to the applicable EPA Quality Assurance Requirements and provides guidance for taking surface water samples from a stream.

## A6.2 Definitions

None.

## A6.3 Procedure

The following equipment is required for surface water sampling:

- Sample containers
- Preservatives
- pH/conductivity/temperature meter(s)
- Disposable cups for collection of samples for field measurements

To prevent cross-contamination of samples, sample collection will start at the most downstream location and continue upstream, and gloves will be changed between sampling locations. Samples are collected while standing downstream of each sample collection location.

Surface water sampling is performed as follows:

- Estimate the water depth (if a measuring device is used, wait to perform this task until after actual sampling to avoid disturbance of the stream bottom).
- Sketch the surface boundaries and sampling locations and photograph the sampling locations and conditions. Mark the location with flagging and survey stakes so it is easy found in the future.
- Record the physical characteristics of the water body such as odor, color, temperature, pH, conductivity, and dead vegetation.
- Put on clean protective gloves immediately prior to sampling.

Collect the sample by immersing the sample container into the water slightly below the surface, and hold at about a 45-degree angle. If the surface water is a flowing stream, river, or brook, hold the sample container at about a 45-degree angle with the mouth of the bottle facing upstream. The sample bottle should also be held in such a manner as to prevent the loss of preservative during the sample collection process. The sampler should take care not to disturb sediments in the immediate area of water collection. Samples collected for volatile organic analysis must contain no air bubbles in the vial after it has been capped; ensure this by turning the vial upside down and tapping it lightly. If air bubbles are present, more sample must be collected.



## Document Information

<b>Document Number:</b> ENV-SOP-MTJL-0100	<b>Revision:</b> 05
<b>Document Title:</b> Volatile Organic Compounds by GC/MS (EPA 8260B, 8260C, 624, 624.1 and SM 6200B)	
<b>Department(s):</b> VOA	

## Date Information

<b>Effective Date:</b> 21 May 2021
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## Notes

<b>Document Notes:</b>
------------------------

All Dates and Times are listed in: Central Time Zone

**Signature Manifest**

**Document Number:** ENV-SOP-MTJL-0100

**Revision:** 05

**Title:** Volatile Organic Compounds by GC/MS (EPA 8260B, 8260C, 624, 624.1 and SM 6200B)

All dates and times are in Central Time Zone.

**ENV-SOP-MTJL-0100**

**QM Approval**

Name/Signature	Title	Date	Meaning/Reason
Elizabeth Turner (007857)	Manager - Quality Program	20 May 2021, 01:02:51 PM	Approved

**Management Approval**

Name/Signature	Title	Date	Meaning/Reason
Kayla Coble (006639)	Supervisor	20 May 2021, 10:28:18 AM	Approved
James Burns (006456)	Manager - EHS	20 May 2021, 10:50:06 AM	Approved
Christopher Johnson (006487)	Manager - Operations	20 May 2021, 11:05:38 AM	Approved




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**STANDARD OPERATING PROCEDURE**

**TITLE:** Volatile Organic Compounds by GC/MS  
(EPA 8260B, 8260C, 8260D, 624, 624.1, and SM6200 B)

**ISSUER:** Pace National – Mt. Juliet, Tennessee

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## 1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of volatile organic compounds by EPA methods 8260B, 8260C, 8260D, 624.1, Standard Method 6200B, GRO, or similar volatile GC/MS analyses. This procedure is applicable to nearly all kinds of samples, regardless of water content, including ground water, aqueous sludge, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils and sediments. The compounds that can be determined using this SOP are listed in Appendix C, which contains a list of the typical primary and secondary ions used in determining these compounds.

### 1.1 Target Analyte List and Limits of Quantitation (LOQ).

The target analytes and the normal LOQ that can be achieved with this procedure are provided in Table 1, Appendix A.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

### 1.2 Specific instructions for employees of the Pace Analytical National Center for Testing & Innovation (Pace National) Sacramento, California laboratory are included in this document as "SAC Notes".

**STATE NOTE:** For samples analyzed in conjunction with the Ohio Voluntary Action Program (VAP) please utilize ENV-SOP-MTJL-0099.

**CLIENT NOTE:** For clients, whose environment laboratory quality program is administered by Environmental Standards Inc. (ESI), see controlled document QUA-30 VOA. \\FAP\NovDiskH\QAQC\Controlled Docs

## 2.0 SUMMARY OF METHOD

2.1 Volatile organic compounds (VOCs) are determined from a 5mL sample withdrawn from a sealed 40mL vial. For water samples analyzed for low levels of analytes using Method 5030 (ENV-SOP-MTJL-0131), the entire vial is placed into the instrument autosampler. The autosampler purges 5mL of sample and adds 1µL of surrogate standards and internal standards. An inert gas is bubbled through a sparger needle inserted into the sample. The purged volatile components then travel via a transfer line to a sorbent trap. When purging is complete, the trap is rapidly heated. The trap is backflushed with a helium carrier gas, to transport the desorbed sample components into a gas chromatographic (GC) column. The GC column separates and carries the components

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**STANDARD OPERATING PROCEDURE**

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to a mass spectrometer (MS) or a specific detector, depending on the determinative method selected.

**METHOD NOTE:** For Method 624.1, different sample sizes in the range of 5–25mL are allowed in order to meet differing sensitivity requirements. Calibration and QC samples must have the same volume as field samples.

- 2.2 For other samples, Method 5035 (ENV-SOP-MTJL-0129), volatile organic compounds are determined from a 5g sample combined with 5mL reagent water.

### 3.0 INTERFERENCES

- 3.1 Major sources of contamination are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components must be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation.
- 3.2 Analyses of reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted.
- 3.3 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. After analysis of a sample containing high concentrations, one or more instrument blanks may be analyzed to check for cross contamination. Alternatively, when analysis of a blank is not possible prior to the next sample, such as when an unattended autosampler is employed, the analyst should review the results for at least the next sample after the high-concentration sample. If analytes in the high-concentration sample are not present in the subsequent field sample, then the lack of carryover has been demonstrated.
- 3.4 This interference may be prevented by rinsing the purging apparatus and sample syringes with portions of organic-free reagent water between samples.
- 3.5 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105 °C.
- 3.6 In extreme situations, the whole purge and trap device may require dismantling and cleaning.
- 3.7 Screening of the samples prior to purge and trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished by using a portable PID or equivalent.
- 3.8 Choice of quantitative ions and qualifier ions: Some compounds may co-elute, so the selection of quantitation ions and qualifier ions must be made carefully so these ions are specific to each of the compounds that co-elute. Qualifier ions that are most commonly used are listed in Appendix C and are recommended from the published 8260 methods.
- 3.9 Special precautions must be taken to avoid contamination when analyzing for methylene chloride. The analytical and sample storage area must be isolated from all atmospheric




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sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing must be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst must be clean since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

- 3.10** Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling and handling protocol can serve as a check on such contamination. A storage blank must be analyzed every two weeks to check for cross contamination of samples while samples are stored in the volatiles laboratory walk-in cooler. The storage blanks are purchased from a vendor and must matrix match the contents of the cold room or refrigerator and is placed in the cooler for a period of two weeks. Every two weeks, the applicable storage blanks are analyzed to verify that no contamination of client samples has taken place due to contamination in the storage unit.
- 3.11** This procedure can be used to quantitate most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately 50 times higher due to poor purging efficiency. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides.
- 3.12** Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If a large amount of effervescent gas is generated, the sample may lose a significant amount of volatile analytes. If a sample effervesces, an unpreserved sample will be collected to eliminate volatiles loss whenever possible. The holding time for unpreserved VOC samples is seven days, rather than 14 days.
- 3.13** An analyst may re-analyze any sample if instrumentation or human error is suspected. This includes all QC samples, which can only be re-analyzed twice. If failure continues, instrument maintenance must be performed and/or the instrument must be re-calibrated.

## 4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

- 4.1** Lower Limit of Quantitation (LLOQ) – For analyses performed according to the requirements of Method 8000D, the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be greater than or equal to the lowest point in the calibration curve.

## 5.0 HEALTH AND SAFETY

- 5.1** The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.
- 5.2** The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data

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sheets (SDS) for all hazardous chemicals are available to all personnel. Employees must abide by the environmental, health, and safety (EHS) policies and procedures specified in this SOP and in the Pace National Chemical Hygiene / Safety Manual.

- 5.3** Personal protective equipment (PPE) such as safety glasses and/or side shields, gloves, a laboratory coat, and shoes that are not cloth, canvas, and/or perforated must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure. When handling glass, needles, knives, or any material with a potential sharp edge, employees must use cut-resistant gloves.
- 5.4** Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids, bases, or oxidizers in a fume hood whenever possible with the appropriate PPE designed for handling these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.
- 5.5** Spill kits are located in each laboratory department. Employees are to familiarize themselves with the location and contents of each spill kit in their area.
- 5.6** Universal precautions should be observed when performing any tests or procedures. Hard surfaces, instrument surfaces may be contaminated and should be handled according to good laboratory practices.
- 5.7** Contact your supervisor or local EHS coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure. Any accidents involving personnel or sample supplies are to be reported immediately to either the Manager and/or to the Safety Officer.

## **6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE**

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory performs samples collection for samples to be analyzed by this SOP in accordance with laboratory SOPs ENV-SOP-MTJL-0067, *Wastewater Sampling*, ENV-SOP-MTJL-0068, *Groundwater Sampling*, and ENV-SOP-MTJL-0313, *Field Sampling*. Refer to these SOPs for these instructions.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with laboratory SOP ENV-SOP-MTJL-0064, *Sample Shipping*. For this test method, immediately after sample collection, samples should be checked for pH and Chlorine and field treated. The bottle kits provided by the laboratory should include field test kits and treatment reagent.

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's quality manual.



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**General Requirements**

Matrix	Routine Container	Minimum Sample Amount <sup>1</sup>	Preservation	Holding Time
Water	40mL vials (in duplicate)	Two 40 mL vials headspace free, 3 vials preferred	pH<2 HCl; ≤6°C; Ascorbic acid or Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if Cl present	14 Days (Method 624: 7 days for aromatics if unpreserved)
Soil	4oz. w/zero headspace; 3 5g samples; 5g or 25g collected in an Encore or equivalent sampling device	5 g	pH<2 NaHSO <sub>4</sub> ; Methanol	14 Days

<sup>1</sup>Minimum amount needed for each discrete analysis.**Field / Matrix QC**

Trip Blank	Equipment Blank	MS/MSD	Field Duplicate
1/cooler	Per Sampling & Analysis Plan	1 pair at 5% Frequency	Per Sampling & Analysis Plan

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory SOP ENV-SOP-MTJL-0060, *Sample Receiving*. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at 6°C until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at 6°C until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 30 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

- 6.1** Volatile analysis for water and methanol preserved soil samples must be completed within 14 days from the time of sample collection. Water samples that are not chemically preserved must be analyzed within seven (7) days. It is also a Pace National requirement that water samples with 2-Chloroethyl vinyl ether (2-CEVE), as a compound of interest, be collected unpreserved and analyzed within 7 days of collection. It has been shown that the acid preservative reacts with the 2-CEVE, which could result in false negative reporting of 2-CEVE in samples. Unpreserved soil samples must be analyzed within 48 hours from the time of collection, added to preservative or otherwise frozen at ≤-7°C. High-level soil samples collected in Encore™ or equivalent type sampling devices must be placed in vials of methanol according to Method 5035 (ENV-SOP-MTJL-0129).

**STATE NOTE:** The State of South Carolina requires that all soil samples must be collected and analyzed using Method 5035. Samples must be preserved within 48 hours from the time of collection, if collected in Encore™ type sampling devices. The holding time for soil samples preserved with methanol or sodium bisulfate is 14 days from the time of collection. Non-Preserved South Carolina VOCs require 7-day holding time.




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- 6.2** Aqueous samples must be collected in at least duplicate in 40mL vials with minimal or no headspace to minimize the loss of highly volatile analytes. Sample kits can be configured to request additional vials per client request. Typically, the pH is adjusted to <2 with HCl, but the preservation requirements may vary by EPA program and may be specified in a regulation or project planning document that requires compliance monitoring for a given contaminant. An example of this is some samples from Missouri or Kansas may be preserved with tri-sodium phosphate and will have a resulting pH>12.
- 6.3** Soil samples may be collected by one of the following: 1) A 4oz. soil jar filled with soil with zero headspace, 2) Two 5g samples preserved in the field with 5mL NaHSO<sub>4</sub> to a pH<2 and one 5g sample preserved in the field with methanol (for high level analysis or data generated on Agilent™ 5977A or 5977B instruments [with an extractor ion or high efficiency source] may be reported to low-level MDL/RL values due to the enhanced sensitivity associated with these instruments) or 3) A 5g or 25g sample collected in an Encore or equivalent type sampling device and frozen in the laboratory within 48 hours from the time of collection.

For all soil samples, a 4oz. soil jar should also be collected to determine percent solids and for screening purposes. All samples and extracts must be shipped and stored at <6°C.

**STATE NOTE:** Soil and Water samples received from the states of Missouri or Kansas may be preserved with tri-sodium phosphate and will have a resulting pH>12.

**STATE NOTE:** For Alaska samples, when using a water miscible solvent (e.g., methanol) to extract soil volatile organic compounds (VOC), the adjustment of solvent volume for soil moisture content must be performed. Significant soil moisture can add to a pronounced dilution when performing methanol extractions. The potential under reporting of volatile concentrations is more pronounced as the percent moisture content increases. See section 10.2.3 for the calculation.

**6.4** Method 624.1 Considerations

- 6.4.1** If acrolein is to be determined, analyze the sample within three (3) days. To extend the holding time to 14 days, acidify a separate sample to pH 4–5 with HCl.
- 6.4.2** Experimental evidence indicates that some aromatic compounds, notably benzene, toluene, and ethyl benzene are susceptible to rapid biological degradation under certain environmental conditions. Refrigeration alone may not be adequate to preserve these compounds in wastewaters for more than seven days. To extend the holding time for aromatic compounds to 14 days, acidify the sample to approximately pH 2.
- 6.4.3** If halocarbons are to be determined, use an acidified sample.
- 6.4.4** Ethers are prone to hydrolysis at pH 2 when a heated purge is used. Aqueous samples should not be acid preserved if ethers are of interest or if the alcohols, they would form upon hydrolysis are of interest and the ethers are anticipated to present.
- 6.4.5** 2-Chloroethyl vinyl ether is subject to hydrolysis at low pH; therefore, determine 2-Chloroethyl vinyl ether from the un-acidified sample.

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## 7.0 EQUIPMENT AND SUPPLIES

The operation, cleaning, and scheduled maintenance procedures, as prescribed by the equipment manufacturer, are followed as provided in the Operator's Manuals. Documentation of maintenance or system modifications is recorded in a maintenance logbook which accompanies each analytical system.

### 7.1 Equipment

**7.1.1 Instrumentation:** All instrumentation meets or exceeds EPA method requirements. There are a total of 46 instruments; GCs include models 5890, 6890, 7890a, 7890b, 8890b, Intuvo or equivalent and MSs include 5973, 5975, 5977a, 5977b, HES (High efficiency source) or equivalent. Specific information for each instrument is included in the associated maintenance logbook.

Sample introduction system: Archon Autosampler, EST Centurion, OI 4760, Encon P & T, EST Evolution, OI 4660 or equivalent

**7.1.2 Analytical Balance,** capable of weighing to 0.01g, or equivalent

### 7.2 Supplies

**7.2.1 Glassware:** volumetric glassware equipped with penny head ground glass stopper. The volumetric flasks are cleaned by rinsing with methanol and laboratory reagent water. The volumetric flasks can be dried in a low temperature oven at less than 120°C and are never cleaned with a brush or strong alkali solution.

**7.2.2** The carrier gas used for volatiles analysis is Helium-5.0 grade.

**7.2.3** Syringes used for preparing the calibration curve and preparing samples and sample dilutions are Hamilton brand (or equivalent). Syringe sizes used are 0.50µL, 10µL, 25µL, 50µL, 100µL, 250µL, 500µL, 1mL, and 5mL.

**7.2.4** Glass Sample (VOA) and Standard Vials

**7.2.4.1** 40mL VOA vials with a Teflon™/silicone septa and polypropylene open-top cap.

**7.2.4.2** 8mL vials with Teflon™/silicone/Teflon™ septa and polypropylene open-top cap. (Used to store unused standards)

**7.2.4.3** 2mL amber vial with Teflon™/silicone/Teflon™ septa (used to store unused standards)

**7.2.5** Miscellaneous

**7.2.5.1** Stainless Steel Spatula or wooden tongue depressor

**7.2.5.2** Teflon™-coated stir bars, 8mm x 16mm

**7.2.5.3** Glass beads –VWR™ #EM1.04015.0500, or equivalent

**7.2.6** Oven: Fisher IsoTemp Forced-Air Oven with capabilities of 100°C, or equivalent

## 8.0 REAGENTS AND STANDARDS

All reagents and standards must be recorded in the appropriate preparation log and assigned a unique number. See ENV-SOP-MTJL-0041, *Standards Logger – Tree Operation*. Additional information regarding reagent preparation can be found in the Standards Logger (Tree) digital

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archive system. All spiking solutions and surrogate standard solutions should be replaced at least every six months or sooner if a problem is detected unless otherwise noted. Working standards for most compounds should be replaced after four weeks unless the integrity of the standard is suspected to of being compromised prior to that time. Working standards for gases should be replaced after one week unless the acceptability of the standard can be documented.

## 8.1 Reagents

**8.1.1** Nanopure water or equivalent: Nanopure water is used in all blanks to ensure it contains less than the method detection limit (MDL) of all compounds of interest. If volatile compounds are detected in the blank above MDL all samples associated with this blank must be flagged (see Section 11.3.1.1).

**NOTE:** For all DoD samples the laboratory water is used in all blanks to assure that it contains less than 1/2 LOQ of all compounds of interest. The blank must be assessed to ensure that the water does not show any detection of any VOC compounds. If volatile compounds are detected in the blank above 1/2 LOQ, then those samples must be flagged.

**8.1.2** Methanol, CH<sub>3</sub>OH – purge-and-trap grade, demonstrated to be free of analytes. Store apart from other solvents.

**8.1.3** Sodium Bisulfate, Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> - QEC Level 3 Certified, or equivalent

## 8.2 Standards

**8.2.1** STOCK SOLUTIONS – Primary and Secondary Sources

- Stock calibration solutions must be purchased as certified solutions.
- Certificates must be kept on file.
- All Stock standards must be stored ≤ 6 °C or as recommended by the standard manufacturer.
- All non-gas stock standards must be replaced after six months, or sooner, if check standards indicate a problem.
- Both gas and liquid standards must be monitored closely by comparison to the initial calibration curve and by comparison to a second source ICV, or Secondary Source Verification Standard (SSCV).
- Gas intermediate/secondary standards must be replaced weekly, or sooner, if comparison to check standards indicates a problem.
- Non-gas intermediate/secondary standards must be replaced after six months, or sooner, if comparison to check standards indicates a problem.

**8.2.1.1** Primary Source - Primary source standards are used to prepare the initial 5-point calibration curve (additional levels may be used as needed), the continuing calibration verification (CCV) standard, LCS and matrix spikes. The CCV is analyzed to verify the initial calibration and is prepared using the primary source standard used to produce the calibration curve. See Section 9.2.3.2.4 through 9.2.3.2.8 for the instrument preparation of the calibration standards. When primary standards are consumed, new standards must meet the same QC criteria as the consumed standards. Stock standards must be stored below -10 °C and have a six-month holding time once opened. The expiration date of the diluted standards must not exceed the expiration




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date of the stock standards from which they are prepared. Once diluted, the standard must be replaced weekly. The standard list of target LCS compounds are those compounds listed in Appendix I. The LCS must be prepared in the appropriate matrix (organic-free reagent water or purified solid) depending upon the matrix within the analytical batch; and contain all of the method target analytes. A subset of the method target analytes could be used based on the project specific requirements.

**STATE NOTE:** South Carolina DHEC and the USACE require that all target analytes are present and evaluated in the LCS

**Calibration Mix**

The calibration mix is prepared in methanol in a 25mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
Phenomenex	Revised 8260 Calibration Mix	AL0-130502	1	100
Phenomenex	Custom Ketones Mix	AL0-130115	1	500
Phenomenex	Acrolein	AL0-130159	1	500
Phenomenex	Custom 8260 Additions Mix	AL0-180004	1.25	100
Phenomenex	Vinyl Acetate	AL0-130117	1	500
Phenomenex	2-Chloroethyl vinyl ether	AL0-130116	1	500
Phenomenex	Ethanol Standard	AL0-130551	0.5	5000
AccuStandard	Vinyl Bromide	S-2688A	1.25	100
Agilent	Custom Standard (1,3,5-TCB)	CUS-0002027	1.25	100

**8.2.1.1.1** The calibration standard mix is prepared by making a 1:1 ratio of the gas standard (Agilent, ULTRAgold™ Custom Standard CUS-27747 or equivalent at 100µg/mL) and the calibration mix standard.

**8.2.1.1.1.1** For soil autosamplers (5mL), a dilution of 10x is required for the ICV mix. The solution is stored in 3mL aliquots in zero headspace vials. The storage temperature is below ≤6° C.

**AP9/Oxygenates Calibration Mix**

The AP9/Oxygenate ICV solution is prepared in methanol in a 25mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
NSI	Ethyl Acetate	681	0.125	50
NSI	Custom AP9 Standard	Q-6603-O	3.125	50
Absolute	Isopropanol	70941	0.625	25
NSI	Tert-Butyl formate	321	0.625	50

**8.2.1.2** Secondary Source - Secondary source standards must be used to prepare the secondary source verification standard (SSCV) or initial calibration verification (ICV). These standards are purchased from a different vendor or the primary vendor can supply different lot numbers, if a separate vendor is not available. The standard is at a concentration near the mid-level calibration standard. Stock standards must be stored at or below ≤6° C and have a six-month holding time once opened, except the ICV

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gases which have 1 week holding time. Once diluted, the standard must be replaced weekly.

**SSCV**

Prepare the SSCV mix in methanol in a 10mL volumetric flask as follows. A separate source or separate lot number is used for standard verification. The standard list of target LCS compounds are those compounds listed in Appendix I.

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
NSI	8260 Custom Mix 2	Q-6354-O	0.4	500
Restek	Acrolein	30645	1	500
SPEX CertiPrep	AZ fuel additive 4 comps.	VO-ESCTN-9	0.8	100
Agilent	Custom Standard	CUS-30388	0.4	Varies
Agilent	Custom Standard (ethanol)	CUS-30649	0.2	5000
Restek	1,3,5-Trichlorobenzene	31081	1	100
SPEX CertiPrep	Vinyl Bromide	S-4049	1	100

**8.2.1.2.1** The working second source (SSCV) or Initial Calibration Verification (ICV) is prepared by making a 1:1 ratio of the gas standard (Phenova, VOA Gas Calibration Mix AL0-130108 or equivalent at 200µg/mL) and the second source standard.

**8.2.1.2.2** For soil autosamplers (5mL), a dilution of 10x is required. The solution is stored in 3mL aliquots in zero headspace vials. The storage temperature is at or below -10° C.

**AP9/Oxygenates SSCV**

The AP9/Oxygenates SSCV is prepared in methanol in a 10mL volumetric flask by adding:

Manufacturer	Product	Cat. #	Amount added (mL)	Final Conc. (ppm)
NSI	Custom AP9 Standard	Q-6603-O	1.25	50
NSI	Ethyl Acetate	681	.05	50
Absolute	Isopropanol	70941	0.25	25
NSI	Tert-Butyl formate	321	0.25	50

**8.3** Surrogate standard stock solutions must be purchased as certified solutions. Certificates must be kept on file. Stock standards must be stored at or below ≤6° C and have a six-month holding time, once opened. Surrogate spiking solutions are purchased from Phenova, Part# AL0-130491, or equivalent, at 20,000ug/mL, which contains both internal standards and surrogate compounds. This solution is then diluted by 100X to obtain a 200ug/mL working solution.

**8.3.1** The following are Pace National designated volatiles' analysis surrogates:

- Toluene-d8
- 4-Bromofluorobenzene




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- 1,2-Dichloroethane-d4
- 8.4** Internal standard stock solutions must be purchased as certified solutions. Certificates must be kept on file. Stock standards must be stored at or below  $\leq 6^{\circ}\text{C}$  and have a six-month holding time, once opened. [Internal standard and surrogate standard – Phenova, Part# AL0-130491, or equivalent]
- 8.4.1** The following are Pace National designated volatiles' analysis internal standards:
- 1,4-Dichlorobenzene-d4
  - Fluorobenzene
  - Chlorobenzene-d5
- SAC NOTE:** Internal Standard/Surrogate Mix – o2si 8260 Internal Surrogate Solution Catalog No.: 121722-08-20PAK. This mix is diluted 250x to produce a 50  $\mu\text{g/mL}$  internal standard and surrogate mix. During analysis, 5 $\mu\text{L}$  of this solution is added to 5mL of sample, resulting in a concentration of 50  $\mu\text{g/L}$ . The internal standards and surrogates are the same as listed in 8.3.1 and 8.4.1 with the addition of Tert-butyl Alcohol-d10 as an internal standard.
- 8.5** 4-Bromofluorobenzene (BFB) standard - The BFB in the custom internal standard mix is used to verify mass spectrometer tuning. Since internal standards and surrogates are added to all samples and standards, BFB is included as part of our initial calibration and calibration verification standards. Certificates of analysis must be kept on file. Stock standards must be stored at or below  $\leq 6^{\circ}\text{C}$  and have a six-month holding time, once opened.
- 8.6** Matrix spike (MS) standard - Stock standards must be stored at or below  $\leq 6^{\circ}\text{C}$  and have a six-month holding time, once opened. Once diluted, the standard must be replaced weekly.
- 8.6.1** The matrix spike standard is prepared from the stock standard in Section 8.2.1.
- 8.6.2** The spike should be at a mid-level of the calibration range. Some contracts may require a site-specific concentration.
- 8.6.3** Standard spiking practice requires the use of ALL TARGET ANALYTES as specified in Appendix I and must be evaluated against the current control limits presented in the LIMS.
- All compounds in the spike solution must be evaluated for acceptable recovery. In the absence of established control limits, default recovery limits are 70 - 130%.

## 9.0 PROCEDURE

**Analysis Summary:** Volatile compounds are introduced into the gas chromatograph by purge and trap, via the Archon autosampler, as described on Section 2. Soils require method 5035 for sample preparation, See ENV-SOP-MTJL-0129.

### 9.1 Equipment Preparation

Chromatographic conditions: All changes in analytical conditions are listed in the Maintenance Log.




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### 9.1.1 Support Equipment

All support equipment used must be calibrated or verified prior to use according to SOP ENV-SOP-MTJL-0039, *General Analytical Balance Operation and Verification in the Laboratory* current revision or replacement.

Screening the sample prior to purge-and-trap analysis provides guidance on whether sample dilution is necessary and prevents contamination of the purge-and-trap system by screening of 5mL of sample using an HNU or equivalent portable PID. See ENV-SOP-MTJL-0102, Volatile Organic Compounds Screening using the RAE Systems Photoionization Gas Detector Model MiniRAE 3000.

### 9.1.2 Instrument

#### 9.1.2.1 Routine Instrument Operating Conditions

9.1.2.1.1 Typical conditions for each instrument and column are listed below:

Inlet	off
Detector	200°C
Oven Equib. Time:	0.50 minutes
Oven Max	240°C
Init Temp	45°C hold 1.0 minute
Ramp	20°C/min to 240 hold 1.0 minute

9.1.2.1.2 Typical conditions for each autosampler are listed below:

Heating sample	1 minute at 40oC
Purge	11 minutes at 40oC
Desorb	1 minutes at 250oC
Bake	2 minutes at 260oC

9.1.2.1.3 Typical conditions for each MS detector are listed below:

Electron energy – 70 volts (nominal)  
Mass range – 35 to 300 amu  
Scan time – 1.2 sec/scan  
Manifold vacuum – 3 x 10<sup>-6</sup> torr

9.1.2.1.4 TUNING - Each GC/MS system must be hardware-tuned (1µL < 50ng) with BFB to meet the criteria listed below. The mass-spectrometer must meet acceptable BFB sensitivity criteria before analysis can begin. The instrument must be tuned every 12 hours for 624.1, 8260B, 8260C, and 6200B. BFB tuning for method 8260D is prior to each calibration.



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<b>BFB Key Ions and Ion Abundance Criteria for 8260B, 8260C, 8260D, 624.1</b>	
<b>Mass</b>	<b>Ion Abundance Criteria</b>
95	50 - 200% of mass 174
96	5 - 9% of mass 95
173	< 2% of mass 174
174	50 - 200% of mass 95
175	5 - 9% of mass 174
176	95 - 105% of mass 174
177	5 - 10% of mass 176

<b>BFB Key Ions and Ion Abundance Criteria for 6200</b>	
<b>Mass</b>	<b>Ion Abundance Criteria</b>
50	15.0-40.0% of mass 95
75	30.0-60.0% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	< 2.0% of mass 174
174	> 50.0% of mass 95
175	5.0-9.0% of mass 174
176	> 95.0%, but less than 101% of mass 174
177	5.0-9.0% of mass 176

**EPA 624.1 NOTE:** The 12-hour tune clock begins after analysis of the BFB, the LCS, and the BLANK and ends 12 hours later. BFB, the LCS, and BLANK are outside of the 12-hour tune clock. The MS and MSD are treated as samples are analyzed within the 12-hour clock.

## 9.2 Initial calibration

### 9.2.1 Calibration Design

Calibration Levels for single analytes

Soil Samples - Soil samples are analyzed with a heated purge in the soil chamber of the Archon, or equivalent autosampler. The calibration curve is generated by injecting the following volumes of Calibration Mix (See Section 8.2.1.1) into 5mL of reagent water. Surrogate standard is prepared by diluting the surrogate by 1:10 using (NSI lab solutions, 8260 Surrogate Mix Q-4392 or equivalent at 1000µg/mL).

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8260 Calibration Curve - GC/MS Soil (into 5mL water)			
Intermediate solution volume (µL)	Concentration of standard (ppb)	Surrogate Added (µL)	Concentration of Surrogate (ppb)
0.025	0.25	0	n/a
0.05	0.5 (LOD Point)	0	n/a
0.1	1	1	1
0.2	2	2	2
0.5	5	3	3
2.5	25	4	4
7.5	75	5	5
10	100	6	6
20	200	7	7

8260 Calibration Curve - GC/MS Soil (into 50mL water) for use with Agilent 5977A or 5977B Only			
Intermediate solution volume (µL)	Concentration of standard (ppb)	Surrogate Added (µL)	Concentration of Surrogate (ppb)
0.02	0.02 (LOD Point)	0	n/a
0.04	0.04	0	n/a
0.1	0.1	0	n/a
0.2	0.2	0	n/a
0.5	0.5	0	n/a
1	1	1	1
2	2	2	2
5	5	3	3
25	25	4	4
75	75	5	5
100	100	6	6

AP9/Oxygenates Calibration Curve - GC/MS Soil (into 5mL of water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
0.5	0.5
1	1.0
5	5
10	10
15	15
20	20

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AP9/Oxygenates Calibration Curve - GC/MS Soil (into 50mL of water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
5	0.5
10	1.0
50	5
100	10
150	15
200	20

**Note 1:** When analyzing soil samples by the low-concentration method (Section 9.4), the calibration standards must be heated to 40°C ± 1°C prior to purging.

**Note 2:** Injections should be performed from the lowest to the highest standards with a cleanup injected after the highest standard and followed by the secondary source standard to verify the initial calibration curve.

Water Samples - Water samples are run with a heated purge using the Archon, or equivalent autosampler. The calibration curve is generated by injecting the following volumes of Calibration Mix (See Section 8.2.1.1) into 50mL of water. Surrogate standard is added to the curve points, (Phenova, Custom IS-SURR Mix AL0-130574 or equivalent at 1000µg/mL).

8260 Calibration Curve - GC/MS Water (into 50mL water)			
Intermediate solution volume (µL)	Concentration of standard (ppb)	Surrogate Added (µL)	Concentration of Surrogate (ppb)
0.02	0.02	0	n/a
0.04	0.04	0	n/a
0.1	0.1	0	n/a
0.2	0.2	0	n/a
0.5	0.5 (LOD Point)	0	n/a
1	1	1	1
2	2	2	2
5	5	3	3
25	25	4	4
75	75	5	5
100	100	6	6
200	200	7	7

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AP9/Oxygenates Calibration Curve - GC/MS Water (into 50mL of water)	
Intermediate solution volume (µL)	Concentration of standard (ppb)
5	0.5
10	1.0
50	5
10	10
150	15
200	20

**SAC NOTE:** See Controlled Document DAV-03 VOA ICAL Reference for calibration table.

### 9.2.2 Calibration Sequence

#### Curve – General Criteria

- A minimum of 5-point calibration is performed using the primary standards listed in Section 8.2.1.1. Additional levels may be included to better meet project or client requirements. Regardless of the specific number, the calibration levels analyzed should correspond to a range of concentrations expected to be found in samples, without exceeding the working range of the GC/MS system.
- A calibration point must be analyzed at or below the reporting limit. The concentration of the lowest calibration standard analyzed should be at least 3-5 times the MDL. The instrument response must be distinguishable from the instrument background noise. The signal to noise ratio is the magnitude of the signal strength detected by the mass spectrometer relative to the magnitude of the background noise of the instrument. Instrument conditions must be optimized before the analysis of a calibration curve to minimize background effects.

**STATE NOTE:** The reporting level standard must be refit after calibration is complete. This standard is required by the state of North Carolina and is used to verify the low end of the calibration curve.

**STATE NOTE:** When analyzing samples from Minnesota, the reporting limit must be verified with each calibration or at least monthly. Verification can be performed by re-quantitation of the low calibration standards using the newly updated calibration curve or by analyzing a separate reporting level standard following calibration curve update. This standard must recover  $\pm 40\%$  of the expected concentration. If the criterion is not met, a higher-level standard may be re-quantitated or analyzed; however, the reporting limit must be amended to reflect the increased concentration of the standard utilized. Analytes known to be poor performers are dealt with on a case-by-case basis.




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- The highest standard must not exceed the linear range of the detector. The concentration of the highest standard must produce a response, which does not cause the MS detector to become saturated. The highest concentration used in the calibration curve must allow the analyte to meet the calibration requirements outlined in Sections 9.2.3.
  - When using Method 5035, ENV-SOP-MTJL-0129, the calibration curve must be prepared in the same solutions used to preserve the field samples.
  - **EPA 8260C NOTE:** The method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve, while the relative percent difference and quadratic methods of calibration do not have this potential bias. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve. It is not necessary to reanalyze a low concentration standard; rather the data system can recalculate the concentrations as if it were an unknown sample. The recalculated concentration of the low calibration point should be within  $\pm 30\%$  of the standard's true concentration.
  - **EPA 8260D NOTE:** The method of linear regression analysis has the potential for a significant bias to the lower portion of a calibration curve, while the relative percent difference and quadratic methods of calibration do not have this potential bias. A minimum quantitation check on the viability of all calibration points should be performed by re-fitting the response from each calibration standard back into the curve. The recalculated concentration of the reporting level point should be within  $\pm 50\%$  of the standard's true concentration. All other recalculated concentration calibration points should be within  $\pm 30\%$  of the standard's true concentration. If a failure occurs in the low point and it is equivalent to the LLOQ, the analyte should be reported as estimated near that concentration or the LLOQ should be reestablished at a higher concentration.
- STATE NOTE:** South Carolina does not allow the use of quadratic regression for compounds that have previously demonstrated linearity.
- The method reference spectra must be updated from the mid-point of each calibration.

### 9.2.3 ICAL Evaluation

#### 9.2.3.1 Curve Fit

##### 9.2.3.1.1 Linear Regression - Criteria

When any compound does not meet the calibration criteria for RF, the most appropriate curve fitting model is used. If linear regression is used, it must be noted on the data (preferably on the CCV RF report), next to the affected compound. It must also meet correlation coefficient (r) criteria of 0.995 or better. SM




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6200 requires a linear regression correlation coefficient of >0.994.

Linear regression is achieved by plotting the instrument response versus the concentration of the standards. The resulting regression line must not be forced through the origin and the origin must not be included as a calibration point.

**STATE NOTE:** For all Wisconsin sample analyses, analysts must evaluate the %RSD of calibrations to ensure that they do not have unacceptable curvature. The %RSD limit criteria, as found in the specific methods listed above, applies to calibrations using average RF calibrations. For linear and quadratic curve fits, a limit of 40% RSD is used for normal target analytes and 50% RSD is utilized for known poor performing compounds.

The most appropriate curve fitting model from among the following choices must be utilized (given in the order of preference): Average Response Factor

- Linear – No Weighting
- Linear – 1/x Weighting
- Linear – 1/x<sup>2</sup> Weighting
- Quadratic

**STATE NOTE:** South Carolina does not allow the use of quadratic regression for compounds that have previously demonstrated linearity.

#### 9.2.3.1.2 Calibration Corrective Action

When the RSD exceeds 15% or linear regression criteria could not be met, plot and inspect the calibration data for abnormal chromatographic responses. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

If calibration criteria are not met, then one of the following options must be applied to the GC/MS initial calibration:

#### 9.2.3.2 Relative Standard Error (RSE)

A measure of relative error must be documented for each calibration. For analytes using an Average curve fit, the %RSD is the relative error, and no further evaluation is required. For analytes using a linear or quadratic curve fit, the relative error must be evaluated for the calibration low and mid-points using one of the procedures below.

##### 9.2.3.2.1 %Relative Error (%RE)

If using this method of determining relative error, the %RE of the calibration mid-point must be between 70-130%. For the calibration low point, %RE must be between 50-150%. The %RE




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is evaluated using the response/resultant concentration in the applicable initial calibration standards.

#### 9.2.3.2.2 %Relative Standard Error (%RSE)

If using this method of determining relative error, the acceptance limits for %RSE are numerically identical to the %RSD criteria. The %RSE is evaluated by creating a copy of the applicable calibration standard files and then quantitating the copied files against the new calibration.

**Note:** Calibration standard files must be copied and re-named for use in this evaluation. DO NOT over-write the initial calibration files.

Adjust the instrument and/or perform instrument maintenance and re-analyze the calibration standards until the RSD of the calibration meets criteria.

**9.2.3.2.3** Narrow the calibration range until the response is linear. If the low standard is below the estimated quantitation limit (i.e., for the poor purgers in a commercially available prepared standard mix), then this standard may be dropped. Re-calculate the RSD without the low standard to determine if the RSD meets the QC limit. If the lowest standard is dropped, the reporting limit could require a change. Check with the supervisor to determine if a point can be removed and not affect reporting limits requirements.

Compounds that are very soluble in water generally are poor purgers. The ketones, vinyl acetate, acrolein, and acrylonitrile fall into this category.

#### 9.2.3.2.4 EPA 8260B: Response Factors (RF's) & Calibration Check Compounds (CCC's) - Soil/Water

Using the RFs for the initial calibration curve from Section 8.2.2, calculate and record the percent relative standard deviation (%RSD) for all compounds. Calculate the percent RSD as in Section 10.2.5. Linearity can be assumed if the RSD criteria is met, thus allowing quantitation calculations to be performed using RF.

CCC Criteria - The %RSD for each individual CCC must be less than 30%. The CCCs are:

1,1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1,2-Dichloropropane	Vinyl chloride

Target Analytes and other Non-CCC's - The RSD must meet the following criteria




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<15% RSD for all 8260B Target Analytes
<20% RSD for all 8260C/D Target Analytes
<35% RSD for all 624.1 Target Analytes
<20% RSD for all KSGRO Samples
<15% RSD for n-Hexane
<20% RSD for 6200 Analytes
<15% appendix 9 Analytes
<10% RSD for all 601/602 Target Analytes

Compounds not meeting the RSD requirement may be considered for linear regression as stated in 8.2.7.3

#### 9.2.3.2.5 EPA 8260C: Response Factors (RF's)

**9.2.3.2.5.1 Calibration Curve Criteria** - Calculate the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte using the following equations. The RSD should be less than or equal to 20% for each target analyte. It is also recommended that a minimum response factor for the most common target analytes as noted in Appendix G, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet these criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project and therefore they may be used as qualified data or estimated values for screening purposes.

**9.2.3.2.6** When the RSD exceeds 20% or linear regression criteria could not be met, plot and inspect the calibration data for abnormal chromatographic responses. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

**NOTE:** To maximize batches calibration RSDs are held to the strictest method reported, the RSD used for all methods is 15%.

#### 9.2.3.2.7 EPA 8260D: Response Factors (RF's)

**9.2.3.2.7.1 Calibration Curve Criteria** – Calculate the mean RF and the relative standard deviation (RSD) of the RFs for each target analyte. The RSD should be <20% for each target analyte. Appendix H contains minimum RFs that may be used as guidance in determining whether the system is behaving properly and as a check to see if






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calibration standards are prepared correctly. It is neither expected nor required that all analytes meet these minimum RFs in the guidance table, however target analytes must not have an RF <0.01. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet these criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project and therefore they may be used as qualified data or estimated values for screening purposes.

**9.2.3.2.7.2** When the RSD exceeds 20% or linear regression criteria could not be met, plot and inspect the calibration data for abnormal chromatographic responses as the inspection can be a useful diagnostic tool.

**NOTE:** To maximize batches calibration RSDs are held to the strictest method reported, the RSD used for all methods is 15%

**9.2.3.2.8 EPA 624.1**

Calculate the mean (average) and relative standard deviation (RSD) of the response factors. If the RSD is less than 35%, the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to fit a linear or quadratic regression of response ratios vs. concentration ratios. If used the regression must be weighted inversely proportional to concentration. The coefficient of determination (R<sup>2</sup>) of the weighted regression must be greater than 0.920 (this value roughly corresponds to the RSD limit of 35%). Alternatively, the relative standard error may be used as an acceptance criterion. As with the RSD, the RSE must be less than 35%. If an RSE less than 35% cannot be achieved for a quadratic regression, system performance is unacceptable, and the system must be adjusted and re-calibrated.

**NOTE:** Using capillary columns and current instrumentation, it is quite likely that a laboratory can calibrate the target analytes in this method and achieve a linearity metric (either RSD or RSE) well below 35%. Therefore, laboratories are permitted to use more stringent acceptance criteria for calibration than described here (e.g., to harmonize their application of this method with those from other sources).

**STATE NOTE:** South Carolina does not allow the use of quadratic regression for compounds that have previously demonstrated linearity.

**9.2.3.3 Initial Calibration Verification**

Calibration Verification for EPA Methods 8260B, 624.1 and SM 6200B:




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**9.2.3.3.1 SSCV's**

After a successful calibration, a Second Source Calibration Verification (SSCV) or Initial Calibration Verification (ICV) must be analyzed to verify the calibration. This standard must be made from a second source, preferable from a different vendor than the calibration standards. The second source calibration standard must perform within following criteria:

CCC and SPCC compounds	± 30%
Other compounds (non-poor performers)	± 40%
Poor Performers (8.3.2)	in-house LCS limits

**9.2.4 Continuing Calibration Verification****9.2.4.1 Internal Standards and Surrogates – Soil/Water**

The autosampler adds 1µL of the IS/surrogate mix to each sample. The addition of 1µL of the surrogate spiking/internal standard solution is equivalent of 16µg/L of each surrogate/internal standard. Internal standard and surrogate standard are contained within the same spiking mix. Internal Standards are listed Section 8.4.1 and Surrogates are listed in Section 8.3.1.

**SAC NOTE:** During analysis, 5µL of this solution is added to 5mL of sample, resulting in a concentration of 50 µg/L

**Tabulation of the Internal Standards**

Tabulate the area response of the characteristic ions (see Appendix C) against each compound's concentration and each internal standard concentration. Then calculate the response factor (RF) for the quantifying ion of each compound relative to the appropriate internal standard according to the calculation provided in Section 9.1. The internal standards used should permit most of the compounds of interest in a chromatogram to have retention times of 0.80 to 1.20, relative to one of the internal standards. The average RF must be calculated and recorded for each compound.

**9.2.4.2 System Performance Check Compounds (SPCCs) – Soil/Water**

A system performance check must be made before the calibration curve can be used. The minimum relative response factor for volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichlorethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

These compounds are typically used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

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Compound	Effect on stability
Chloromethane	This compound is the most likely compound to be lost if the purge flow is too fast.
Bromoform	This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.
1,1,2,2-Tetrachlorethane and 1,1-Dichloroethane	Contaminated transfer lines degrade these compounds in purge-and-trap systems. Active sites in trapping materials also can cause problems.

Adjust the purge gas (Helium or Argon) flow rate to 25-40mL/min on the purge-and-trap device. Optimize the flow rate to provide the best response for chloromethane and bromoform. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces bromoform response.

**9.2.4.3 CCC's**

The curve must be verified daily by a calibration standard known as the Continuing Calibration Verification standard (CCV) and is analyzed every 12 hours. This standard is prepared at or near the mid-point of the calibration curve. A maximum of 20% criteria would be expected for CCC analytes (Listed in Section 8.2.7.1) and n-Hexane when requested as a target analyte.

Compounds on average response factor use % difference,

$$\% \text{ Difference} = (RF_v - R_{f_{ave}}) / R_{f_{ave}} \times 100\%$$

Compounds on regression fit model use percent drift,

$$\% \text{ Drift} = (\text{Calculated conc} - \text{Theoretic conc}) / \text{Theoretic conc} \times 100\%$$

Criteria for both is  $\leq 20\%$ .

**9.2.4.4 SPCC's**

The SPCC's must have a minimum response factor as stated in Section 8.2.6. If these criteria are exceeded, then corrective action is required.

**9.2.4.5 All Target Analytes and Non-CCC's**

When analyzing 8260B and 624.1 concurrently, calibration verifications are evaluated using both the 8260B criteria (sections 8.3.1.2 through 8.3.1.3 and the 624.1 criteria (Appendix F). For analytes not on the 624.1 list, all target analytes (except for the poor performers (9.2.4.7) must meet a maximum of 40% drift from the calibration curve. The analyst evaluates all analytes carefully and the experience of the analyst weighs heavily when determining the usability of the data.




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Poor performers are allowed a maximum of 50% drift from the calibration curve. See section 9.2.4.7 for a listing of poor performing analytes.

**STATE NOTE:** For South Carolina 624.1 samples, the target analytes reported must agree with the compound list found in the EPA 624.1 published method.

Calibration Verification for EPA Method 8260C/D:

#### 9.2.4.6 SSCV's

After a successful calibration, a Second Source Calibration Verification (SSCV) or Initial Calibration Verification (ICV) must be analyzed to verify the calibration. This standard must be made from a second source, preferable from a different vendor than the calibration standards. The second source calibration standard must perform within following criteria:

All compounds	± 30%
Poor Performers (9.2.4.7)	in-house LCS limits

#### 9.2.4.7 Target Analytes

The curve must be verified initially by a calibration standard known as the Continuing Calibration Verification standard (CCV) and is analyzed every 12 hours). This standard is prepared at or near the mid-point of the calibration curve. A maximum of 20% criteria would be expected for all target analytes and n-Hexane when requested as a target analyte.

If the percent difference (average RSD) or percent drift (linear regression models) for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large numbers of compounds that may be analyzed by this method, some compounds will fail to meet the criteria. If the criterion is not met (i.e., greater than 20% difference or drift) for more than 20% of the compounds included in the initial calibration, then corrective action must be taken prior to the analysis of samples. In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when the failed compound is present, a reporting level verification is analyzed. Reference QUA-12 *CCV Criteria for 8260C and 8270D* for assessing reporting level verification.

All compounds must have a minimum response factor. If these criteria are exceeded, then corrective action is required. The minimum response factors are generated in house based off historical data.

Poor Performers:

The poor performers are as follows:

Propene	2-Chloroethylvinyl Ether
Dichlorodifluoromethane	Acrolein
Carbon Disulfide	Vinyl acetate
Bromomethane	trans-1, 4-dichloro-2-butene




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Chloroethane	Alcohols (Ethanol, TBA, TAA, ETBA, Butanol)
1,3-Butadiene	Iodomethane
1,2-Dibromo-3-chloropropane	Naphthalene
1- Methyl-naphthalene	2-Butanone
2- Methyl-naphthalene	2-Hexanone
Acetone	4-Methyl-2-pentanone
Pentachloroethane	Cyclohexanone
Tert-butyl Formate	Methyl cyclohexane

**9.2.4.8 Laboratory Control Standard (LCS):** A laboratory control sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Also note the LCS for water sample matrices is typically prepared in organic-free reagent water similar to the continuing calibration verification standard. In these situations, a single analysis can be used for both the LCS and continuing calibration verification.

- QC Limits are found in the LIMS for LCS and MS/MSD.
- If the stated criteria are exceeded, then corrective action is required.
- See Section 12.9.1, on marginal exceedances.

**STATE NOTE:** All South Carolina DHEC compliance testing, the LCS responses must be within 70 – 130% for Method 8260 and within the limits given in Appendix F for Method 624.1. High failures are acceptable for 8260 or 624.1, as long as the sample results are below detection level. Qualifiers cannot be used therefore low failures require a batch re-analysis. See Section 11 for QC evaluation. For samples analyzed from South Carolina that are not utilized for compliance purposes, in house established acceptance limits are utilized to demonstrate controlled analyses.

**STATE NOTE:** For EPA Region IV only (AL, FL, GA, KY, MS, NC, SC, TN), when running Acrolein and Acrylonitrile by V624.1, the QC criteria from EPA Method 603 should be used for control demonstration. The LCS criteria from EPA Method 603 include Acrolein recovery within 88-118% and Acrylonitrile recovery within 71-135%.

**9.2.4.9 Internal Standard Evaluation**

When a calibration is performed at the beginning of an analytical run:

The internal standard areas must be evaluated against the mid-point of the curve. Samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against mid-point of the curve.




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Then a CCV is analyzed, this is compared to the mid-point of the initial calibration curve. Additional samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against the previous acceptable CCV.

When an analytical run is started using a passing ICV (which is compared against the initial calibration mid-point to verify the calibration curve): Samples are analyzed within a 12-hour window, the internal standards of those samples are evaluated against the daily CCV. Then a CCV is analyzed, this is compared to the mid-point of the curve. Additional samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against the previous acceptable CCV.

### 9.3 Sample Preparation

Screening the sample prior to purge-and-trap analysis provides guidance on whether sample dilution is necessary and prevents contamination of the purge-and-trap system by screening of 5mL of sample using an HNU or equivalent portable PID.

#### 9.3.1 Homogenization and SubSampling

Compositing samples prior to GC/MS analysis – Site or project-specific requirements may require compositing of samples, which is performed according to the instructions below. Compositing of samples is only performed at the request of the client.

**9.3.1.1** All vials for the sample are combined in an appropriately sized volumetric flask that will allow for the least amount of headspace. (If 4 vials are to be composited then a 200mL volumetric flask will be used to combine the samples.) Practice special precautions to maintain zero headspace in the syringe.

**9.3.1.2** The samples must be cooled to 4 °C or less during composition to minimize the loss of volatiles. Sample vials may be placed in a tray of ice to prevent volatile loss during this process.

**9.3.1.3** Invert the volumetric flask three (3) times. Pour the volume out of the volumetric flask into the original 40mL VOA vial containers. The sample is now ready to be analyzed.

**NOTE:** Samples are not routinely composited; however, if site-specific requirements state procedures for compositing samples, the laboratory makes every effort to comply with those requirements.

### 9.4 Analysis

#### 9.4.1 Example Analytical Sequence

**9.4.1.1** Gas chromatographic analysis:

**9.4.1.1.1** Typical sequence order for loading the autosampler with calibration:

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Sample/QC Type	Use
Cleanup Blank	Verify system is contamination free
BFB Tune	Tuning criteria
Calibration standard(s)	Initial volatiles calibration and 5-point for GRO (if analyzed)
Second Source Calibration Verification (SSCV) or Initial Calibration Verification (ICV)	Verify initial calibration with second source.
Laboratory Control Sample	Laboratory blank, spiked with known amount(s) of analyte of interest
Matrix Spike/Matrix Spike Dup.	Sample spiked with known number(s) of analytes of interest
Method blank	Ensure that carry over has not occurred from the calibration standard and that the analytical system does not show contamination above the established detection limits
12-hour window	Client samples
Continuing Calibration Verification (CCV)	Single-point calibration verification standard, if needed.
12-hour window	Client samples

**9.4.1.1.2** Typical sequence order for loading the autosampler with calibration verification only:

Sample/QC Type	Use
Cleanup Blank	Verify system is contamination free
BFB Tune	Tuning criteria
Initial Calibration Verification (ICV)	Verify initial calibration.
Laboratory Control Sample	Laboratory blank, spiked with known amount(s) of analyte of interest
Matrix Spike/Matrix Spike Dup.	Sample spiked with known amount(s) of analytes of interest
Method blank	Ensure that carry over has not occurred from the calibration standard and that the analytical system does not show contamination above the established detection limits
12-hour window	Client samples
Continuing Calibration Verification (CCV)	Single-point calibration verification standard, if needed.
12-hour window	Client samples

**9.4.2** All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

**9.4.3** BFB tuning criteria and GC/MS calibration criteria must be met before analyzing samples

Load the unopened VOA vial onto the autosampler for analysis.

**9.4.4** After the sample has been analyzed on the instrument, check the pH of the sample using the remaining sample in the VOA vial. Use universal pH paper and record the sample pH to the nearest whole pH unit. Samples not passing the pH requirements are flagged with a "G1" qualifier. All samples that report 2-CEVE as a target analyte and have a pH < 2 are qualified with a "G2".

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**STATE NOTE:** For West Virginia compliance samples, check for residual chlorine after sample analysis. Samples containing residual chlorine must be flagged as such.

Sample Dilution -- When necessary, samples can be diluted before purging. This can be performed in a clean 50mL volumetric flask. The sample is measured through the use of an appropriate microliter syringes and added to the flask which is then filled with reagent water to the meniscus.

Surrogate/Internal Standards – The autosampler adds 1 $\mu$ L of the IS/surrogate mix to each sample. The addition of 1 $\mu$ L of the surrogate spiking/internal standard solution is equivalent to 16 $\mu$ g/L of each surrogate standard. Internal standard and surrogate standard are contained within the same spiking mix.

**SAC NOTE:** During analysis, 5 $\mu$ L of this solution is added to 5mL of sample, resulting in a concentration of 50  $\mu$ g/L of each surrogate and internal standard.

If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be re-analyzed at a higher dilution. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Secondary ion quantitation is allowed only when there are sample interferences with the routinely quantitated primary ion. When a sample is analyzed that has saturated the detector, the samples following must be analyzed for contamination. If any sample shows contamination, they must be re-analyzed.

#### 9.4.5 GC/MS Analysis - Water-miscible liquids

**9.4.5.1** Water-miscible liquids are analyzed as water samples after first diluting them at least 25-fold with laboratory water.

**9.4.5.2** Initial and serial dilutions can be prepared by pipetting a known amount of the sample to a 50mL volumetric flask and diluting to volume with organic-free reagent water. Transfer immediately to a clean/baked 40mL vial using a 5mL syringe.

**9.4.5.3** Alternatively, prepare dilutions directly in a clean 40mL vial filled with organic-free reagent water by adding at least 0.5 $\mu$ L, but not more than 25mLs of liquid sample. The sample is ready for addition of internal and surrogate standards. Proceed with Section 9.2.4.1.

#### 9.4.6 GC/MS Analysis - Sediment/soil and waste samples

These samples may contain percent quantities of purgeable organics that will contaminate the purge-and-trap system and require extensive cleanup and instrument downtime. The screening of samples is highly recommended. Screening data should be used in conjunction with site-specific DQOs, if known,






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to determine whether to use the low-concentration method (0.005 - 1mg/Kg) or the high-concentration method (>1mg/Kg).

**9.4.6.1** Low-concentration method -- This is designed for samples containing individual purgeable compounds of <1mg/Kg. It is limited to sediment/soil samples and waste that is of a similar consistency (granular and porous). The low-concentration method is based on purging a heated sediment/soil sample mixed with organic-free reagent water containing the surrogate and internal standards. All QC samples and standards are to be analyzed under the same conditions as the samples, using 5g of glass beads or equivalent blank matrix.

**STATE NOTE:** This option cannot be used for South Carolina samples. Please refer to ENV-SOP-MTJL-0129 that addresses Method 5035 for sample preparation.

**9.4.6.2** Use a 5g sample if the expected concentration is <0.1mg/Kg, or a 1g sample for expected concentrations between 0.1 and 1mg/Kg.

**9.4.6.3** The GC/MS system must be set up prior to the preparation of the sample to avoid loss of volatiles from standards and samples. Both the initial and daily calibration standards must be heated to 40°C purge temperature.

**9.4.6.4** The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents (using slow but precise movement to limit the loss of volatiles) of the sample container with a narrow metal spatula. Weigh the amount determined into a tared purge device. Note and record the actual weight to the nearest 0.1 g.

**9.4.6.5** Add nanopure water to the purging vial, which contains the weighed amount of sample, and place the vial in the purge-and-trap system.

**NOTE:** Prior to the placement of the vial, the procedures in Sections 9.4.6.5 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

**9.4.7** High-concentration method -- The method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. Wastes (i.e., petroleum and coke wastes) that are soluble in methanol are diluted. An aliquot of the extract is added to organic-free reagent water containing surrogate and internal standards. This may be purged at higher temperatures than ambient temperature as long as all calibration standards, field samples, and associated QC samples are purged at the same temperature and the laboratory demonstrates acceptable method performance. All samples with an expected concentration of >1.0 mg/Kg must be analyzed by this method.




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**STATE NOTE:** This method is not suitable for samples from South Carolina, North Carolina, or Indiana. South Carolina does not recognize the practices in sections 9.4.7.1 and 9.4.7.2. 5035 must be used for all high-level soil samples, see ENV-SOP-MTJL-0129.

**9.4.7.1** The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents (using slow but precise movement to limit the loss of volatiles) of the sample container with a narrow metal spatula. For sediment/soil and solid wastes that are insoluble in methanol, weigh 10g (wet weight) of sample into a tared 40mL vial. Use a top-loading balance. Note and record the actual weight to 0.1g. For waste that is soluble in methanol, tetraglyme, or PEG, weigh 10g (wet weight) into a 40mL vial.

**9.4.7.2** Add 10mL Methanol and vortex. See ENV-SOP-MTJL-0129.

**NOTE:** Sections 9.4.7.1 and 9.4.7.2 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

**9.4.7.3** The GC/MS system must be set up as in Sections 9.

**9.4.7.4** If a screening procedure was followed, use the estimated concentration to determine the appropriate volume. If the sample was submitted as a high-concentration sample, start with 200 $\mu$ L.

**9.4.7.5** In a clean/baked vial filled with reagent water, inject the corresponding aliquot of methanol extract. Immediately cap and place in the autosampler. The autosampler adds 1 $\mu$ L of the IS/surrogate mix to all of the samples.

**9.4.7.6** Proceed with the analysis as outlined in Sections 9. Analyze all blanks on the same instrument as that used for the samples.

**9.4.7.7** For a matrix spike in the high-concentration of sediment/soil samples, Add a 200 $\mu$ L aliquot of this extract to 5mL of organic-free reagent water for purging (as per Section 9.4.7.6) in a clean/baked 40mL VOA vial and add 20 $\mu$ L spiking solution, 1 $\mu$ L internal and surrogate standard solution (IS/Surr solution added by autosampler).

**9.4.7.8** Data generated from soil samples prepared in methanol on Agilent™ 5977A or 5977B instruments (with an extractor ion or high efficiency source) may be reported to low-level MDL/RL values due to the enhanced sensitivity associated with these instruments.




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## 10.0 DATA ANALYSIS AND CALCULATIONS

### 10.1 Qualitative Identification

The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. All hits must be visually compared to the reference spectrum for confirmation. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the criteria below are met.

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

The RRT of the sample component is within + 0.06 RRT units of the RRT of the standard component.

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

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

Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra are important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum contains extraneous ions contributed by the co-eluting compound.

#### 10.1.1 Tentatively Identified Compounds (TICS)

##### TIC's - Tentatively Identified Compounds

Periodically, clients may request additional identification of compounds that are not normally calibrated. This identification is limited to the compounds in the current mass spectral library employed by Pace National.

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative




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identification. The necessity to perform this type of identification is determined by the type of analyses being conducted. At the client request, when serving the role of QA (or referee) laboratory, tentatively identified compounds (TICs) must always be reported. Guidelines for making tentative identification are:

- (1) Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.
- (2) The relative intensities of the major ions should agree within 15% to be consistent with target compound list identification. (Example: For an ion with an abundance of 50% in the standard spectrum the corresponding sample ion abundance must be between 20 and 80%).
- (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 or presence of co-eluting compounds.
- (5) Ions present in the reference spectrum but not in the sample spectrum must be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches does the mass spectral interpretation specialist assign a tentative identification.

**10.1.1.1** Routinely, Pace National employs a minimum Q value of 85% for tentative identifications and a minimum concentration of 10ppb. Peaks below a Q value of 85% but above 10ppb may be reported as "Unknown". Any identified peaks below 10ppb are removed as these could result from baseline noise or other interferences, not necessarily attributable to the field sample or reliably quantifiable using GCMS technology. Additionally, any peaks that are attributable to instrument contamination (e.g., siloxanes) are also removed.

**10.1.1.2** If multiple TICs, with same exact name, exist for a sample, the LIMS will only display the TIC with the highest quality match per sample.

**10.1.1.3** TIC names assigned as "Unknown" may initially have the same name as another "Unknown" until parsed and displayed in LIMS where it is given a hyphen and incremental number which then becomes a unique TIC (e.g., Unknown-1).

**10.1.1.4** When reporting "Total TIC" for any client sample, only concentrations per above requirements will be used to sum the Total TIC concentration.

## **10.1.2 Manual Integration**

Manual changes to automated integration are called manual integration. Manual integration is sometimes necessary to correct inaccurate automated integrations but must never be used to meet QC criteria or to substitute for proper instrument

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maintenance and/or method set-up. To assure that all manual integrations are performed consistently and are ethically justified, all manual integrations must be performed, reviewed, and recorded in accordance with corporate SOP ENV-SOP-CORQ-0006, *Manual Integration*.

**10.2 Quantitative Identification**

When a compound has been identified, the quantitation of that compound is based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation is accomplished using the internal standard technique, as described in Section 9. The internal standard used must be the one nearest the retention time of that of a given analyte.

Sediment/soil samples are reported on a dry weight basis, while sludge and wastes are reported on a wet weight basis. The percent dry weight of the sample (see Section 9.7) must be reported along with the data in either instance. At Pace National, the dry weight conversion calculations for sample reporting are performed by the LIMS system. [Dry weight only when requested]. The LIMS Final Client Report represents the reporting basis as either wet weight or dry weight, depending upon the calculation used.

**10.2.1 Concentration of Target Analytes in Water and Water-Miscible Waste**

$$\text{Concentration(ug/L)} = \frac{(A_x)(I_s)(D)}{(A_{is})(\text{ave.RF})(V_s)}$$

where:

$A_x$  = Area (or height) of the peak for the analyte in the sample.

$A_{is}$  = Area (or height) of the peak for the internal standard.

$I_s$  = Mass (amount) of the internal standard in the concentrated sample extract (ng). This is not just the mass injected into the instrument, but the total mass of internal standard in the concentrated extract.

$D$  = Dilution factor if the sample or extract was diluted prior to analysis. If no dilution was made,  $D = 1$ . The dilution factor is always dimensionless.

ave.RF = Mean response factor from the initial calibration.

$V_s$  = Volume of the aqueous sample extracted or purged (mL). If units of liters are used for this term, multiply the results by 1000.

**10.2.2 Concentration of Target Analytes in Sediment/Soil, Sludge, and Waste****10.2.2.1 High-concentration procedure**

$$\text{Concentration(ug/L)} = \frac{(A_x)(I_s)(V_t)}{(A_{is})(\text{ave.RF})(V_i)(W_s)}$$

where:

$A_x$ ,  $I_s$ ,  $A_{is}$ , ave.RF are the same as in water and water-miscible waste above.

$V_t$  = Volume of total extract ( $\mu\text{L}$ ) (use 10,000 $\mu\text{L}$  or a factor of this when dilutions are made).

$V_i$  = Volume of extract added ( $\mu\text{L}$ ) for purging.

$W_s$  = Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

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**10.2.2.2 Low-concentration procedure**

$$\text{Concentration}(\mu\text{g/L}) = \frac{(A_x)(I_s)(V_t)}{(A_{is})(\text{ave.RF})(V_i)(W_s)}$$

where:

A, I<sub>s</sub>, A<sub>is</sub>, RF are the same as in water and water-miscible waste above.

V<sub>t</sub> = Volume of total extract (μL) (use 10,000μL or a factor of this when dilutions are made).

V<sub>i</sub> = Volume of extract added (μL) for purging.

W<sub>s</sub> = Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

**10.2.2.3 Soil Weight determination with Methanol (samples received with MeOH).**

SoilWeight = VialTotalWeight(vial, soil, MeOH) – TareVialWeight – MeOHWeight.

**10.2.3** In order to report results for volatiles analysis of samples prepared in methanol containing significant moisture (>10%) content on an "as received" basis, the calculated concentration needs to be corrected using the total solvent/water mixture volume represented as V<sub>t</sub>. This total solvent/water volume is calculated as follows:

$$\mu\text{L solvent/water } V_t = \left[ \frac{\text{mL of solvent} + (\% \text{ moisture} \times \text{g of sample})}{100} \right] \times 1000 \mu\text{L/mL}$$

**10.2.4 Percent Error (%Error)**

$$\%Error = \frac{x_i - x'_i}{x_i} * 100$$

where:

x'<sub>i</sub> = Measured amount of analyte at the calibration level *i*, in mass or concentration units

x<sub>i</sub> = True amount of analyte at calibration level *i*, in mass or concentration units

**10.2.5** Relative Standard Error (%RSE) – As an alternative to using the average response factor when using Method 624.1, the quality of the calibration may be evaluated using the Relative Standard Error (RSE). The acceptance criterion for the RSE is the same as the acceptance criterion for Relative Standard Deviation (RSD), in the method. RSE is calculated as:

$$\%RSE = 100 \times \frac{\sum_{i=1}^n \left[ \frac{x'_i - x_i}{x_i} \right]^2}{(n - p)}$$

where:

x'<sub>i</sub> = Calculated concentration at level *i*

x<sub>i</sub> = Actual concentration of the calibration level *i*

n = number of calibration points

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p = Number of terms in the fitting equation (average = 1; linear = 2; quadratic = 3)

**10.3 Calculations**

See the Laboratory Quality Assurance Manual for equations for common calculations.

**11.0 QUALITY CONTROL AND METHOD PERFORMANCE****11.1 Quality Control**

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples. If batch exceeds, 20 samples, every 20.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples. If batch exceeds, 20 samples, every 20.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per batch of 20 or fewer samples. If batch exceeds, 20 samples, every 20.
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples. If batch exceeds, 20 samples, every 20.
Sample Duplicate	Not required for this method
Trip Blank	As requested by client
Surrogate	All field and QC samples
Internal Standard	All field and QC samples

**11.2 Instrument QC**

The following Instrument QC checks are performed. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Tune	Prior to ICAL and every 12-hour period of sample analysis
Initial Calibration	At instrument setup and prior to sample analysis
Initial Calibration Verification	After each ICAL prior to sample analysis
Initial Calibration Blank	After each ICAL prior to sample analysis and as needed
Continuing Calibration Verification	Daily before analysis, after every 12-hour period of sample analysis and at the end of the analytical sequence
Continuing Calibration Blank	After each CCV and as needed to verify the system is contamination free
RT Window	Once per ICAL and at the beginning of the analytical sequence
Relative Retention Time	With each sample
Breakdown Check	Not required for this method

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**11.3 Method Performance****11.3.1. Method Validation****11.3.1.1 Detection Limits** **11.3.1.2 Method 624.1 Requirements**

**11.3.1.2.1** Establish MDLs for the analytes of interest using the MDL procedure at 40 CFR part 136, appendix B. The laboratory's MDLs must be equal to or lower than those listed in Table 1 for those analytes which list MDL values, or lower than one-third the regulatory compliance limit, whichever is greater. For MDLs not listed in Table 1, the laboratory must determine the MDLs using the MDL procedure at 40 CFR part 136, appendix B under the same conditions used to determine the MDLs for the analytes listed in Table 1. All procedures used in the analysis must be included in the DOC.

<b>624.1 Purgeable Analytes</b>	<b>MDL (ug/L)</b>	<b>Limit for s (%)</b>	<b>Range for <math>\bar{X}</math> (%)</b>
Acrolein		30	50-150
Acrylonitrile		30	50-150
Benzene	4.4	33	75-125
Bromodichloromethane	2.2		
Bromoform	4.7		
Bromomethane		90	D-242
Carbon tetrachloride	2.8	26	65-125
Chlorobenzene	6.0	29	82-137
Chloroethane		47	42-202
2-Chloroethylvinyl ether		130	D-252
Chloroform	1.6	32	68-121
Chloromethane		472	D-230
Dibromochloromethane	3.1	30	69-133
1,2-Dichlorobenzene		31	59-174
1,3-Dichlorobenzene		24	75-144
1,4-Dichlorobenzene		31	59-174
1,1-Dichloroethane	4.7	24	71-143
1,2-Dichloroethane	2.8	29	72-137
1,1-Dichloroethene	2.8	40	19-212
trans-1,2-Dichloroethene	1.6	27	68-143
1,2-Dichloropropane	6.0	69	19-181
cis-1,3-Dichloropropene	5.0	79	5-195
trans-1,3-Dichloropropene		52	38-162
Ethylbenzene	7.2	34	75-134
Methylene chloride	2.8	192	D-205
1,1,2,2-Tetrachloroethane	6.9	36	68-136
Tetrachloroethene	4.1	23	65-133
Toluene	6.0	22	75-134
1,1,1-Trichloroethane	3.8	21	69-151
1,1,2-Trichloroethane	5.0	27	75-136
Trichloroethene	1.9	29	75-138
Trichlorofluoromethane		50	45-158
Vinyl chloride		100	D-218

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- 11.3.1.3** Prepare and analyze four LCSs by adding an appropriate volume of the second source standard (Section 7.5.2.1) to each of four aliquots of reagent water.
- 11.3.1.4** Calculate the average percent recovery ( $\bar{X}$ ) and the standard deviation of the percent recovery (s) for each analyte using the four results.
- 11.3.1.5** For each analyte, compare s and  $\bar{X}$  with the corresponding acceptance criteria for precision and recovery in Section 10.1.1.1. For analytes not listed, DOC QC acceptance criteria must be developed by the laboratory. Alternatively, acceptance criteria for analytes not listed may be based on laboratory control charts. If s and  $\bar{X}$  for all analytes of interest meet the acceptance criteria, system performance is acceptable, and analysis of blanks and samples may begin. If any individual s exceeds the precision limit or any individual  $\bar{X}$  falls outside the range for recovery, system performance is unacceptable for that analyte.

**NOTE:** The large number of analytes present, a substantial probability that one or more will fail at least one of the acceptance criteria when many or all analytes are determined simultaneously. Therefore, the analyst is permitted to conduct a “re-test” as described here.

When one or more of the analytes tested fail at least one of the acceptance criteria, repeat the test for only the analytes that failed. If results for these analytes pass, system performance is acceptable, and analysis of samples and blanks may proceed. If one or more of the analytes again fail, system performance is unacceptable for the analytes that failed the acceptance criteria. Correct the problem and repeat the test. To maintain the validity of the test and re-test, system maintenance and/or adjustment is not permitted between this pair of tests.

**11.3.1.6 Batches**

**11.3.1.6.1 Extraction Batches:**

Extraction batches are defined as sets of 1 - 20 samples. Extraction batches must include the following: 1 method blank, 1 Laboratory Control Sample (LCS), 1 Matrix Spike/Spike Duplicate (MS/MSD) pair (if sufficient sample is available). Exceptions are made for waste dilution samples where the minimum batch QC must include a blank, and an LCS.

Additional instructions on Batch QC including required frequency and corrective actions can be found in Section 12 while acceptance criteria are found in the LIMS.

**STATE NOTE:** For samples from FL, AZ, MN, and MA, 1 Laboratory Control Sample/Laboratory Control Sample Duplicate pair (LCS/LCSD) is required per batch.

**11.3.1.6.2 Analytical Batches (sequences):**




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Analytical batches analysis must include the following: 1 Initial Calibration Verification (ICV) and BFB tune at the beginning of run, and/or 1 Continuing Calibration Verification (CCV) and BFB tune every 12 hours.

- 11.3.1.7** Perform BFB tune every 12 hours for 624.1, 8260B, 8260C, and 6200B. BFB tuning for method 8260D is prior to calibration curve. Tuning acceptance criteria are presented in Section 9.1.2.1.4. Once the data is acquired, the following options are available for acquiring the spectra for reference to meet the BFB tuning requirements. It is recommended that each initial tune verification utilize the "Autofind" function and be set up to look at three scans (the apex &+1 scan) and average the three scans then perform background subtraction. If Autofind is not utilized, select the mass spectrum at the peak apex for evaluation, or use an average mass spectrum across the entire BFB peak. Background subtraction is conducted using a single scan prior to the elution of BFB but no more than 20 scans prior. No part of the BFB peak or any other discrete peak should be subtracted.

If a tune cannot be achieved that meets the criteria in Section 9.1.2.1.4 retune the mass spectrometer and run a new calibration or maintenance may be necessary.

**11.3.1.7.1 Method 624.1 Requirements**

- 11.3.1.7.1.1** Verify calibration after the criteria for BFB are met and prior to analysis of a blank or sample. After verification, analyze a blank to demonstrate freedom from contamination and carry-over at the MDL. Tests for BFB, the CCV, and the blank are outside of the 12-hour shift, and the 12-hour shift includes samples and matrix spikes and matrix spike duplicates. The total time for analysis of BFB, the CCV, the blank, and the 12-hour shift must not exceed 14 hours.

- 11.3.1.8** Run a minimum of a 5-point initial calibration curve (3-point can be used if 624/6200B are being run independently of 8260B), using the primary source standards each time major instrument maintenance occurs, or if the CCV does not meet acceptance criteria. Acceptance criteria for initial calibration are presented in Section 9.2. Calibration is verified by analyzing Second Source Calibration Verification (SSCV) standard; acceptance criteria for the SSCV are presented in Section 9.2.3.3.1.

**11.3.1.8.1 Method 624.1**

If the RSD is less than 35%, the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to fit a linear or quadratic regression of response ratios vs. concentration ratios. If used, the regression must be weighted inversely proportional to concentration. The coefficient of determination (R<sup>2</sup>) of the weighted regression must be greater than 0.920 (this value roughly corresponds to the RSD limit of 35%). Alternatively, the relative standard error may be used as an acceptance criterion.




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As with the RSD, the RSE must be less than 35%. If an RSE less than 35% cannot be achieved for a quadratic regression, system performance is unacceptable, and the system must be adjusted and re-calibrated.

**STATE NOTE:** South Carolina does not allow the use of quadratic regression for compounds that have previously demonstrated linearity.

**11.3.1.9** Run a mid-point Continuing Calibration Verification (CCV) using the primary source standards every 12 hours before sample analysis and every 12 hours during an analytical sequence for 8260B, 8260C, 8260D, 624.1 and 6200B. See sections 9.2.4-9.2.4.5 for acceptance criteria.

**11.3.1.10** Retention Time Evaluation

**11.3.1.10.1** Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 10 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

**11.3.1.10.2** Evaluation of retention times - The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units. Late-eluting target analytes usually have much better agreement.

**11.3.1.11** Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes baking traps and columns, polishing detector windows, changing injection port liners, changing pump oil, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

**11.3.1.12** METHOD BLANK - The analyst must confirm that this blank was analyzed at the required frequency of 1 per batch of 20 samples. The method blank must not exhibit any contamination of any analyte above the method detection limit for any of the method target analytes.

**11.3.1.12.1** If more than one instrument blank or method blanks are analyzed, evaluate, and assess the blank and field samples under the same conditions for possible mid-level standard carryover using the subsequent blank after the mid-level standard on a per analyte basis.

**11.3.1.12.2** Method 624.1 – If any analyte of interest is found in the blank at a concentration greater than the MDL for the analyte, at




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a concentration greater than one-third the regulatory compliance limit, or at a concentration greater than one-tenth the concentration in a sample analyzed during the 12-hour shift, whichever is greater; analysis of samples must be halted, and samples affected by the blank must be re-analyzed. If, however, continued re-testing results in repeated blank contamination, the laboratory must document and report the failures (e.g., as qualifiers on results), unless the failures are not required to be reported as determined by the regulatory/control authority. Results associated with blank contamination for an analyte regulated in a discharge cannot be used to demonstrate regulatory compliance.

**11.3.1.13 LABORATORY CONTROL SAMPLES** - Assess that the LCS was prepared at the required frequency of 1 per batch of 20. If the same injection is used for the LCS and the CCV, ensure that no more than 20 samples are analyzed in conjunction. Routine LCS Control limits are presented in the LIMS.

**STATE NOTE:** For all samples analyzed from South Carolina, the LCS/LCSD RPD must be <20%, recoveries must be 70-130% in a soil matrix, and recoveries must be within the following limits in a water matrix:

Benzene	70 – 130%
Bromodichloromethane	70 – 130%
Bromoform	70 – 130%
Bromomethane	70 – 130%
Carbon tetrachloride	70 – 130%
Chlorobenzene	70 – 130%
Chloroethane	70 – 130%
2-Chloroethyl vinyl ether	70 – 130%
Chloroform	70 – 130%
Chloromethane	70 – 130%
Dibromochloromethane	70 – 130%
Dichlorodifluoromethane	70 – 130%
1,2-Dichlorobenzene	70 – 130%
1,3-Dichlorobenzene	70 – 130%
1,4-Dichlorobenzene	70 – 130%
1,1-Dichloroethane	70 – 130%
1,2-Dichloroethane	70 – 130%
1,1-Dichloroethene	70 – 130%
Trans-1,2-Dichloroethene	70 – 130%
1,2-Dichloropropane	70 – 130%
Cis-1,3-Dichloropropene	70 – 130%
Trans-1,3-Dichloropropene	70 – 130%
Ethyl benzene	70 – 130%
Methylene chloride	70 – 130%
Methyl tert-butyl ether	70 – 130%
1,1,2,2-Tetrachloroethane	70 – 130%
Tetrachloroethene	70 – 130%
Toluene	70 – 130%
1,1,1-Trichloroethane	70 – 130%

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1,1,2-Trichloroethane	70 – 130%
Trichloroethene	71 – 130%
Trichlorofluoromethane	70 – 130%
Vinyl chloride	70 – 130%
Xylenes, total	70 – 130%

**11.3.1.14 Method 624.1 Requirements**

**11.3.1.14.1** Compare the percent recovery (Q) for each analyte with its corresponding QC acceptance criterion in the following table. For analytes of interest not listed in the following table, use QC acceptance criteria developed for the LCS. If the recoveries for all analytes of interest fall within their respective QC acceptance criteria, analysis of blanks and field samples may proceed. If any individual Q falls outside the range, proceed according to Section 8.3.5.1.4.

624.1 Purgeable Analytes	Range for Q (%)
Acrolein	60-140
Acrylonitrile	60-140
Benzene	65-135
Bromodichloromethane	65-135
Bromoform	70-130
Bromomethane	15-185
Carbon tetrachloride	70-130
Chlorobenzene	65-135
Chloroethane	40-160
2-Chloroethylvinyl ether	D-225
Chloroform	70-135
Chloromethane	D-205
Dibromochloromethane	70-135
1,2-Dichlorobenzene	65-135
1,3-Dichlorobenzene	70-130
1,4-Dichlorobenzene	65-135
1,1-Dichloroethane	70-130
1,2-Dichloroethane	70-130
1,1-Dichloroethene	50-150
trans-1,2-Dichloroethene	70-130
1,2-Dichloropropane	35-165
cis-1,3-Dichloropropene	25-175
trans-1,3-Dichloropropene	50-150
Ethylbenzene	60-140
Methylene chloride	60-140
1,1,2,2-Tetrachloroethane	60-140
Tetrachloroethene	70-130
Toluene	70-130
1,1,1-Trichloroethane	70-130
1,1,2-Trichloroethane	70-130
Trichloroethene	65-135
Trichlorofluoromethane	50-150
Vinyl chloride	5-195

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**11.3.1.14.2** Repeat the test only for those analytes that failed to meet the acceptance criteria (Q). If these analytes now pass, system performance is acceptable, and analysis of blanks and samples may proceed. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, repeat the test using a fresh LCS or an LCS prepared with a fresh QC check sample concentrate, or perform and document system repair. Subsequent to repair, repeat the calibration verification/LCS test. If the acceptance criteria for Q cannot be met, re-calibrate the instrument. To maintain the validity of the test and re-test, system maintenance and/or adjustment is not permitted between the pair of tests

**11.3.1.15 MATRIX SPIKE/MATRIX SPIKE DUPLICATE ASSESSMENT:** Assess that matrix spike/matrix spike duplicates were analyzed at required frequency of 1 per batch of 20 if volume allows.

- The analyst also verifies that the samples were spiked at the appropriate level.
- The order of preference for spiking levels is as follows:
  - 1) If the target analyte concentrations are known, spike to increase the background concentration by a factor of approximately two
  - 2) If an action level exists, spike at this level
  - 3) If neither of the first two conditions applies, spike at a level that corresponds between the low and mid-level calibration standards.
  - 4) All RPD results must be within the indicated control limits found in the LIMS.

Acceptance criteria are that all %Recovery and/or RPD results must be within the indicated control limits on the appropriate MS control charts. See the LIMS for LCS/LCSD & MS/MSD limits and QC acceptance.

**STATE NOTE:** For all water samples analyzed from South Carolina, the MS/MSD recoveries must be within the most stringent limits comparing in-house derived recovery limits to those given in Table 3 of Method 608. The following are the current water limits:

Benzene	58.6 - 133%
Bromodichloromethane	69.2 – 127%
Bromoform	66.3 – 140%
Bromomethane	16.6 – 183%
Carbon tetrachloride	70 – 139%
Chlorobenzene	70.1 – 130%
Chloroethane	33.3 – 155%
2-Chloroethyl vinyl ether	5 – 149%
Chloroform	66.1 – 133%




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Chloromethane	40.7 – 139%
Dibromochloromethane	69.2 – 127%
Dichlorodifluoromethane	42.2 – 146%
1,2-Dichlorobenzene	77.4 – 127%
1,3-Dichlorobenzene	67.9 – 136%
1,4-Dichlorobenzene	74.4 – 123%
1,1-Dichloroethane	64 – 134%
1,2-Dichloroethane	60.7 – 132%
1,1-Dichloroethene	48.8 – 144%
Trans-1,2-Dichloroethene	61 – 132%
1,2-Dichloropropane	69.7 – 132%
Cis-1,3-Dichloropropene	71.1 – 129%
Trans-1,3-Dichloropropene	66.3 – 136%
Ethyl benzene	62.7 – 136%
Methylene chloride	61.5 – 125%
Methyl tert-butyl ether	61.4 – 136%
1,1,2,2-Tetrachloroethane	64.9 – 145%
Tetrachloroethene	64 – 141%
Toluene	67.8 – 124%
1,1,1-Trichloroethane	58.7 – 134%
1,1,2-Trichloroethane	74.1 – 130%
Trichloroethene	71 – 148%
Trichlorofluoromethane	39.9 – 165%
Vinyl chloride	44.3 – 143%
Xylenes, total	65.6 – 133%

**11.3.1.16 Method 624.1 Requirements**

**11.3.1.16.1** Spike at least 5% of the samples in duplicate from each discharge being monitored to assess accuracy (recovery and precision). If direction cannot be obtained from the data user, the laboratory must spike at least one sample in duplicate per extraction batch of up to 20 samples with the analytes in the table in Section 11.3.1.1.1. Spiked sample results should be reported only to the data user whose sample was spiked, or as requested or required by a regulatory/control authority, or in a permit.

**11.3.1.16.2** If the concentration of a specific analyte will be checked against a regulatory concentration limit, the concentration of the spike should be at that limit; otherwise, the concentration of the spike should be one to five times higher than the background concentration, at or near the mid-point of the calibration range, or at the concentration in the LCS whichever concentration would be larger.

**11.3.1.16.3** Compare the percent recoveries (P1 and P2) and the RPD for each analyte in the MS/MSD aliquots with the corresponding QC acceptance criteria in the following table. A laboratory may develop and apply QC acceptance criteria more restrictive than the presented criteria if desired.

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624.1 Purgeable Analytes	Range for P1, P2 (%)	Limit for RPD
Acrolein	40-160	60
Acrylonitrile	40-160	60
Benzene	37-151	61
Bromodichloromethane	35-155	56
Bromoform	45-169	42
Bromomethane	D-206	61
Carbon tetrachloride	70-140	41
Chlorobenzene	37-160	53
Chloroethane	14-230	78
2-Chloroethylvinyl ether	D-305	71
Chloroform	51-138	54
Chloromethane	D-273	60
Dibromochloromethane	53-149	50
1,2-Dichlorobenzene	18-190	57
1,3-Dichlorobenzene	59-156	43
1,4-Dichlorobenzene	18-190	57
1,1-Dichloroethane	59-155	40
1,2-Dichloroethane	49-155	49
1,1-Dichloroethene	D-234	32
trans-1,2-Dichloroethene	54-156	45
1,2-Dichloropropane	D-210	55
cis-1,3-Dichloropropene	D-227	58
trans-1,3-Dichloropropene	17-183	86
Ethylbenzene	37-162	63
Methylene chloride	D-221	28
1,1,2,2-Tetrachloroethane	46-157	61
Tetrachloroethene	64-148	39
Toluene	47-150	41
1,1,1-Trichloroethane	52-162	36
1,1,2-Trichloroethane	52-150	45
Trichloroethene	70-157	48
Trichlorofluoromethane	17-181	84
Vinyl chloride	D-251	66

**11.3.1.16.4** If any individual P falls outside the designated range for recovery in either aliquot, or the RPD limit is exceeded, the result for the analyte in the unspiked sample is suspect.

**11.3.1.16.5** If in-house QC limits are developed, at least 80% of the analytes tested in the MS/MSD must have in-house QC acceptance criteria that are tighter than those in Section 10.10.1.3 and the remaining analytes (those other than the analytes included in the 80%) must meet the acceptance criteria in Section 11.3.1.1.1. If an in-house QC limit for the RPD is greater than the limit in Section 10.10.1.3, then the limit in the table must be used. Similarly, if an in-house lower limit for recovery is below the lower limit in Section 11.3.1.1.1, then the lower limit in the table must be used, and if an in-house upper limit for recovery is above the upper limit in Section 11.3.1.1.1, then the upper limit in the table must be used.






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**11.3.1.17 SURROGATE EVALUATION:** Check the surrogate calculations for correctness for all samples, blanks, ICV/CCV/SSCV, LCS/LCSD, MS and MSD. Acceptance criteria can be found in the LIMS: The surrogate recoveries for all QC samples must be within established control limits.

**11.3.1.17.1 Method 624.1 –** Spike the surrogates into all samples, blanks, LCSs, and MS/MSDs. Compare surrogate recoveries against limits must be developed by the laboratory. In-house QC acceptance criteria must be updated at least every two years. If any recovery fails its criteria, attempt to find, and correct the cause of the failure.

**11.3.1.18 INTERNAL STANDARD AREA COUNT:** When a calibration is performed at the beginning of an analytical run, the internal standard areas must be evaluated against the mid-point of the curve. Internal standard responses must be -50% to 200% to be acceptable. Samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against mid-point of the curve. Then a CCV is analyzed, this is compared to the mid-point of the initial calibration curve. Additional samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against the previous acceptable CCV. When an analytical run is started using a passing ICV (which is compared against the initial calibration mid-point to verify the calibration curve): Samples are analyzed within a 12-hour window, the internal standards of those samples are evaluated against the daily CCV. Then a CCV is analyzed, this is compared to the mid-point of the curve. Additional samples are analyzed within a 12-hour window; the internal standards of those samples are evaluated against the previous acceptable CCV.

**CLIENT NOTE:** For Marathon, the internal standard area counts for all calibration standards, QC samples, and samples for quantitation must not change by a factor of greater than (-50% to +130%) as per section 9.2.4

**11.3.1.18.1 Method 624.1 –** The responses of each internal standard in each sample, blank, and MS/MSD must be within 50% to 200% (1/2 to 2×) of its respective response in the mid-point calibration standard. If, as a group, all internal standards are not within this range, perform and document system repair, repeat the calibration verification/LCS test, and re-analyze the affected samples. If a single internal standard is not within the 50% to 200% range, use an alternative internal standard for quantitation of the analyte referenced to the affected internal standard. It may be necessary to use the data system to calculate a new response factor from calibration data for the alternative internal standard/analyte pair. If an internal standard fails the 50–200% criteria and no analytes are detected in the sample, ignore the failure, or report it if required by the regulatory/control authority.

**11.3.1.19 SECOND SOURCE:** The second source calibration verification or initial calibration verification standard must be analyzed following each new initial calibration to verify the validity of the calibration standards. The recovery of




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the analytes in the SSCV (ICV) must be within 30% of the expected concentration for CCC and SPCC compounds and within 40% for non-CCC/SPCC compounds. Poor performers listed in section 9.2.4.7 must recover within in-house calculated LCS recovery acceptance limits.

**STATE NOTE:** For all samples analyzed from South Carolina, the SSCV must recover  $\pm 30\%$  for all target analytes.

**STATE NOTE:** For all samples analyzed from Minnesota, the reporting limit must be verified at least monthly. The reporting limit verification (RLV) must recovery within  $\pm 40\%$  of the expected concentration. If these criteria are not met, the RLV may be re-analyzed once, instrument maintenance can be performed, or a higher concentration standard can be analyzed. If a higher concentration standard is utilized, the reporting limit must be raised to the higher level verified.

**11.3.1.19.1** Method 624.1 Calibration verification/LCS—The working calibration curve or RF must be verified immediately after calibration and at the beginning of each 12-hour shift by the measurement of an LCS. The LCS must be from a source different from the source used for calibration, but may be the same as the sample prepared for the DOC.

**11.3.1.20** The laboratory participates in semi-annual proficiency testing that meets and/or exceeds the requirements of the Quality Control Sample as listed in the published Standard Method SM 6200B-2011, *Volatile Organic Compounds*, and SM 6020-2011, *Quality Assurance/Quality Control*.

**11.3.1.21** For sample analyzed per the requirements of Method 8000D, the LLOQ (see Section 12.7.1) must be verified at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels.

**11.3.1.21.1** The LLOQ verification (to be performed after the initial calibration) is prepared by spiking a clean control material with the analyte(s) of interest at 0.5-2 times the LLOQ concentration level(s).

**11.3.1.21.2** The LLOQ check is carried through the same preparation and analytical procedures as environmental samples and other QC samples.

**11.3.1.21.3** It is recommended to analyze the LLOQ verification on every instrument where data is reported; however, at a minimum, the lab must rotate the verification among similar analytical instruments such that all are included within three (3) years.

**11.3.1.21.4** Recovery of target analytes in the LLOQ verification must be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria  $\pm 20\%$  (i.e., lower limit minus 20% and upper limit plus 20%) may be used for the LLOQ acceptance criteria.




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## 12.0 DATA REVIEW AND CORRECTIVE ACTION

### 12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MTJL-0038, *Data Review* for specific instructions and requirements for each step of the data review process.

### 12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.




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Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

- 12.3** SITE-SPECIFIC requirements and STATE SPECIFIC criteria must be reviewed and used, if known, for data review.
- 12.4** All data must undergo a primary review by the analyst. The analyst must check the performance of the initial calibration, mid-point check standard, and continuing calibrations to ensure that they meet the criteria of the method. The analyst must review any sample that has quantifiable compounds and make sure that they have been confirmed. The analyst must also verify that reported results are derived from quantitation between the MDL and the highest standard of the initial calibration curve. All calculations must be checked (any dilutions, %solids, etc.). Data must be checked for the presence or absence of appropriate flags. Comments must be noted when data is flagged.
- 12.5** Tune Check - A successful BFB tune must be achieved prior to initial calibration or daily calibration verification. If a tune does not meet the acceptance criteria detailed above, then re-inject the tuning solution. If the failure persists, instrument maintenance or detector adjustment is required. The instrument is equipped with detector adjustments in routines called "Autotunes" that can make minor adjustments to m/z ratios and detector setting and can align the analytical system to return the system to peak performance. If after performing the Autotune routine, the injected tuning standard still fails, the system may require injector and/or detector cleaning, column cutting or replacement, injection liner cleaning or replacement, or other maintenance as specified by the manufacturer.
- 12.6** INITIAL AND CONTINUING CALIBRATION VERIFICATION STANDARD: An Initial Calibration Verification (ICV) standard is analyzed before sample analysis can begin and a continuing calibration verification (CCV) standard was analyzed every 12 hours and meets the criteria in Section 9.0. If these criteria are exceeded and analysis of a second consecutive (immediate) calibration verification fails to produce results within acceptance criteria, corrective actions shall be performed. The laboratory shall demonstrate acceptable performance after the final round of corrective action with two consecutive calibration verifications, or a new initial instrument calibration shall be performed.
- Method 8000D:** To determine calibration function acceptability, refit the initial calibration data back to the calibration model and calculate %Error (see Section 10.2.4). Percent error between the calculated and expected amounts of an analyte must be  $\leq 30\%$  for all standards. For some data uses,  $\leq 50\%$  may be acceptable for the lowest calibration point.
- 12.7** METHOD BLANK: Blank contamination above the report limit – All samples containing detectable amounts above the reporting limit must be re-analyzed or qualified. Samples with no detectable amounts above the reporting limit do not require re-analysis, but the samples must be qualified with blank contamination and it must be mentioned in the case narrative in the data package.

General guidelines for qualifying sample results with regard to method blank quality are as follows:

- If the method blank concentration is less than the MDL and sample results are greater than the RL, then no qualification is required.
- No qualification is necessary when an analyte is detected in the method blank but not in the associated samples.




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- If the concentration in a sample is more than ten times the concentration in the method blank, then no qualification is required.
- If the method blank concentration is greater than the MDL but less than the RL and sample results are greater than the MDL, then qualify associated sample results to indicate that analyte was detected in the method blank.
- If the method blank concentration is greater than the RL, further corrective action and qualification is required. An analyst should consult their supervisor for further instruction.

Instrument blanks may be injected at any time in the sequence to verify absence of contamination. The source of contamination must be investigated and reduced or eliminated. Any time contamination is noted in the method blank, the situation and impact on the data should be discussed in the case narrative.

**12.7.1 Method 8000D:** When samples that are extracted together are analyzed on separate instruments or in separate analytical shifts, the method blank associated with those samples (e.g., extracted with the samples) must be analyzed on at least one of those instruments. A solvent blank must be analyzed on all other instruments on which the set of samples was analyzed to demonstrate the instrument is not contributing contaminants to the samples. At least one method blank or instrument blank must be analyzed on every instrument after calibration standard(s) and prior to the analysis of any samples.

When sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same cleanup procedures.

Results of the method blank should be less than the LLOQ for the analyte or less than the level of acceptable blank contamination specified in the approved QAPP or other appropriate systematic planning document. Blanks are generally considered to be acceptable if target analyte concentrations are less than one-half the LLOQ or are less than project-specific requirements.

When new reagents or chemicals are received, the lab should monitor the blanks associated with samples for any signs of contamination. It is not necessary to test every new batch of reagents or chemicals prior to sample preparation if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents.

**12.8 LABORATORY CONTROL SAMPLES** - If the recovery does not meet criteria, see section 12.9 for marginal failures. If it is still out of control limits, then all field and QC samples in the batch must be re-analyzed.

Qualifiers must be applied to any LCS compound that does not meet these criteria and are considered out of control. The percent difference for all method target analytes must be within QC RPD limits. If not, re-analyze the duplicate(s) or prepare a new calibration curve, as necessary.

**STATE NOTE: SOUTH CAROLINA DHEC Compliance LCS:** responses must be within 70 – 130% for Method 8260 and within the limits given in Appendix F for Method 624. Qualifiers cannot be used. (If an LCS standard is above the acceptable QC criteria and all samples being reported are below the reporting limit, the data is acceptable based on a high bias with undetectable levels in the field samples. Any positive samples require reanalysis.) Failures require a batch re-analysis. For samples analyzed




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from South Carolina that are not utilized for compliance purposes, in house established acceptance limits are utilized to demonstrate controlled analyses.

## 12.9 LCS/LCSD & MS/MSD CRITERIA

### 12.9.1 Quality control criteria must be checked for the LCS and LCSD.

LCS samples that do not pass the acceptable QC criteria must be re-analyzed. LCS failures can meet marginal exceedance criteria below. Normal compound list for 8260/624.1 contains typical 90 analytes; therefore, only five analytes can be considered as marginal exceedances. If the failure persists, re-prepare and re-analyze the entire sample batch.

When a large number of analytes exist in the LCS, it is statistically possible for a few analytes to be outside of control limits. Upper and lower marginal exceedance (ME) limits are established by +/- four times the standard deviation. The number of marginal exceedance is based on the number of analytes in the LCS.

Number of allowable marginal exceedances:

>90 analytes, 5 analytes allowed in the ME limit  
 71 – 90 analytes, 4 analytes allowed in the ME limit  
 51 – 70 analytes, 3 analytes allowed in the ME limit  
 31 – 50 analytes, 2 analytes allowed in the ME limit  
 11 – 30 analytes, 1 analyte allowed in the ME limit  
 < 11 analytes, no analyte allowed in the ME limit

Marginal exceedances must be random events.

**STATE NOTE:** For South Carolina DHEC compliance samples, marginal exceedances do not apply. All outliers in QC require corrective action when possible and the data must be flagged when necessary.

**12.9.2 Method 8000D:** If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit or action level, the spike should be at or below the limit, or 1 - 5 times the background concentration (if historical data are available), whichever concentration is higher. If historical data are not available, a background sample of the same matrix from the site may be submitted for matrix spiking purposes to ensure that high concentrations of target analytes and/or interferences will not prevent calculation of recoveries. If the background sample concentration is very low or non-detect, a spike of greater than five (5) times the background concentration is still acceptable. To assess data precision with duplicate analyses, it is preferable to use a low concentration field sample to prepare a MS/MSD for organic analyses. This spiking procedure will be performed when project-specific instructions are received from the client.

If the concentration of a specific analyte in a sample is not being checked against a limit specific to that analyte, then the analyst may spike the matrix spike or MS/MSD sample(s) at the same concentration as the reference sample at 20 times the estimated LLOQ in the matrix of interest, or at a concentration near the middle of the calibration range. It is suggested that a background sample of the same matrix from the site be submitted as a sample for matrix spiking purposes.




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**NOTE:** Preparing the spiking solution from the same source as the calibration standards helps minimize additional variability due to differences between sources. Typically, spiking concentrations are near the middle of the calibration range.

To develop precision and bias data for the spiked compounds, the analyst has two choices: analyze the original sample, and an MS/MSD pair; or analyze the original sample, a duplicate sample, and one spiked sample. If samples are not expected to contain the target analytes of concern, then the laboratory may use a MS/MSD pair. If samples are expected to contain the target analytes of concern, then the laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample as an alternative to the MS/MSD pair.

The laboratory should use 70 - 130% as interim acceptance criteria for recoveries of spiked analytes, until in-house LCS limits are developed. Where in-house limits have been developed for matrix spike percent recoveries, the LCS results should be similar to or tighter than those limits, as the LCS is prepared in a clean matrix.

**12.10 MATRIX SPIKE ASSESSMENT:** If acceptance criteria are not met, perform the following corrective actions as appropriate.

- If both LCS and MS/MSD recoveries are unacceptable, then the entire batch of field and QC samples must be re-analyzed.
- If the MS/MSD is unacceptable, but the LCS is acceptable, then a potential matrix effect has been identified. Reported data must be flagged. Reasonable attempts must be made to address matrix interference.
- If analyst error appears to be the root cause of the failure (i.e., no spiked) reanalysis of the MS/MSD and parent is required if volume permits.

Acceptance criteria are that all RPD results must be within the current control limits found in the LIMS. If these conditions are not met, perform the following corrective actions as appropriate.

- Re-analyze the sample to verify a matrix effect only if analyst error appears to be the root cause.
- If the duplicate precision is still unacceptable, and LCS precision is acceptable, then a potential matrix effect has been identified.

**STATE NOTE:** South Carolina DHEC compliance analyses require that all target compounds meet the established MS/MSD criteria. No qualifiers can be applied, except in the circumstance where matrix interference is apparent.

**PROJECT SPECIFIC CRITERIA (Non-South Carolina Samples):** Acceptance criteria are that all %Recovery and/or RPD results meet project-established goals.

**12.11 SURROGATE EVALUATION:** If the surrogate recoveries are outside limits for Blank, ICV/CCV/SSCV, and LCS/LCSD, re-analysis must be performed for verification. If recoveries are still outside control limits, corrective action is necessary. All samples associated with batch or sequence needs to be re-analyzed. The surrogate recoveries for all field samples must be within established control limits. If more than two surrogate recoveries are outside limits, re-analysis must be performed for verification. If recoveries are still outside control limits, corrective action is necessary which includes qualifying




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data with J1 (outside upper limit) or J2 (outside lower limit). When surrogates fail, data is qualified with J1 (outside upper limit) or J2 (outside lower limit).

- 12.12 INTERNAL STANDARD AREA COUNT:** If the area response for any of the internal standards changes by a factor of two (-50% to +100%) as per section 9.2.4, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. In the event the internal standard area counts fail these criteria, the following corrective actions should be considered.
- Check to be sure there are no errors in the internal standards preparation or addition. Also check instrument performance.
  - If any internal standard fails high (> +100%), sample must be re-analyzed with possible dilution. If recoveries are still outside control limits, corrective action is necessary which includes qualifying compounds with associated internal standard with J9 (IS high, data is likely to show low bias).
  - If more than two internal standard fails low « -50%), sample must be re-analyzed. If recoveries are still outside control limits, corrective action is necessary which includes qualifying compounds with associated internal standard with J8 (IS low, data is likely to show high bias).
  - If one or two internal standard criteria fails low « -50%), corrective action is necessary which includes qualifying compounds with associated internal standard with J8 (IS low, data is likely to show high bias).
- 12.13 CALIBRATION RANGE:** The analyst must verify all reported results are derived from analytical results that are below the highest standard of the initial calibration curve and above the low standard. Values reported below the low standard are to be reported as estimated values (J values). For samples that exceed the calibration curve, dilute and analyze an appropriate sample aliquot.
- 12.14 SECOND SOURCE (SSCV) or Initial Calibration Verification (ICV):** If the SSCV does not meet acceptance criteria, it can be reanalyzed once. If the failure persists, a new initial calibration curve must be prepared and analyzed.
- 12.15** Data that does not meet acceptable QC criteria may be acceptable for use in certain circumstances.
- 12.15.1** If a method blank contains an amount of target analyte, but all samples are non-detected or the samples contain analyte at a level that is greater than 10 times the level present in the blank, the data is reported with the appropriate "B" flag.
- 12.15.1.1** When comparing analyte contamination in the blank to possible analyte contamination in the field sample, utilize the sample concentration without applying the multiplier value unless the same multiplier has been applied to the quantitation of the target analytes in the blank.
- 12.15.2** If the sample surrogate is above the acceptable QC range, but the samples are non-detected for all target analytes, flag the sample with a J1 and report. If the surrogate is below the acceptable QC range, re-analyze the sample if the surrogate still fails, re-extract and re-analyze or flag data.
- 12.15.3** Matrix spike failures must be flagged with "J5" (high) or "J6" (low), when QC limits are exceeded. If there is an RPD failure, the data is flagged with a "J3".
- 12.16** Quantitation and manual integration of all QC samples and client samples must follow the procedures outlined in ENV-SOP-CORQ-0006, *Manual Integration*. "Before" and "After"






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quantitation reports must be printed in order to verify that any manual integration is performed properly and consistently.

**12.17** Data must be checked to ascertain if it conforms to accepted practices. All sample analytical results used for final data reporting must be between the low standard and the high calibration standard. Values falling above the high standard must be diluted and re-analyzed.

**12.17.1** Site specific DQO's may require values below the reporting limit but above the method detection limit be reported as "UJ" or estimated value. The reporting limit is the concentration of the lowest standard used in the calibration.

**12.17.2** All tentatively identified compounds (TICs) are reported with a "J" qualifier for estimated value and an "N" for presumptive evidence of material.

**12.18** For samples analyzed per the requirements of Method 8000D, reported concentrations of target analytes between the MDL and the LLOQ must be qualified as estimated.

### 13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

**13.1** The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner. See *ENV-SOP-MTJL-0051, Waste Management Plan*.

**13.2** See *ENV-SOP-MTJL-0046, Environmental Sustainability & Pollution Prevention*.

### 14.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP *ENV-SOP-CORQ-0011 Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

**14.1** Adjustments to the concentrations of standards/spiking solutions, standards providers, and quality control are subject to change to better meet client/project/regulatory needs or to improve laboratory method performance.

**14.2** Modifications to this method are noted in the body of the text as state notes. Compliance analyses performed in conjunction with specific state requirements must be performed as noted within the specific state(s) note listed.

**14.3** Heated purge may be used for all samples regardless of matrix or method.




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## 15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

## 16.0 Attachments

- 16.1 Appendix A: Target Analyte List and Routine LOQ
- 16.2 Appendix B: QC Summary
- 16.3 Appendix C: Characteristic Masses (m/z) for Purgeable Organic Compounds as printed from SW-846 Method 8260B Table 5
- 16.4 Appendix D: Potential Compounds to be Analyzed by this Procedure
- 16.5 Appendix E: The SIM Mode
- 16.6 Appendix F: EPA 624.1 CCC Criteria
- 16.7 Appendix G: EPA 8260C Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification
- 16.8 Appendix H: EPA 8260D (Table 4) Guidance Response Factors Criteria From EPA Contract Laboratory Program (Min RF)
- 16.9 Appendix I: Laboratory Control Standard and Matrix Spike Typically Spiked Compounds
- 16.10 Appendix J: DoD Requirements

## 17.0 REFERENCES

- 17.1 *Determinative Chromatographic Separations*, SW-846 Method 8000B, Revision 2, December 1996.
- 17.2 *Determinative Chromatographic Separations*, SW-846 Method 8000C, Revision 3, March 2003.
- 17.3 *Determinative Chromatographic Separations*, SW-846 Method 8000D, Revision 4, July 2014.
- 17.4 *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, SW-846 Method 8260B, Revision 2, December 1996.
- 17.5 *Purgeables*, 40 CFR 136, EPA Method 624/
- 17.6 *Volatile Organic Compounds by the Purge and Trap Capillary-Column Gas Chromatographic/Mass Spectrometric Method*, SM 6200B, 20<sup>th</sup> edition.
- 17.7 Policy Document, NELAC Standard, Chapter 2: Proficiency Testing Program Standard and the relevant section of NELAC Standard Chapter 5 National Environmental Laboratory Accreditation Conference
- 17.8 *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, SW-846 Method 8260C, Revision 3, August 2006.

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- 17.9 *Volatile Organic Compounds by the Purge and Trap Capillary-Column Gas Chromatographic/Mass Spectrometric Method, SM 6200B-2011.*
- 17.10 *Purgeables by GC/MS, EPA Method 624.1, Federal Register, Volume 82, Number 165, August 28, 2017.*
- 17.11 40 Code of Federal Regulations §136.6(b)(4)(j).
- 17.12 *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, SW-846 Method 8260D, Revision 4, June 2018.*

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**18.0 Revision history**

This Version:

Section	Description of Change
All	Complete SOP reformat.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MTJL-0100	Volatile Organic Compounds by GC/MS (EPA 8260B, 8260C, 624, 624.1 and SM 6200B)	04

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**Appendix A: Target Analyte List and Routine LOQ**

Compound	Water		Low Soil		High Soil	
	RL	Units	RL*	Units	RL	Units
1,1,1,2-Tetrachloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1,1-Trichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1,2,2-Tetrachloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1,2-Trichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1-Dichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1-Dichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,1-Dichloropropene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2,3-Trichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2,3-Trichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2,4-Trichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2,4-Trimethylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dibromo-3-Chloropropane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
1,2-Dibromoethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dichloroethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,2-Dichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,3,5-Trimethylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,3-Butadiene	0.002	mg/L	0.002	mg/Kg	0.125	mg/Kg
1,3-Dichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,3-Dichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
1,4-Dichlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
2,2,4-Trimethyl Pentane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
2,2-Dichloropropane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
2-Butanone (MEK)	0.01	mg/L	0.01	mg/Kg	0.5	mg/Kg
2-Chloroethyl vinyl ether	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
2-Chlorotoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
4-Chlorotoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
4-Ethyltoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
4-Methyl-2-pentanone (MIBK)	0.01	mg/L	0.01	mg/Kg	0.5	mg/Kg
Acetone	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
Acrolein	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
Acrylonitrile	0.01	mg/L	0.01	mg/Kg	0.5	mg/Kg
Benzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromodichloromethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromoform	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Bromomethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Carbon tetrachloride	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Chlorobenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Chlorodibromomethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Chloroethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg

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Compound	Water		Low Soil		High Soil	
	RL	Units	RL*	Units	RL	Units
Chloroform	0.005	mg/L	0.005	mg/Kg	0.1	mg/Kg
Chloromethane	0.0025	mg/L	0.0025	mg/Kg	0.05	mg/Kg
cis-1,2-Dichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
cis-1,3-Dichloropropene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Dibromomethane	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Dichlorodifluoromethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Dicyclopentadiene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Di-isopropyl ether	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Ethylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Hexachlorobutadiene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Hexane	0.01	mg/L	0.01	mg/Kg	0.5	mg/Kg
Isopropylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Methyl tert-butyl ether	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Methylene Chloride	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Naphthalene	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
n-Butylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
n-Propylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
p-Isopropyltoluene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Propene	0.0025	mg/L	0.0025	mg/Kg	0.125	mg/Kg
sec-Butylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Styrene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
tert-Butylbenzene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Tetrachloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Toluene	0.001	mg/L	0.001	mg/Kg	0.25	mg/Kg
trans-1,2-Dichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
trans-1,3-Dichloropropene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Trichloroethene	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Trichlorofluoromethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Vinyl chloride	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Xylenes, Total	0.003	mg/L	0.003	mg/Kg	0.15	mg/Kg
<b>Additional Compounds</b>						
1,4-Dioxane+	0.1	mg/L	0.1	mg/Kg	5	mg/Kg
2-Butanol	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
2-Hexanone	0.01	mg/L	0.01	mg/Kg	0.5	mg/Kg
2-Propanol	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Acetonitrile	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
Allyl Chloride	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Bromoethane	0.01	mg/L	0.001	mg/Kg	0.05	mg/Kg
Carbon Disulfide	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Chloroprene	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
Cyclohexanone	0.01	mg/L	0.01	mg/Kg	0.5	mg/Kg
Dichlorofluoromethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Di-isopropyl ether	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg

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Compound	Water		Low Soil		High Soil	
	RL	Units	RL*	Units	RL	Units
Ethanol	0.1	mg/L	0.1	mg/Kg	2.5	mg/Kg
Ethyl methacrylate	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Ethyl tert-butyl ether	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Iodomethane	0.01	mg/L	0.01	mg/Kg	0.5	mg/Kg
Isobutanol	0.1	mg/L	0.1	mg/Kg	5	mg/Kg
Isobutanol	0.1	mg/L	0.001	mg/Kg	0.05	mg/Kg
Methacrylonitrile	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
Methyl Methacrylate	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Pentachloroethane	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Propionitrile	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
Tert Amyl Alcohol	0.05	mg/L	0.05	mg/Kg	0.25	mg/Kg
Tert Butyl Ethyl Alcohol	0.1	mg/L	0.1	mg/Kg	5	mg/Kg
Tert-Amyl Methyl Ether	0.001	mg/L	0.001	mg/Kg	0.05	mg/Kg
Tert-Butyl Alcohol	0.05	mg/L	0.05	mg/Kg	2.5	mg/Kg
Tert-Butyl Formate	0.02	mg/L	0.02	mg/Kg	1	mg/Kg
Tetrahydrofuran	0.005	mg/L	0.005	mg/Kg	0.25	mg/Kg
Trans-1,4-Dichloro-2-butene	0.0025	mg/L	0.0025	mg/Kg	0.125	mg/Kg
Vinyl Acetate	0.01	mg/L	0.01	mg/Kg	2.5	mg/Kg

RLs are based on a 5mL purge volume

Low Soil - Using a 5g soil sample to 5mL water – See Method 5035 (ENV-SOP-MTJL-0129) Section 8.2.4.1

High Soil – Using 200uL extract from 10g soil sample to 10mL methanol; see Method 5035 (ENV-SOP-MTJL-0129) Sect. 8.3.1.2

+ 1,4-Dioxane has a RL of .002 when run using the SIM mode.

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**Appendix B: QC Summary**

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
ICAL	At instrument set up, after CCV failure	Must meet one of curve fit options presented in Section 9.0.  For any curve fit other than Average RF (RSD), curve must also pass RSE test at the low and midpoint calibration standard.	Identify and correct source of problem, repeat	None. Do not proceed with analysis
ICV	After Each ICAL	All analytes must be within $\pm 30\%$ recovery (%R) of the true value with the exception of poor performers which must be within in-house limits.	Identify source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
RT Window Position (Daily)	Once per ICAL and at the beginning of the analytical window.	Position is set using the mid-point of the ICAL on the day ICAL is performed; otherwise mid-point of CCV is used	NA	NA
RT Window Study	At method set-up and after major instrument maintenance	Window is $\pm 3$ times the standard deviation among three data points across 72 hours.	NA	NA
CCV	Daily, before sample analysis, after every X, and at end of analytical window.	Opening CCV: All analytes within $\pm 20\%$ %D Ending CCV: All analytes within $\pm 50\%$ %D	See Section 12 for required corrective actions based on circumstance.	Qualify analytes with CCV out of criteria.
Internal Standards	Every field sample, standard and QC sample	Must meet criteria specified in Section 9.0.	Troubleshoot instrument performance. Reanalyze samples.	Qualify outages and explain in case narrative.
Surrogate	All field and QC samples.	Routine limits are presented in the LIMS	Correct problem, then reprep and reanalyze all failed samples for all surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference is present, reanalysis may not be necessary, but the client must be notified prior to reporting data and the failures must be discussed in the case narrative.	Qualify all associated analytes if acceptance criteria are not met and explain in the case narrative.

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Method Blank	One per preparatory batch.	No analytes detected above the MDL for any of the method target analytes	Correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.
LCS	One per preparatory batch.	Routine LCS control limits are presented in the LIMS	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available	Qualify all associated analytes if acceptance criteria are not met and explain in the case narrative.
MS/MSD	One per preparatory batch.	All RPD results must be within the indicated control limits found in the LIMS.	Examine the project specific requirements. Contact the client as to additional measures to be taken.	Qualify all associated analytes if acceptance criteria are not met and explain in the case narrative.
Trip Blank	1 per cooler	No analytes detected above the MDL for any of the method target analytes	Examine the project specific requirements. Contact the client as to additional measures to be taken.	NA
Tune Standard	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB from method.	Retune instrument and verify.	Flagging is not appropriate.
Performance Check	Prior to ICAL	Must meet the minimum, average RF criteria for each analyte according to Section 9.2.3.2.4-9.2.3.2.7	Identify source of problem and troubleshoot instrument performance. Repeat ICAL.	None. Do not proceed with analysis

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**Appendix C: Characteristic Masses (m/z) for Purgeable Organic Compounds as printed from SW-846 Method 8260B Table 5**

Compound	Primary Characteristic	Secondary Characteristic
	Ion	Ion(s)
Acetone	58	43
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl alcohol	57	58, 39
Allyl chloride	76	41, 39, 78
Benzene	78	-
Benzyl chloride	91	126, 65, 128
Bromoacetone	136	43, 138, 93, 95
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
1,3-Butadiene	39	54
iso-Butanol	74	43
n-Butanol	56	41
2-Butanone	72	43
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chloral hydrate	82	44, 84, 86, 111
Chloroacetonitrile	48	75
Chlorobenzene	112	77, 114
1-Chlorobutane	56	49
Chlorodibromomethane	129	208, 206
Chloroethane	64 (49*)	66 (51*)
2-Chloroethanol	49	44, 43, 51, 80
Bis(2-chloroethyl) sulfide	109	111, 158, 160
2-Chloroethyl vinyl ether	63	65, 106
Chloroform	83	85
Chloromethane	50 (49*)	52 (51*)
Chloroprene	53	88, 90, 51
3-Chloropropionitrile	54	49, 89, 91
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Dicyclopentadiene	66	132
1,2-Dibromo-3-chloropropane	157**	75, 155
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,2-Dichlorobenzene-d4	152	115, 150
1,3-Dichlorobenzene	146	111, 148

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Compound	Primary Characteristic	Secondary Characteristic
	Ion	Ion(s)
1,4-Dichlorobenzene	146	111, 148
cis-1,4-Dichloro-2-butene	75	53, 77, 124, 89
trans-1,4-Dichloro-2-butene	53	88, 75
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43, 81, 49
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39
1,2,3,4-Diepoxybutane	55	57, 56
Diethyl ether	74	45, 59
1,4-Dioxane	88	58, 43, 57
Epichlorohydrin	57	49, 62, 51
Ethanol	31	45, 27, 46
Ethyl acetate	88	43, 45, 61
Ethylbenzene	91	106
Ethylene oxide	44	43, 42
Ethyl methacrylate	69	41, 99, 86, 114
4-Ethyltoluene	105	120
Hexachlorobutadiene	225	223, 227
Hexachloroethane	201	166, 199, 203
Hexane	57	86, 56
2-Hexanone	43	58, 57, 100
2-Hydroxypropionitrile	44	43, 42, 53
Iodomethane	142	127, 141
Isobutyl alcohol	43	41, 42, 74
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Malononitrile	66	39, 65, 38
Methacrylonitrile	41	67, 39, 52, 66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86, 49
Methyl ethyl ketone	72	43
Methyl iodide	142	127, 141
Methyl methacrylate	69	41, 100, 39
4-Methyl-2-pentanone	100	43, 58, 85
Naphthalene	128	-
Nitrobenzene	123	51, 77
2-Nitropropane	46	-
2-Picoline	93	66, 92, 78
Pentachloroethane	167	130, 132, 165, 169
Propargyl alcohol	55	39, 38, 53

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Compound	Primary Characteristic	Secondary Characteristic
	Ion	Ion(s)
Propene	41	39, 42
Propiolactone	42	43, 44
Propionitrile (ethyl cyanide)	54	52, 55, 40
n-Propylamine	59	41, 39
n-Propylbenzene	91	120
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzen	180	182, 145
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tert-butyl formate	59	57, 41
Tetrachloroethene	164	129, 131, 166
Toluene	92	91
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
<b>Internal Standards/Surrogates:</b>		
1,4-Difluorobenzene	114	63
1,4-Dichlorobenzene-d4	152	115, 150
1,1,2-Trichloroethane-d3	100	
4-Bromofluorobenzene	95	174, 176
Chloroform-d1	84	
Dibromofluoromethane	113	
4-Bromofluorobenzene	95	174, 176
Chloroform-d1	84	
Dibromofluoromethane	113	
Dichloroethane-d4	102	
Toluene-d8	98	
Pentafluorobenzene	168	
Fluorobenzene	96	77

\* Characteristic ion for an ion trap mass spectrometer (to be used when ion-molecule reactions are observed).

\*\* Primary ion modified due to coelution.

**STANDARD OPERATING PROCEDURE**

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**Appendix D: Potential Compounds to be Analyzed by this Procedure**

Acetone	Dicyclopentadiene
Acetonitrile	1,4-Dioxane
Acrolein	Epichlorohydrin
Acrylonitrile	Ethanol
Allyl alcohol	Ethylbenzene
Allyl chloride	Ethylene oxide
Benzene	Ethyl methacrylate
Benzyl chloride	n-Hexane
Bromoacetone	2-Hexanone
Bromochloromethane (I.S.)	2-Hydroxypropionitrile
Bromodichloromethane	Iodomethane
4-Bromofluorobenzene (Surr.)	Isobutylalcohol
Bromoform	Malononitrile
Bromomethane	Methacrylonitrile
2-Butanone	Methylene chloride
Carbon disulfide	Methyl iodide
Carbon tetrachloride	Methyl methacrylate
Chloral hydrate	4-methyl-2-pentanone
Chlorobenzene	Pentachloroethane
Chlorobenzene d-5 (I.S.)	2-Picoline
Chlorodibromomethane	Propargyl alcohol
2-Propanol	Propene
Chloroethane	B-propiolactone
2-Chloroethanol	Propionitrile
bis-(2-Chloroethyl) sulfide	n-Propylamine
2-Chloroethyl vinyl ether	Pyridine
Chloroform	Styrene
Chloromethane	1,1,1,2-Tetrachloroethane
Chloroprene	1,1,2,2-Tetrachloroethane
3-Chloropropionitrile	Tetrachloroethene
1,2- Dibromo-3-chloropropane	Toluene
1,2-Dibromoethane	1,3-Butadiene
Dibromomethane	1,1,1-Trichloroethane
1,4-Dichloro-2-butene	1,1,2-Trichloroethane
dichlorodifluoromethane	Trichloroethene
1,1-Dichloroethane	Trichlorofluoromethane
1,2-Dichloroethane	1,2,3-Trichloropropane
1,2-Dichloroethane d-4 (surr.)	Vinyl acetate
1,1-Dichloroethene	Vinyl chloride
Trans-1,2-dichloroethene	Xylene (total)
Cis-1,2-dichloroethene	1,2,3,4-Diepoxybutane
1,2-dichloropropane	4-Ethyltoluene



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**Appendix E: The SIM Mode:**

An alternate way of running compounds to achieve lower detection limits is by way of the Single Ion Monitoring (SIM) method. The SIM method allows the Mass spec to dwell on certain ions rather than scanning the full range of masses from 35 to 300. This process allows for much lower detection limit of desired compounds. This method is only for the detection of known compounds while a TIC cannot be performed while running the SIM method. Currently 1,4-Dioxane is the only compound that is analyzed using the SIM method in the volatiles laboratory.

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**Appendix F: EPA 624.1 CCC Criteria:**

Table 7 – LCS (Q), DOC (s and $\bar{X}$ ), and MS/MSD (P and RPD) Acceptance Criteria <sup>1</sup>					
Analyte	Range for Q (%)	Limit for s (%)	Range for $\bar{X}$ (%)	Range for P <sub>1</sub> P <sub>2</sub> (%)	Limit for RPD
Acrolein	60-140	30	50-150	40-160	60
Acrylonitrile	60-140	30	50-150	40-160	60
Benzene	65-135	33	75-125	37-151	61
Benzene-d <sub>6</sub>					
Bromodichloromethane	65-135	34	50-140	35-155	56
Bromoform	70-130	25	57-156	45-169	42
Bromomethane	15-185	90	D-206	D-242	61
2-Butanone-d <sub>6</sub>					
Carbon tetrachloride	70-130	26	65-125	70-140	41
Chlorobenzene	65-135	29	82-137	37-160	53
Chloroethane	40-160	47	42-202	14-230	78
Chloroethane-d <sub>5</sub>					
2-Chloroethylvinyl ether	D-225	130	D-252	D-305	71
Chloroform	70-135	32	68-121	51-138	54
Chloroform- <sup>13</sup> C					
Chloromethane	D-205	472	D-230	D-273	60
Dibromochloromethane	70-135	30	69-133	53-149	50
1,2-Dichlorobenzene	65-135	31	59-174	18-190	57
1,2-Dichlorobenzene-d <sub>4</sub>					
1,3-Dichlorobenzene	70-130	24	75-144	59-156	43
1,4-Dichlorobenzene	65-135	31	59-174	18-190	57
1,1-Dichloroethane	70-130	24	71-143	59-155	40
1,2-Dichloroethane	70-130	29	72-137	49-155	49
1,2-Dichloroethane-d <sub>4</sub>					
1,1-Dichloroethene	50-150	40	19-212	D-234	32
1,1-Dichloroethene-d <sub>2</sub>					
trans-1,2-Dichloroethene	70-130	27	68-143	54-156	45
1,2-Dichloropropane	35-165	69	19-181	D-210	55
1,2-Dichloropropane-d <sub>6</sub>					
cis-1,3-Dichloropropene	25-175	79	5-195	D-227	58
trans-1,3-Dichloropropene	50-150	52	38-162	17-183	86
trans-1,3-Dichloropropene-d <sub>4</sub>					
Ethyl benzene	60-140	34	75-134	37-162	63
2-Hexanone-d <sub>7</sub>					
Methylene chloride	60-140	192	D-205	D-221	28
1,1,2,2-Tetrachloroethane	60-140	36	68-136	46-157	61
1,1,2,2-Tetrachloroethane-d <sub>2</sub>					
Tetrachloroethene	70-130	23	65-133	64-148	39
Toluene	70-130	22	75-134	47-150	41
Toluene-d <sub>8</sub>					
1,1,1-Trichloroethane	70-130	21	69-151	52-162	36
1,1,2-Trichloroethane	70-130	27	75-136	52-150	45
Trichloroethene	65-135	29	75-138	70-157	48
Trichlorofluoromethane	50-150	50	45-158	17-181	84
Vinyl chloride	5-195	100	D-218	D-251	66
Vinyl chloride-d <sub>3</sub>					

<sup>1</sup> Criteria were calculated using an LCS concentration of 20 µg/L.

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**Appendix G: EPA 8260C Minimum Relative Response Factor Criteria for Initial and Continuing Calibration Verification:**

Volatile Compound	Minimum Response Factor (RF)
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone	0.100
Carbon disulfide	0.100
Methyl Acetate	0.100
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone	0.100
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100

Volatile Compound	Minimum Response Factor (RF)
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone	0.100
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone	0.100
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
meta-/para-Xylene	0.100
ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200



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**Appendix H: EPA 8260D (Table 4) Guidance Response Factors Criteria From EPA Contract Laboratory Program (Min RF):**

Analyte	RF
Acetone	0.01
Benzene	0.2
Bromochloromethane	0.1
Bromodichloromethane	0.3
Bromofom	0.1
Bromomethane	0.01
2-Butanone	0.01
Carbon disulfide	0.1
Carbon tetrachloride	0.1
Chlorobenzene	0.4
Chloroethane	0.01
Chlorofom	0.3
Chloromethane	0.01
Cyclohexane	0.01
Dibromochloromethane	0.2
1,2-Dibromo-3-chloropropane	0.01
1,2-Dibromoethane (EDB, Ethylene dibromide)	0.2
1,2-Dichlorobenzene	0.6
1,3-Dichlorobenzene	0.5
1,4-Dichlorobenzene	0.6
Dichlorodifluoromethane	0.01
1,1-Dichloroethane	0.3
1,2-Dichloroethane	0.07
1,1-Dichloroethene (Vinylidene chloride)	0.06
cis-1,2-Dichloroethene	0.2
trans-1,2-Dichloroethene	0.1
1,2-Dichloropropane	0.2
cis-1,3-Dichloropropene	0.3
trans-1,3-Dichloropropene	0.3
Ethylbenzene	0.4
2-Hexanone	0.01
Isopropylbenzene	0.4
Methyl acetate	0.01
4-Methyl-2-pentanone	0.03
Methyl tert-butyl ether (MTBE)	0.1
Methylcyclohexane	0.05
Methylene chloride	0.01
Styrene	0.2
1,1,2,2-Tetrachloroethane	0.2
Tetrachloroethene	0.1
Toluene	0.3
1,2,3-Trichlorobenzene	0.4
1,2,4-Trichlorobenzene	0.4
1,1,1-Trichloroethane	0.05
1,1,2-Trichloroethane	0.2
1,1,2-Trichlorotrifluoroethane	0.05
Trichloroethene (Trichloroethylene)	0.2
Trichlorofluoromethane	0.01
Vinyl chloride	0.01
m,p-Xylene	0.2
o-Xylene	0.2

Values in this table are referenced from the CLP Statement of Work SOM 02.4. These response factors are provided as guidance only and are not intended to be a requirement. See Appendix B for additional information.




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**Appendix I: Laboratory Control Standard and Matrix Spike Typically Spiked Compounds****STANDARD ANALYTE LIST**

1,1,1,2-TETRACHLOROETHANE	CHLORODIBROMOMETHANE
1,1,1-TRICHLOROETHANE	CHLOROETHANE
1,1,2,2-TETRACHLOROETHANE	CHLOROFORM
1,1,2-TRICHLOROETHANE	CHLOROMETHANE
1,1,2-TRICHLOROTRIFLUOROETHANE	CIS-1,2-DICHLOROETHENE
1,1-DICHLOROETHANE	CIS-1,3-DICHLOROPROPENE
1,1-DICHLOROETHENE	DIBROMOMETHANE
1,1-DICHLOROPROPENE	DICHLORODIFLUOROMETHANE
1,2,3-TRICHLOROBENZENE	DICHLOROFLUOROMETHANE
1,2,3-TRICHLOROPROPANE	DICYCLOPENTADIENE
1,2,3-TRIMETHYLBENZENE	DI-ISOPROPYL ETHER
1,2,4-TRICHLOROBENZENE	ETHYL ETHER
1,2,4-TRIMETHYLBENZENE	ETHYLBENZENE
1,2-DIBROMO-3-CHLOROPROPANE	HEXACHLORO-1,3-BUTADIENE
1,2-DIBROMOETHANE	IODOMETHANE
1,2-DICHLOROBENZENE	ISOPROPYLBENZENE
1,2-DICHLOROETHANE	M&P-XYLENE
1,2-DICHLOROPROPANE	METHYL TERT-BUTYL ETHER
1,3,5-TRICHLOROBENZENE	METHYLENE CHLORIDE
1,3,5-TRIMETHYLBENZENE	NAPHTHALENE
1,3-BUTADIENE	N-BUTYLBENZENE
1,3-DICHLOROBENZENE	N-HEXANE
1,3-DICHLOROPROPANE	N-PROPYLBENZENE
1,4-DICHLOROBENZENE	O-XYLENE
1-METHYLNAPHTHALENE	P-ISOPROPYLTOLUENE
2,2,4-TRIMETHYLPENTANE	PROPENE
2,2-DICHLOROPROPANE	SEC-BUTYLBENZENE
2-BUTANONE (MEK)	STYRENE
2-CHLOROETHYL VINYL ETHER	TERT-BUTYLBENZENE
2-CHLOROTOLUENE	TETRACHLOROETHENE
2-HEXANONE	TETRAHYDROFURAN
2-METHYLNAPHTHALENE	TOLUENE
4-CHLOROTOLUENE	TPH (GC/MS) LOW FRACTION
4-ETHYLTOLUENE	TRANS-1,2-DICHLOROETHENE
4-METHYL-2-PENTANONE (MIBK)	TRANS-1,3-DICHLOROPROPENE
ACETONE	TRANS-1,4-DICHLORO-2-BUTENE
ACROLEIN*ACRYLONITRILE*	TRICHLOROETHENE

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**STANDARD ANALYTE LIST**

ACRYLONITRILE\* BENZENE

BENZENE BROMOBENZENE

BROMOBENZENE

BROMOCHLOROMETHANE

BROMODICHLOROMETHANE

BROMOFORM

BROMOMETHANE

CARBON DISULFIDE

CARBON TETRACHLORIDE

CHLOROBENZENE

TRICHLOROFLUOROMETHANE

VINYL ACETATE

VINYL BROMIDE

VINYL CHLORIDE

XYLENES, TOTAL

**SURROGATE LIMITS**

4-BROMOFLUOROBENZENE

2,2-DICHLOROETHANE-D4

TOLUENE-D8

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**Appendix J: DoD Requirements****1.0 Equipment/Instrument Maintenance**

Instrument maintenance must be performed routinely to optimize instrument performance and improve chromatography. Commonly performed maintenance includes baking traps and columns, changing injection port liners, changing pump oil, etc. A new calibration curve must be analyzed following any major maintenance performed on the analytical system.

**2.0 Computer Hardware and Software**

Software name and version: HP Chemstation G1701CA Version C.00.00 or equivalent

**3.0 Troubleshooting**

<b>Table 1. GCMS Troubleshooting Guide</b>		
<b>Problem</b>	<b>Cause</b>	<b>Treatment</b>
Peaks broaden and tail	Poor column installation causing dead volume in the injector	Reinstall column in injector. Check seal at ferrule. Check insertion depth. Ensure a good column cut.
	Solvent flashing in hot injector	Reduce injection speed on hot injectors and if possible, reduce injector temperature
	Injector not being purged properly after splitless injection	For splitless injection, the vent flow should be 70 ml/min, and the injector should be switched to the split mode 0.5_1.5 min after injection.
Tailing sample peaks for active components	Active sites in the injector insert or liner	Change or clean the injector insert
	Active sites or degraded phase in column	Remove the front 15 cm of the column and reinstall. If retention times are changing or cutting the column does not help, replace the column.
	Injector not hot enough for higher boiling compounds	Increase the injector temperature and lower the injection speed. Check that the graphite ferrule is free of cracks and the septum support is tight.
Low response and tailing of high boiling point compounds	Injector is not hot enough to vaporize high boilers	Increase injector temperature
	Interface/ion source not getting to adequate temperature	Change the manifold heater
Leading sample peaks	Column overload due to excess amount of component injected	Dilute the sample or do split injection
	Degradation of stationary phase	Change the column
	Carrier gas velocity too low	Increase carrier gas flow rate
Poor chromatographic resolution	Column temperature or program not optimized	Modify method by changing temperature ramp segment slopes

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<b>Table 1. GCMS Troubleshooting Guide</b>		
<b>Problem</b>	<b>Cause</b>	<b>Treatment</b>
	Carrier gas flow rate not optimized	Decrease carrier gas linear velocity
	Stationary phase has degraded	Replace the column
Peak splitting, especially low boilers	Sample is flashing in the injector simulating two injections	Lower injector temperature
Retention times shift in chromatogram	Unstable carrier gas flow controller/regulator	Check pneumatics for leaks. Replace flow controller/ regulator if necessary.
	Column contamination or degradation	Condition or replace column
	Leaks at septum or column to injector connection	Replace septum regularly and check that the septum nut and the capillary column nut are tight
Cannot reach operating vacuum	Analyzer contaminated by diffusion pump oil	Shut down and clean mass spec
	Major air leak around column fitting into interface	Replace column ferrule and reseal compression fitting
No tune peaks	Cal gas valve not open	Open cal gas valve
	PFTBA solenoid valve stuck open. All PFTBA has evaporated.	Have solenoid replaced. Put fresh PFTBA in the cal gas vial.
Analysis sensitivity has decreased	Background has increased	Check column bleed, septum bleed, pump oil, and ion source contamination
	Detector needs replacement	Replace detector
	Defective syringe	Try a new or proven syringe
	"Blown" septum or other massive leaks at the inlet or with carrier gas flow. Poor peak shapes usually result from bad leaks.	Find and fix leaks and adjust gas flow.
	Purge flow or split ratio too high	Adjust gas flow rates

**4.0 Other Requirements**

- 4.1 All hardcopy laboratory notebooks must be reviewed by the Supervisor, or their designee, on a monthly basis.
- 4.2 If not self-explanatory (e.g., a typo or transposed number), corrections to technical and quality records shall also include a justification for the change.
- 4.3 A storage blank must be stored with all volatile organic samples, regardless of suspected concentration levels.
- 4.4 A person performing a manual integration must sign and date each manually integrated chromatogram and record the rationale for performing manual integration. Electronic signatures are acceptable.

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- 4.5 The results of calibration and verification of support equipment must be within the specifications required of the application for which this equipment is used, or the equipment must be removed from service until repaired. Calibration and verification records, including those of established correction factors, must be maintained. In the absence of method-specific requirements, the minimum requirements are as follows:

<b>Performance Check</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>
Balance calibration check [Using two standard weights that bracket the expected mass]	Daily prior to use	Top-loading balance: $\pm 2\%$ or $\pm 0.02\text{g}$ , whichever is greater Analytical balance: $\pm 0.1\%$ or $\pm 0.5\text{ mg}$ , whichever is greater
Verification of standard mass [Using weights traceable to the International System of Units (SI) through a NMI]	Every 5 years	Certificate of Calibration from ISO/IEC 17025 accredited calibration laboratory
Monitoring of refrigerator/freezer temperatures	Daily (i.e., 7 days per week) [use MIN/MAX thermometers or data loggers equipped with notification of out of control event capabilities if personnel not available to record daily]	Refrigerators: $0^{\circ}\text{C}$ to $6^{\circ}\text{C}$ Freezers: $\leq -10^{\circ}\text{C}$
Thermometer verification check [Using a thermometer traceable to the SI through an NMI] [Performed at two temperatures that bracket the target temperature(s). Assume linearity between the two bracketing temperatures.] [If only a single temperature is used, at the temperature of use]	Liquid in glass: Before first use and annually  Electronic: Before first use and quarterly	Apply correction factors or replace thermometer
Volumetric labware	Class B: By lot before first use  Class A and B: Upon evidence of deterioration	Bias: Mean within $\pm 2\%$ of nominal volume Precision: RSD $\leq 1\%$ of nominal volume (based on 10 replicate measurements)
Non-volumetric labware [Applicable only when used for measuring initial sample volume and final extract/ digestates volume]	By lot before first use or upon evidence of deterioration	Bias: Mean within $\pm 3\%$ of nominal volume Precision: RSD $\leq 3\%$ of nominal volume (based on 10 replicate measurements)
Mechanical volumetric pipette	Quarterly	Bias: Mean within $\pm 2\%$ of nominal volume Precision: RSD $\leq 1\%$ of nominal volume (based on minimum of 3 replicate measurements) [Note: for variable volume pipettes, the nominal volume is the volume of use]

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<b>Performance Check</b>	<b>Frequency</b>	<b>Acceptance Criteria</b>
Glass microliter syringe	Upon receipt and upon evidence of deterioration	General Certificate of Bias & Precision upon receipt Replace if deterioration is evident
Drying oven temperature check	Daily prior to and after use	Within $\pm 5\%$ of set temperature
Water purification system	Daily prior to use	See method blank criteria given in Section 4.20 of this addendum

- 4.6 The expiration date of the prepared standard shall not exceed the expiration date of the primary standard. All containers must bear a preparation date.
- 4.7 To avoid preparing non-representative samples, the laboratory shall not “target” within a relatively small mass range (e.g.,  $1.00 \pm 0.01\text{g}$ ) because such targeting will produce non-representative subsamples if the sample has high heterogeneity. The laboratory shall not manipulate the sample material so the sample aliquot weighs exactly  $1.00\text{g} \pm 0.01\text{g}$ , as an example.
- 4.8 In the absence of project-specific requirements, the minimum standard data qualifiers to be used are:
- U Analyte was not detected and is reported as less than the LOD or as defined by the customer. The LOD has been adjusted for any dilution or concentration of the sample.
  - J The reported result is an estimated value (e.g., matrix interference was observed, or the analyte was detected at a concentration outside the quantitation range).
  - B Blank contamination. The recorded result is associated with a contaminated blank.
  - N Non-target analyte. The analyte is a tentatively identified compound using mass spectrometry or any non-customer requested compounds that are tentatively identified.
  - Q One or more quality control criteria failed (e.g., LCS recovery, surrogate spike recovery, or CCV recovery).
- Additional data qualifiers may be used, or different letters or symbols to denote the qualifiers listed above, as long as they are appropriately defined and their use is consistent with project-specific requirements (e.g., QSM 5.0, the contract, and project-planning documents).
- 4.9 If the time of the sample collection is not provided, assume the most conservative time of day. For the purpose of batch processing, the start and stop dates and times of the batch preparation shall be recorded.
- 4.10 Each preparation method listed on the scope of accreditation must have quarterly LOD/LOQ verifications. However, not all possible combinations of preparation and cleanup techniques are required to have LOD/LOQ verifications. If LOD/LOQ verifications are not performed on all combinations, the laboratory must base the

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LOD/LOQ verifications on the worst-case basis (preparation method with all applicable cleanup steps).

- 4.11 After each MDL determination, the laboratory must establish the LOD by spiking a quality system matrix at a concentration of at least two (2) times but no greater than four times the MDL. This spike concentration establishes the LOD and the concentration at which the LOD shall be verified. It is specific to each suite of analyte, matrix, and method (including sample preparation). The following requirements apply to the initial LOD establishment and to the LOD verifications:
- The apparent signal to noise (S/N) ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean method blank concentration. This is initially estimated based on a minimum of four method blank analyses and later established with a minimum of 20 method blank results.
  - If the LOD verification fails, then the laboratory must repeat the MDL determination and LOD verification or perform and pass two consecutive LOD verifications at a higher spike concentration and set the LOD at the higher concentration.
  - The laboratory shall maintain documentation for all MDL determinations and LOD verifications.
  - The DL and LOD must be reported for all analyte-matrix-methods suites unless it is not applicable to the test or specifically excluded by project requirements.
- 4.12 The LOD shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOD verifications on a one per batch basis. All verification data will be in compliance, reported, and available for review.
- 4.13 For DoD, at a minimum, the LOQ shall be verified quarterly. In situations where methods are setup and used on an infrequent basis, the laboratory may choose to perform LOQ verifications on a one per batch basis.
- 4.14 All initial instrument calibrations must be verified with a standard obtained from a second manufacturer prior to analyzing any samples. The use of a standard from a second lot obtained from the same manufacturer (independently prepared from different source materials) is acceptable for use as a second source standard. The concentration of the second source standard shall be at or near the midpoint of the calibration range. The acceptance criteria for the initial calibration verification must be at least as stringent as those for the continuing calibration verification.
- 4.15 Exclusion of calibration points without documented scientifically valid technical justification is not permitted.
- 4.16 The concentration of the CCV standard shall be greater than the low calibration standard and less than or equal to the midpoint of the calibration range.
- 4.17 All CCVs analyzed must be evaluated and reported. If a CCV fails, reanalysis or corrective actions must be taken.
- If a CCV fails, the laboratory can immediately analyze two additional consecutive CCVs (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed CCV and the two additional CCVs). This

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approach allows for spurious failures of analytes to be reported without reanalysis of samples. Any corrective actions that change the dynamics of the system (e.g., clip column, clean injection port, run blanks) requires that all samples since the last acceptable CCV be reanalyzed.

- Both of these CCVs must meet acceptance criteria in order for the samples to be reported without reanalysis.
- If either of these two CCVs fail or if the laboratory cannot immediately analyze two CCVs, the associated samples cannot be reported and must be reanalyzed.
- Corrective action(s) and recalibration must occur if the above scenario fails. All affected samples since the last acceptable CCV must be reanalyzed.
- Flagging of data for a failed CCV is only appropriate when the affected samples cannot be reanalyzed. The laboratory must notify the client prior to reporting data associated with a failed CCV.

- 4.18 The results of all MS/MSDs must be evaluated using the same acceptance criteria used for the DoD LCS limits (see Tables 3 and 4) or project limits, if specified. If the specific analyte(s) are not available in the Tables 3 and 4, the laboratory shall use their LCS in-house limits (see the LIMS) as a means of evaluating MS/MSDs. The MS and MSD must be spiked with all reported analytes.
- 4.19 Surrogate spike results shall be compared with DoD LCS limits (see Tables 3 and 4) or acceptance criteria specified by the client. If these criteria are not available, the laboratory shall compare the results with its in-house statistically established LCS criteria (see the LIMS).
- 4.20 The method blank shall be considered to be contaminated if:
- The concentration of any target analyte (chemical of concern) in the blank exceeds 1/2 the LOQ and is greater than 1/10th the amount measured in any associated sample, or 1/10th the regulatory limit, whichever is greater.
  - The concentration of any common laboratory contaminant in the blank exceeds the LOQ.
  - If a method blank is contaminated as described above, then the laboratory shall reprocess affected samples in a subsequent preparation batch, except when sample results are below the LOD. If insufficient sample volume remains for reprocessing, the results shall be reported with appropriate data qualifiers.
- 4.21 Sporadic Marginal Exceedances are not allowed for target analytes (chemicals of concern as identified by a project) without project-specific approval. Target analytes are considered those few analytes that are critical for the success of a project (such as risk drivers) where sporadic marginal exceedances cannot be allowed. Laboratories should consult with clients whenever long lists of analytes are requested for analysis to determine if marginal exceedances will not be allowed.
- 4.22 DoD considers the same analyte exceeding the LCS control limit two (2) out of three (3) consecutive LCS to be indicative of non-random behavior, which requires corrective action and reanalysis of the LCS.

**STANDARD OPERATING PROCEDURE**

**TITLE:** Volatile Organic Compounds by GC/MS  
(EPA 8260B, 8260C, 8260D, 624, 624.1, and SM6200 B)

**ISSUER:** Pace National – Mt. Juliet, Tennessee

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**Table 3. LCS Control Limits – Method 8260 Solid Matrix**

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
630-20-6	1,1,1,2-Tetrachloroethane	11115	101.1	7.8	78	125
71-55-6	1,1,1-Trichloroethane	12156	101.6	9.4	73	130
79-34-5	1,1,2,2-Tetrachloroethane	11670	97	8.9	70	124
79-00-5	1,1,2-Trichloroethane	11772	99.7	7.2	78	121
76-13-1	1,1,2-Trifluoro-1,2,2-trichloroethane [Freon-113]	9760	100.8	11.7	66	136
75-34-3	1,1-Dichloroethane	11856	100.4	8.1	76	125
75-35-4	1,1-Dichloroethene	12352	100.3	10.1	70	131
563-58-6	1,1-Dichloropropene	10793	100.5	8.3	76	125
87-61-6	1,2,3-Trichlorobenzene	10572	97.8	10.6	66	130
96-18-4	1,2,3-Trichloropropane	10925	99.1	8.8	73	125
526-73-8	1,2,3-Trimethylbenzene	1948	99.8	6	82	118
120-82-1	1,2,4-Trichlorobenzene	10980	98	10.4	67	129
95-63-6	1,2,4-Trimethylbenzene	11085	98.7	7.9	75	123
96-12-8	1,2-Dibromo-3-chloropropane	11380	96.6	11.7	61	132
106-93-4	1,2-Dibromoethane	11408	100.1	7.3	78	122
95-50-1	1,2-Dichlorobenzene	11785	99.1	7.2	78	121
107-06-2	1,2-Dichloroethane	12328	100.5	9.2	73	128
17060-07-0	1,2-Dichloroethane-d4	5951	103.1	10.8	71	136
540-59-0	1,2-Dichloroethene	7748	99.9	7.3	78	122
78-87-5	1,2-Dichloropropane	12145	99.5	7.8	76	123
354-23-4	1,2-Dichlorotrifluoroethane [Freon 123a]	1269	97.8	11.3	64	132
108-70-3	1,3,5-Trichlorobenzene	4723	99.4	9.6	71	128
108-67-8	1,3,5-Trimethylbenzene	11080	98.4	8.4	73	124
541-73-1	1,3-Dichlorobenzene	11619	98.9	7.4	77	121
142-28-9	1,3-Dichloropropane	10713	99.1	7.3	77	121
542-75-6	1,3-Dichloropropene	3714	101.6	8.1	77	126
106-46-7	1,4-Dichlorobenzene	11848	97.5	7.6	75	120
105-05-5	1,4-Diethylbenzene	1896	96.6	5.9	79	114
123-91-1	1,4-Dioxane	7698	96.4	13.7	55	138
544-10-5	1-Chlorohexane	2543	100.4	9.8	71	130
594-20-7	2,2-Dichloropropane	10703	99.7	11.1	67	133
78-93-3	2-Butanone [MEK]	11514	99.6	16.3	51	148
126-99-8	2-Chloro-1,3-butadiene	6667	99	11.3	65	133
110-75-8	2-Chloroethyl vinyl ether	6957	96.1	17.6	43	149
95-49-8	2-Chlorotoluene	10838	98.5	7.9	75	122
591-78-6	2-Hexanone	11004	99.1	15.4	53	145
79-46-9	2-Nitropropane	4969	98.3	17.1	47	150
67-63-0	2-Propanol [Isopropyl alcohol]	1696	99.8	13.4	60	140
460-00-4	4-Bromofluorobenzene	6267	98.9	6.8	79	119
106-43-4	4-Chlorotoluene	10785	98.3	8.6	72	124
108-10-1	4-Methyl-2-pentanone [MIBK]	11364	99.6	11.6	65	135
67-64-1	Acetone	11089	99.6	21.4	36	164
75-05-8	Acetonitrile	5697	98.5	14.8	54	143
107-02-8	Acrolein [Propenal]	7528	101.1	18	47	155

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**ISSUER:** Pace National – Mt. Juliet, Tennessee

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<b>Table 3. LCS Control Limits – Method 8260 Solid Matrix</b>						
<b>CAS ID</b>	<b>Analyte</b>	<b>N Records</b>	<b>Mean</b>	<b>Standard Deviation</b>	<b>Lower Control Limit</b>	<b>Upper Control Limit</b>
107-13-1	Acrylonitrile	8293	99.7	11.4	65	134
107-05-1	Allyl chloride	6908	101.1	11.2	68	135
71-43-2	Benzene	12853	99.2	7.4	77	121
100-44-7	Benzyl chloride	2743	92.1	9.4	64	120
108-86-1	Bromobenzene	10974	99.3	7.3	78	121
74-97-5	Bromochloromethane	11023	101.4	7.8	78	125
75-27-4	Bromodichloromethane	11850	101	8.5	75	127
75-25-2	Bromoform	11890	99.1	10.8	67	132
74-83-9	Bromomethane	11416	98.3	15	53	143
75-15-0	Carbon disulfide	11132	97.9	11.5	63	132
56-23-5	Carbon tetrachloride	12090	102.3	10.7	70	135
108-90-7	Chlorobenzene	12382	99.7	6.9	79	120
124-48-1	Chlorodibromomethane	11852	100.2	8.7	74	126
75-00-3	Chloroethane	11444	98.8	13.3	59	139
67-66-3	Chloroform	12344	100.3	7.6	78	123
74-87-3	Chloromethane	11876	93.3	14.3	50	136
156-59-2	cis-1,2-Dichloroethene	11645	99.9	7.6	77	123
10061-01-5	cis-1,3-Dichloropropene	11805	99.8	8.7	74	126
1476-11-5	cis-1,4-Dichloro-2-butene	977	106	12.4	69	143
110-82-7	Cyclohexane	8827	98.9	10.6	67	131
108-94-1	Cyclohexanone	3764	93.2	20.9	30	156
1868-53-7	Dibromofluoromethane	2142	98.1	6.8	78	119
74-95-3	Dibromomethane	10913	101.1	7.9	78	125
75-71-8	Dichlorodifluoromethane [Freon-12]	11467	88.9	20.1	29	149
75-05-8	Acetonitrile	5697	98.5	14.8	54	143
107-02-8	Acrolein [Propenal]	7528	101.1	18	47	155
107-13-1	Acrylonitrile	8293	99.7	11.4	65	134
107-05-1	Allyl chloride	6908	101.1	11.2	68	135
71-43-2	Benzene	12853	99.2	7.4	77	121
100-44-7	Benzyl chloride	2743	92.1	9.4	64	120
108-86-1	Bromobenzene	10974	99.3	7.3	78	121
74-97-5	Bromochloromethane	11023	101.4	7.8	78	125
75-27-4	Bromodichloromethane	11850	101	8.5	75	127
75-25-2	Bromoform	11890	99.1	10.8	67	132
74-83-9	Bromomethane	11416	98.3	15	53	143
75-15-0	Carbon disulfide	11132	97.9	11.5	63	132
56-23-5	Carbon tetrachloride	12090	102.3	10.7	70	135
108-90-7	Chlorobenzene	12382	99.7	6.9	79	120
124-48-1	Chlorodibromomethane	11852	100.2	8.7	74	126
75-00-3	Chloroethane	11444	98.8	13.3	59	139
67-66-3	Chloroform	12344	100.3	7.6	78	123
74-87-3	Chloromethane	11876	93.3	14.3	50	136
156-59-2	cis-1,2-Dichloroethene	11645	99.9	7.6	77	123
10061-01-5	cis-1,3-Dichloropropene	11805	99.8	8.7	74	126
1476-11-5	cis-1,4-Dichloro-2-butene	977	106	12.4	69	143

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**Table 3. LCS Control Limits – Method 8260 Solid Matrix**

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
110-82-7	Cyclohexane	8827	98.9	10.6	67	131
108-94-1	Cyclohexanone	3764	93.2	20.9	30	156
1868-53-7	Dibromofluoromethane	2142	98.1	6.8	78	119
74-95-3	Dibromomethane	10913	101.1	7.9	78	125
75-71-8	Dichlorodifluoromethane [Freon-12]	11467	88.9	20.1	29	149
75-43-4	Dichlorofluoromethane	717	100.8	18	47	155
60-29-7	Diethyl ether	6283	99.6	9.6	71	129
108-20-3	Diisopropyl ether	8542	98.3	9.7	69	127
64-17-5	Ethanol	3958	102.2	18.9	45	159
141-78-6	Ethyl acetate	4516	95.4	14.5	52	139
97-63-2	Ethyl methacrylate	7075	98.9	9.9	69	129
637-92-3	Ethyl tert-butyl ether	7514	98.9	9.1	72	126
100-41-4	Ethylbenzene	12427	99.1	7.7	76	122
462-06-6	Fluorobenzene	689	97.3	5.4	81	114
142-82-5	Heptane	5420	93.4	14.9	49	138
87-68-3	Hexachlorobutadiene	10264	98.1	12.4	61	135
67-72-1	Hexachloroethane	3265	102.5	10.1	72	133
110-54-3	Hexane	7116	93.6	16.1	45	142
74-88-4	Iodomethane	9457	100.9	10.1	71	131
78-83-1	Isobutyl alcohol	6162	97.5	12.6	60	135
108-21-4	Isopropyl acetate [Acetic acid]	2885	94.2	12.2	58	131
98-82-8	Isopropylbenzene	11596	100.8	11.1	68	134
179601-23-1	m/p-Xylene [3/4-Xylene]	10612	100.4	7.7	77	124
126-98-7	Methacrylonitrile	6736	99.2	11.1	66	132
79-20-9	Methyl acetate	8320	98.7	15.2	53	144
80-62-6	Methyl methacrylate	7050	98.4	11.9	63	134
1634-04-4	Methyl tert-butyl ether [MTBE]	11253	98.9	8.7	73	125
108-87-2	Methylcyclohexane	8565	99.4	11.2	66	133
75-09-2	Methylene chloride	12024	98.9	9.7	70	128
123-86-4	n-Butyl acetate	2981	95.1	11	62	128
71-36-3	n-Butyl alcohol	4800	92.9	12.6	55	131
104-51-8	n-Butylbenzene	10921	98.7	9.7	70	128
103-65-1	n-Propylbenzene	10947	98.9	8.8	73	125
91-20-3	Naphthalene	10602	95.6	11.2	62	129
95-47-6	o-Xylene	11940	100	7.7	77	123
99-87-6	p-Isopropyltoluene [p-Cymene]	10953	100.3	9	73	127
76-01-7	Pentachloroethane	5957	102	11.1	69	135
107-12-0	Propionitrile [Ethyl cyanide]	6734	101	11.1	68	134
135-98-8	sec-Butylbenzene	10960	99	8.8	73	126
100-42-5	Styrene	11809	100.2	8	76	124
994-05-8	tert-Amyl methyl ether [TAME]	7153	99.8	8.9	73	126
75-65-0	tert-Butyl alcohol	7492	100.5	10.7	68	133
98-06-6	tert-Butylbenzene	10974	98.8	8.6	73	125

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**Table 3. LCS Control Limits – Method 8260 Solid Matrix**

CAS ID	Analyte	N Records	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
127-18-4	Tetrachloroethene	12091	100.5	9.2	73	128
109-99-9	Tetrahydrofuran	8039	98	12.4	61	135
108-88-3	Toluene	12499	99.3	7.3	77	121
2037-26-5	Toluene-d8	6232	100.7	5.2	85	116
156-60-5	trans-1,2-Dichloroethene	11849	99.2	8.6	74	125
10061-02-6	trans-1,3-Dichloropropene	11805	100.9	9.8	71	130
110-57-6	trans-1,4-Dichloro-2-butene	8307	98.6	12.3	62	136
79-01-6	Trichloroethene	12440	100.2	7.6	77	123
75-69-4	Trichlorofluoromethane [Freon-11 ]	11530	101	13.1	62	140
108-05-4	Vinyl acetate	7260	100.3	16.9	50	151
75-01-4	Vinyl chloride	12129	95.6	13.2	56	135
1330-20-7	Xylenes [total]	8623	100.7	7.7	78	124
104-51-8	n-Butylbenzene	10921	98.7	9.7	70	128
103-65-1	n-Propylbenzene	10947	98.9	8.8	73	125
91-20-3	Naphthalene	10602	95.6	11.2	62	129
95-47-6	o-Xylene	11940	100	7.7	77	123
99-87-6	p-Isopropyltoluene [p-Cymene]	10953	100.3	9	73	127
76-01-7	Pentachloroethane	5957	102	11.1	69	135
107-12-0	Propionitrile [Ethyl cyanide]	6734	101	11.1	68	134
135-98-8	sec-Butylbenzene	10960	99	8.8	73	126
100-42-5	Styrene	11809	100.2	8	76	124
994-05-8	tert-Amyl methyl ether [TAME]	7153	99.8	8.9	73	126
75-65-0	tert-Butyl alcohol	7492	100.5	10.7	68	133
98-06-6	tert-Butylbenzene	10974	98.8	8.6	73	125
127-18-4	Tetrachloroethene	12091	100.5	9.2	73	128
109-99-9	Tetrahydrofuran	8039	98	12.4	61	135
108-88-3	Toluene	12499	99.3	7.3	77	121
2037-26-5	Toluene-d8	6232	100.7	5.2	85	116
156-60-5	trans-1,2-Dichloroethene	11849	99.2	8.6	74	125
10061-02-6	trans-1,3-Dichloropropene	11805	100.9	9.8	71	130
110-57-6	trans-1,4-Dichloro-2-butene	8307	98.6	12.3	62	136
79-01-6	Trichloroethene	12440	100.2	7.6	77	123
75-69-4	Trichlorofluoromethane [Freon-11 ]	11530	101	13.1	62	140
108-05-4	Vinyl acetate	7260	100.3	16.9	50	151
75-01-4	Vinyl chloride	12129	95.6	13.2	56	135
1330-20-7	Xylenes [total]	8623	100.7	7.7	78	124

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<b>Table 4. LCS Control Limits – Method 8260 Water Matrix</b>						
<b>CAS ID</b>	<b>Analyte</b>	<b>N Records</b>	<b>Mean</b>	<b>Standard Deviation</b>	<b>Lower Control Limit</b>	<b>Upper Control Limit</b>
630-20-6	1,1,1,2-Tetrachloroethane	24511	101.1	7.6	78	124
71-55-6	1,1,1-Trichloroethane	28223	102.7	9.6	74	131
79-34-5	1,1,2,2-Tetrachloroethane	27450	96.4	8.3	71	121
79-00-5	1,1,2-Trichloroethane	27338	99.5	6.5	80	119
76-13-1	1,1,2-Trifluoro-1,2,2-trichloroethane [Freon-113]	21122	103	11.1	70	136
75-34-3	1,1-Dichloroethane	28154	101.3	8	77	125
75-35-4	1,1-Dichloroethene	29436	101	10	71	131
563-58-6	1,1-Dichloropropene	23631	102	7.8	79	125
87-61-6	1,2,3-Trichlorobenzene	24271	98.7	10.1	69	129
96-18-4	1,2,3-Trichloropropane	24525	97.5	8	73	122
526-73-8	1,2,3-Trimethylbenzene	2965	100.9	6.2	82	120
120-82-1	1,2,4-Trichlorobenzene	25290	99.8	10.1	69	130
95-63-6	1,2,4-Trimethylbenzene	27917	99.6	8	76	124
96-12-8	1,2-Dibromo-3-chloropropane	24955	94.9	11.1	62	128
106-93-4	1,2-Dibromoethane	29096	99	7.2	77	121
95-50-1	1,2-Dichlorobenzene	27583	99.4	6.5	80	119
107-06-2	1,2-Dichloroethane	32965	100.3	9.2	73	128
17060-07-0	1,2-Dichloroethane-d4	8673	99.5	6.1	81	118
540-59-0	1,2-Dichloroethene	18667	100.2	7.1	79	121
78-87-5	1,2-Dichloropropane	27787	100.1	7.2	78	122
354-23-4	1,2-Dichlorotrifluoroethane [Freon 123a]	3144	103.1	10.9	70	136
108-70-3	1,3,5-Trichlorobenzene	10037	102.1	9.2	75	130
108-67-8	1,3,5-Trimethylbenzene	27820	99.5	8.1	75	124
106-99-0	1,3-Butadiene	1202	100.6	19.2	43	158
541-73-1	1,3-Dichlorobenzene	26951	99.7	6.5	80	119
142-28-9	1,3-Dichloropropane	23811	99.1	6.5	80	119
542-75-6	1,3-Dichloropropene	9784	99.9	7.6	77	123
106-46-7	1,4-Dichlorobenzene	27715	98.3	6.5	79	118
105-05-5	1,4-Diethylbenzene	1980	98.4	6.4	79	118
123-91-1	1,4-Dioxane	17866	99	13.4	59	139
544-10-5	1-Chlorohexane	5790	99.6	8	76	124
540-84-1	2,2,4-Trimethylpentane [Isooctane]	5432	95.2	12.3	58	132
594-20-7	2,2-Dichloropropane	23775	99.7	13.2	60	139
75-85-4	2-Butanol	4332	92.7	9.1	66	120
78-93-3	2-Butanone [MEK]	26659	99.6	14.6	56	143
126-99-8	2-Chloro-1,3-butadiene	15673	100	11.7	65	135
110-75-8	2-Chloroethyl vinyl ether	18225	94.7	14.7	51	139
95-49-8	2-Chlorotoluene	23750	100	7.2	79	122
591-78-6	2-Hexanone	25368	97.9	13.5	57	139
91-57-6	2-Methylnaphthalene	3754	79.4	20.9	17	142
79-46-9	2-Nitropropane	10213	92.6	14.5	49	136
67-63-0	2-Propanol [Isopropyl alcohol]	2034	98.8	14.4	56	142
624-95-3	3,3-Dimethyl-1-butanol	6491	90.9	13.9	49	133

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